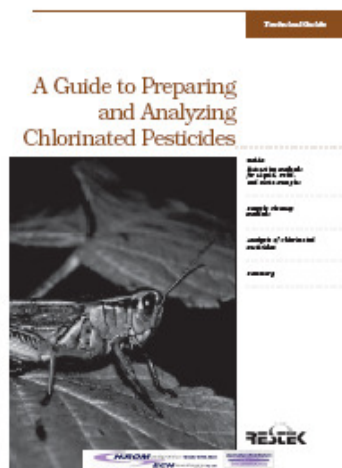
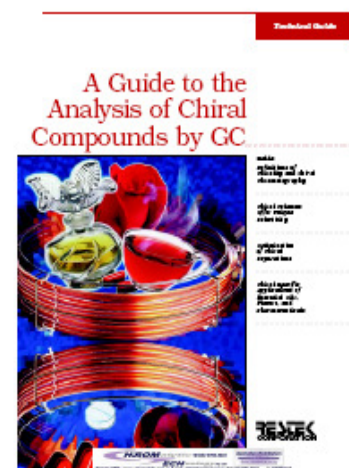
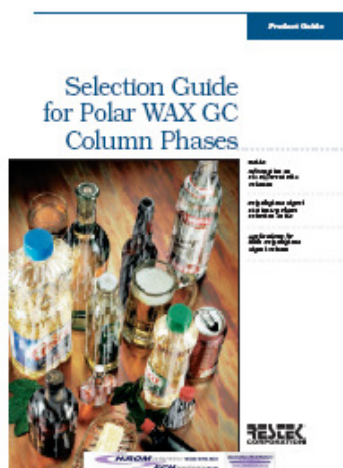
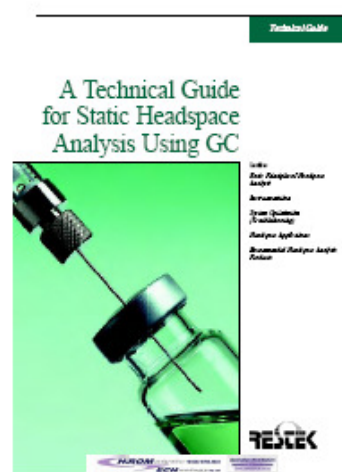
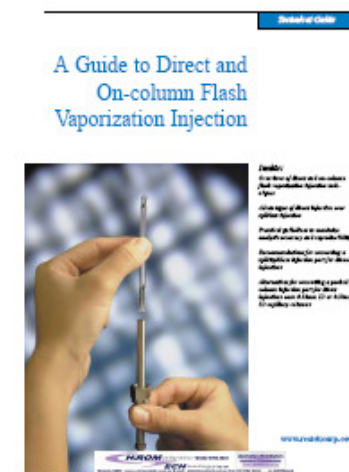
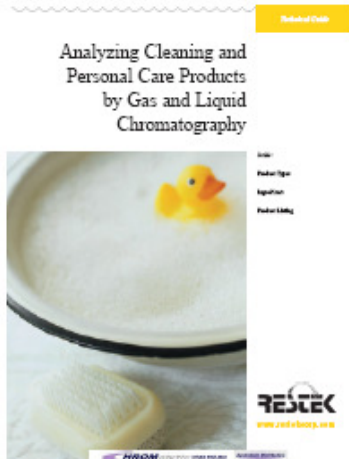
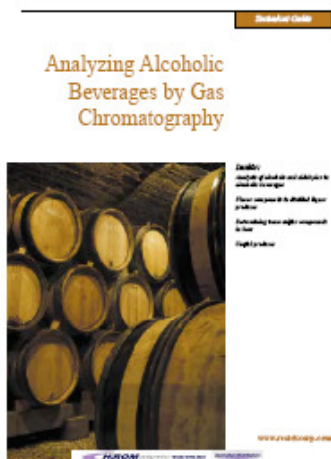


RESTEK Technical Guides

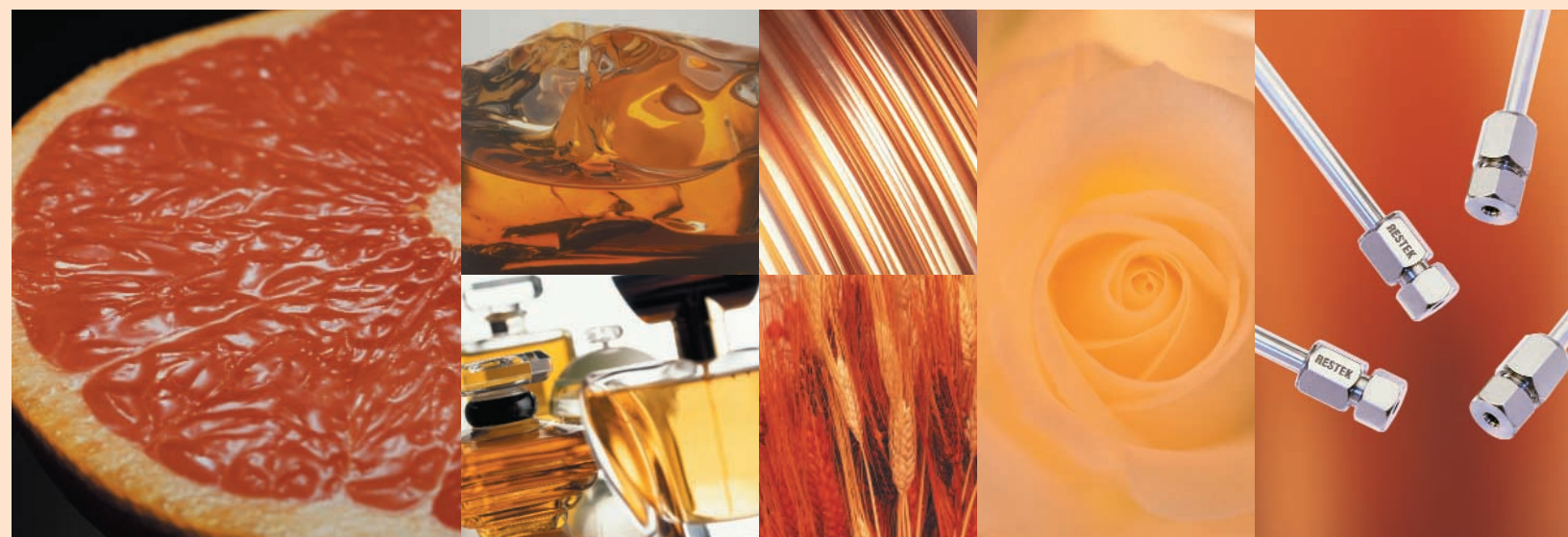


Foods Flavors & Fragrances In-Review

Restek manufactures many chromatography columns and sample preparation products for foods, flavors, or fragrances analyses.

Helpful **free technical literature** relative to these applications is summarized in this review. To obtain any of these publications, simply check and return the enclosed business reply card, or visit our website:

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Foods

Analysis of Cholesterol and Other Dietary Sterols (lit. cat.# 59581)

Analyze many sterols without derivatization, using inert capillary GC columns. Capillary GC is a time-saving, efficient, and accurate approach for qualitative and quantitative analyses of sterols. Nonpolar stationary phases generally are suitable for monitoring dietary sterols, but a more polar column sometimes is needed to resolve complex mixtures. This 2-page note describes and illustrates analyses of derivatized and underivatized sterols.

Analyzing Free Fatty Acids (lit. cat.# 59583)

Avoid derivatizing samples and save time, effort, and expense

Typical sample preparation procedures for fatty acids analysis call for derivatizing these analytes, but a properly designed GC analysis of the free acids saves valuable time, effort, and materials. Read this 4-page note for information about selecting a column, and steps to take to ensure accurate results, when analyzing free fatty acids.

High-Resolution Analyses of Fatty Acid Methyl Esters (FAMES) by Gas Chromatography (lit. cat.# 59489A)

Characterizing fats and oils, or determining fat content in food, calls for highly efficient separations of fatty acid methyl esters (FAMES) on capillary GC columns. A properly chosen column can provide accurate information about total fat, *trans* fat, or total omega-3 polyunsaturate content.

Polyethylene glycol-type stationary phases typically are used for separating, identifying, and quantifying saturated and unsaturated FAMES. More polar biscyanopropyl phases are needed to resolve *cis* and *trans* isomers of polyunsaturates or quantify total *trans* fat. This 4-page note discusses these challenging separations.

HPLC Analysis of Vitamins (lit. cat.# 59181)

Monitor fat-soluble or water-soluble vitamins in foods or dietary supplements

The need to accurately quantify vitamins in food products and dietary supplements necessitates simple, reliable, and accurate analytical procedures. Our 2-page note describes an HPLC column and conditions for analyzing fat-soluble vitamins and equivalent information for analyzing water-soluble vitamins.

Detection of Synthetic and Natural Antioxidants in Food (lit. cat.# 59582)

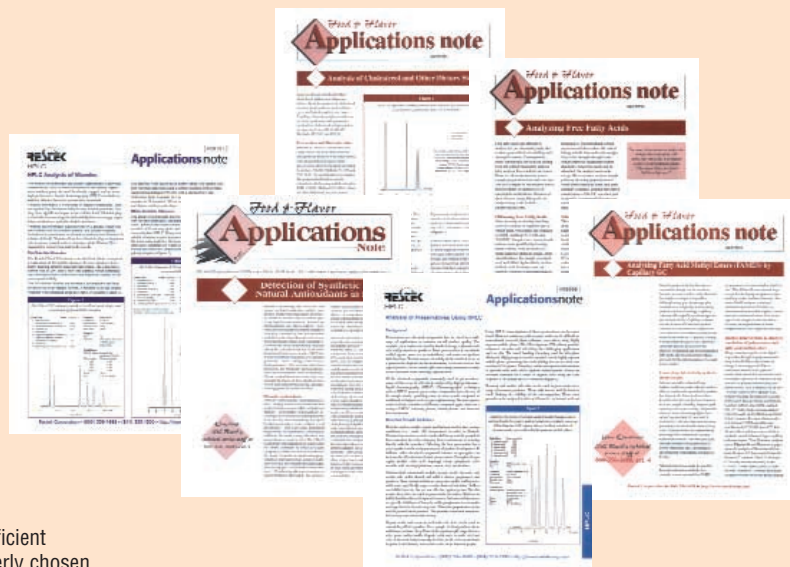
Analyze antioxidants, using capillary GC

Phenolic antioxidants (BHA, BHT, etc.) can be analyzed simultaneously—quickly and effectively—on an intermediate-polarity capillary GC column. In addition, inert capillary GC columns save time in tocopherols analysis because the analytes do not have to be derivatized. This 2-page note describes analytical columns and conditions for antioxidants analyses.

Analysis of Preservatives, Using HPLC (lit. cat.# 59398)

Optimize retention and selectivity

HPLC is a powerful tool for quantifying these compounds in food and beverage products. Analyses illustrated in this 6-page note include benzoic and sorbic acids, parabens, phenolic antioxidants, and tocopherols.



Nutraceuticals

Analyzing Nutraceutical Products by Liquid and Gas Chromatography (lit. cat.# 59364)

HPLC and GC methodologies meet the varied challenges of herbal products analysis

HPLC analyses of allicin in garlic, hyperforin in St. John's wort, and phenolics in Echinacea, and GC analysis of fatty acids in saw palmetto, described in our 4-page note, show HPLC and GC to be excellent tools for analyzing marker compounds in botanical materials.

The Institute for Nutraceutical Advancement (INA) Validates GC Methods for Saw Palmetto, Using Rtx®-5 and Stabilwax® Columns (lit. cat.# 59136)

Gas chromatography is a useful tool for monitoring marker compounds in saw palmetto

Among the methods established by the INA are capillary GC analyses of fatty acids (as FAMES) and of phytosterols in saw palmetto. This 2-page note presents the analytical conditions for these analyses, using Stabilwax® and Rtx®-5 columns, respectively.

Determination of Omega-3 (n-3) and Omega-6 (n-6) Fatty Acid Composition in Evening Primrose Oil, Flax Seed Oil, Black Currant Oil, and Borage Oil (lit. cat.# 59128)

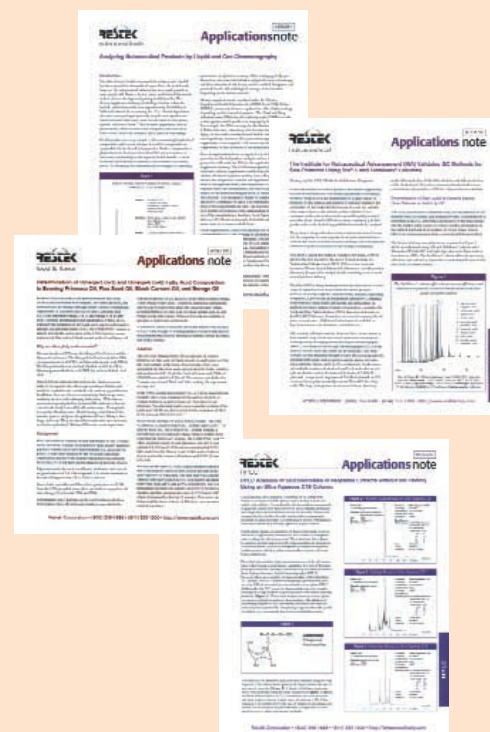
Capillary GC is an ideal approach to monitoring essential fatty acids in plant oils

This 4-page note describes and illustrates capillary GC analyses of omega-3 and omega-6 essential fatty acids in plant oils. The polar polyethylene glycol-based column resolves saturated and monounsaturated FAMES from the EFAs of interest, and resolves the isomers of linolenic acid as well.

HPLC Analysis of Glucosinolates in Vegetable Extracts, without Ion Pairing, Using an Ultra Aqueous C18 Column (lit. cat.# 59335)

Convenient, reproducible analyses for hydrophilic molecules with widely varying polarity

Glucosinolates with hydrophilic functional groups are very difficult to retain by conventional reversed phase HPLC, and when the functional groups in a sample are diverse, polarity can differ widely, complicating the problem. Until now, reversed phase HPLC with an ion pairing reagent has been the inconvenient, but necessary, approach to these analyses. This 2-page note describes a column and conditions for analyses of glucosinolates, without ion pairing.



Flavors and Fragrances

Analyzing the Heat Level of Spicy Foods, Using an Ultra C18 HPLC Column

(lit. cat.# 59199)

Add zest to spicy foods—but keep their analysis bland

As society's taste for spicy foods grows, so does the need to test and classify raw materials and final products for pungency. This 2-page note describes an HPLC column and analytical conditions for monitoring capsaicin and related compounds - major contributors to "heat" levels in chili pepper-containing foods. Samples require only minimal preparation, and results can be converted to traditional organoleptic Scoville heat units (SHU).

Analysis of Vanillin and Ethyl Vanillin in Vanilla Flavors, Using Ultra C8 Column

(lit. cat.# 59186)

A fast, efficient, and sensitive HPLC analysis for primary flavor components

AOAC Method 990.25 describes an HPLC analysis of flavor compounds in vanilla extract and artificial vanilla flavors. The analysis takes approximately 40 minutes, and the ethyl vanillin peak is significantly broadened. An Ultra C8 column and conditions presented in our 2-page note ensure a more efficient analysis, while reducing the run time by 15 minutes.

GC Analysis of Chiral Flavor Compounds in Apple Juices, Using the Rt-BDEXsm™ and Rt-BDEXse™ Columns

(lit. cat.# 59546)

Detect adulteration by examining enantiomer ratios of chiral molecules

Many flavor constituents are chiral compounds. In nature one enantiomer usually is prevalent, but when synthetically prepared the same compound is an equal (racemic) mixture. Consequently, gas chromatographic resolution and quantification of the enantiomers is a sensitive and reliable approach to detecting synthetic adulterants. The 4-page note describes this principle, using example chromatographic separations.

A Guide to the Analysis of Chiral Compounds by GC

(lit. cat.# 59889)

Resolve critical enantiomer pairs quickly and reliably

In flavor and fragrance analyses, enantiomers often must be resolved for separate quantification. Five cyclodextrin-based Restek chiral capillary GC columns offer a unique range of selectivities, to enable you to accomplish these often-difficult separations. Many example chromatograms in this 24-page guide will help you find the best column - or columns - for your particular need.

Analyzing Alcoholic Beverages by Gas Chromatography

(lit. cat.# 59462)

Selectivity, sensitivity, and minimal sample preparation make GC a powerful tool for monitoring alcoholic beverage composition

Volatile component profiles of alcoholic beverages reveal a wide range of compounds: acids, alcohols, aldehydes, and others. This 16-page guide describes packed column GC and capillary GC approaches to monitoring these complex mixtures of analytes. A separate section is devoted to detailed information about quantifying trace sulfur compounds in beer.



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chromatogram archive of both GC
and HPLC applications!

Catalogs and Technical Guides

Foods, Flavors, & Fragrances (lit. cat.# 59260)

A brief summary of many applications and chromatography products

This 48-page guide is a good overview of the foods - flavors - fragrances applications for which Restek GC and HPLC columns have been used. Application areas include amino acids, carbohydrates, chiral separations, essential oils, fats and oils, flavors and fragrances, organic acids, preservatives, and vitamins. Includes a handy index of retention times for many flavor and fragrance compounds on a dimethylpolysiloxane or a PEG-type capillary GC column.

Genuine Restek Replacement Parts for Agilent GCs (lit. cat.# 59627C)

Restek chromatography supplies and accessories - designed by chromatographers, for chromatographers.

This 44-page reference manual lists the consumer-replaceable items, supplies, and accessories you need to keep your Agilent GC running at top performance: injector and inlet supplies, detector parts and supplies, gas system components, tools, vials, syringes, and much more. Many items have been designed to save you time or improve your results, and are exclusive to Restek. Many other items are manufactured specifically to the instrument manufacturer's specifications.

Inlet Supplies (lit. cat.# 59893A)

for Agilent - PerkinElmer - Shimadzu - Thermo Finnigan - Varian gas chromatographs.

Most gas chromatography problems can be traced to the inlet or the injection. When you pay a little extra attention to the inlet - by changing liners, septa, and ferrules at regular intervals, changing the style of inlet liner when you change the type of analysis you are performing, etc. - you often avoid unanticipated downtime. Our handy, pocket-sized, 44-page guide lists inlet liners, seals, septa, ferrules, and other necessities to ensure the supplies you need are at hand.

Selection Guide for Polar WAX GC Column Phases (lit. cat.# 59890)

Confusion about "wax" columns resolved.

Polyethylene glycol-based stationary phases are the most versatile and thermally stable polar GC phases, but PEG-based columns from different manufacturers can differ significantly in chemical makeup and performance. Restek PEG-based capillary columns are designed for specific categories of applications, for low bleed, excellent inertness, and unsurpassed column-to-column reproducibility. This 16-page guide discusses the performance characteristics of and applications for Restek PEG-based columns.

A Technical Guide for Static Headspace Analysis, Using GC (lit. cat.# 59895A)

A wealth of information about a time- and money-saving technique

Static headspace analysis will save you time and money when assaying a variety of sample matrices - and often is easier than alternative sampling approaches. This 20-page guide discusses basic principles, instrumentation, applications, and troubleshooting. Restek capillary GC columns and an extensive selection of accessories will simplify getting started.

Operating Hints for Using Split/Splitless Injectors (lit. cat.# 59880A)

A popular Restek guide revised, updated, and expanded

What design of inlet liner is best for analyzing dirty samples by split injection? What design minimizes analyte breakdown in splitless injections? Learn about the advantages of these commonly used sample injection techniques—and their disadvantages—to obtain the best results and avoid problems. This 36-page guide is focused on system optimization, maintenance, and troubleshooting. An extensive selection of inlet liners and other products is included.



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Food Contaminants

Monitoring Volatile Compounds in Food Contact Packaging, Using Purge and Trap GC/MS and an Rtx®-5MS Capillary Column (lit. cat.# 59348)

An optimized procedure for monitoring common volatiles released by food contact packaging

All food packaging materials have a potential for generating volatile compounds when heated. These volatiles become a concern if they migrate into the food product in the package. This 4-page note summarizes one approach to testing food contact materials and describes an effective sampling - chromatography - detection system.

Acrylamide Analysis by Gas Chromatography (lit. cat.# 59485)

GC is a simple, low-cost, efficient way of detecting acrylamide in prepared foods

A proposed LC/MS/MS method for analyses of acrylamide in foods requires reversed phase HPLC in a highly aqueous mobile phase, a positive ion electrospray MS interface, and quantification based on comparison to a ¹³C-labeled internal standard. The GC alternative described in this 2-page note is rapid and cost-effective. Detection limits can approach 0.01µg/mL solution; for greater sensitivity, extracted acrylamide can be brominated, then quantified using an electron capture detector.

Improved Analysis of Organophosphorus Pesticides, Using Rtx®-OPPesticides and Rtx®-OPPesticides2 Columns (lit. cat.# 59359)

Fast analyses with notably few coelutions

To ensure sensitivity for low ppb levels of target compounds, dual-column GC analyses with ion-specific detectors (e.g., NPD / FPD) are used in analyses of organophosphorus pesticides. An Rtx®-OPPesticides / Rtx®-OPPesticides2 column pair minimizes the number of analyte coelutions and separates more than 50 OPPs in less than 25 minutes. This 4-page note describes conditions for both dual column / ion-specific detection and GC/MS analyses.

A Guide to Preparing and Analyzing Chlorinated Pesticides (lit. cat.# 59892)

Invaluable information that can simplify a challenging analysis

Analyses of chlorinated pesticides can be difficult because samples often are contaminated with non-target compounds (e.g., lipids), and the method can require rigorous quality control. Our 24-page guide covers sample extraction methodology, sample cleanup, and chromatography. A chromatographic analysis of widely used chlorinated herbicides also is illustrated. One of our most popular technical guides.

CarboPrep™ SPE Cleanup of Method 8081A Chlorinated Pesticides (lit. cat.# 59110)

Conserve solvent, ensure cleaner extracts and high recovery of target pesticides

A good companion publication to chlorinated pesticides guide 59892, this 2-page note describes the benefits of using graphitized carbon-based CarboPrep™ SPE tubes and presents an example extraction and GC analysis. Reduced solvent consumption during the extraction process, cleaner extracts, and excellent recovery rates for target pesticides make CarboPrep™ SPE tubes an excellent choice for this application.

CarboPrep™ SPE Cleanup of Method 8141A Organophosphorus Pesticides and Herbicides (lit. cat.# 59142)

Reduced solvent consumption, cleaner extracts, high recovery of target compounds

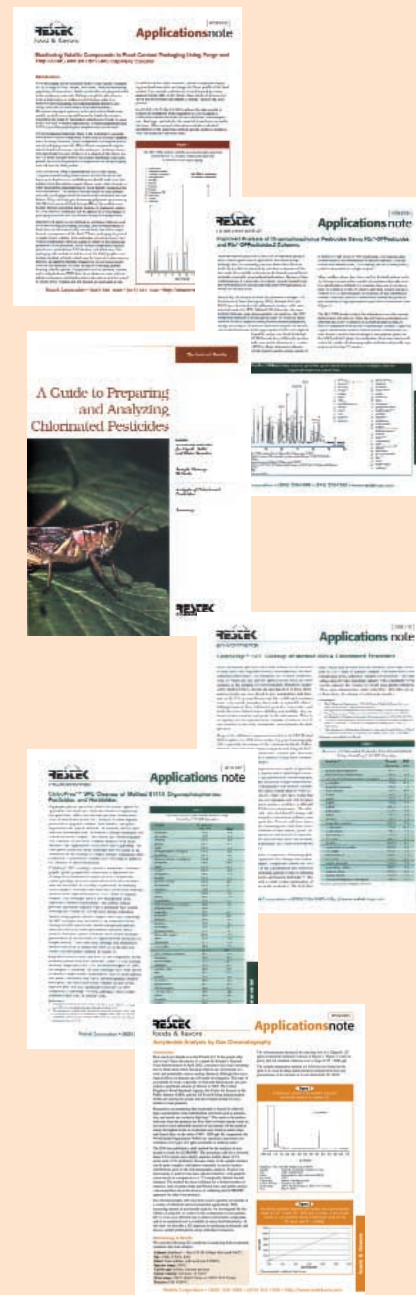
The same characteristics that make graphitized carbon-based CarboPrep™ SPE tubes an excellent choice for extracting chlorinated pesticides make them a better option than GPC or Florisil® SPE in analyses of organophosphorus pesticides and herbicides. The 2-page note describes the extraction procedure and GC analysis, and summarizes recovery data for more than 40 common OPPs.

GC Wall Chart (lit. cat.# 59668A)

Important information that saves time; could save a capillary column.

HPLC Column Selection Guide (lit. cat.# 59454)

Keep this useful chart with your workbooks, or post it on a wall
Quickly scan important characteristics of Restek HPLC columns. Includes brief, practical guidelines for choosing stationary phase, particle size, pore diameter, and column dimensions.



HPLC Accessories (lit. cat.# 59362)

This 4-page Fast Facts includes a balanced selection of replacement items and products that are optional, but which can make life in your laboratory easier.

Trident™ Direct Guard Column System (lit. cat.# 59314)

The ultimate combination of convenience and column protection

The Trident™ Direct system gives you three options for column protection: protection from particles, protection from particles and sample impurities, and protection from particles and heavy contamination. Cut costs by matching the level of column protection to your particular need. This 2-page Fast Facts summarizes the features and benefits of the Trident™ system and answers commonly asked questions.



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Analyzing Alcoholic Beverages by Gas Chromatography



Inside:

Analysis of alcohols and aldehydes in alcoholic beverages

Flavor compounds in distilled liquor products

Determining trace sulfur compounds in beer

Useful products

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HOT tech tip

Fusel Alcohols

Fusel alcohols are higher-order (i.e., secondary or tertiary) alcohols, traces of which usually are present in all beers. They are produced through a pathway very similar to the pathway for ethanol, the preferred alcohol of beer. Fusel alcohols contribute a hot, spicy, solvent-like flavor and an alcohol "burn". Small amounts of these components can be desirable in a strong ale or barley wine, but they can be offensive, and therefore are unwanted, in a Pilsner or other lager. In addition to their influence on flavor, they usually cause low carbonation and poor head retention in bottle-conditioned beers, because they are deadly to yeast. Higher fermentation temperatures can produce excessively rapid yeast growth, and yeast mutations, which, in turn, stimulate the formation of these components.

Introduction

The volatile component profiles of alcoholic beverage products consist of a wide range of compounds, including acids, alcohols, aldehydes, and other trace level flavor compounds. Analysts trained in the sensory evaluation of distilled liquors, wines, or beers tell us no two products are exactly alike. The unique sensory properties of different types and brands of distilled liquor products often are due to minor differences among the volatile components present. By using instrumental methods for qualitatively or quantitatively evaluating these differences, in addition to sensory techniques, quality assurance analysts can obtain a wealth of information about their products.

In addition to alcohols and flavor compounds, impurities such as sulfur gases occasionally are present, and might lead to off odors or flavors in the product. Because even parts per billion (ppb) levels of sulfur compounds can impact product quality, a sensitive and selective method of analysis is needed to detect these impurities. The majority of these contaminants are present in the gas phase, necessitating a gas phase sampling and analysis system. Because sulfur compounds also can be very reactive, an inert analysis system is highly desirable.

Gas chromatography (GC) is a powerful tool in the analysis of alcoholic beverage products. Minimal sample preparation, in general, is required, since the samples are in the liquid state in an alcohol or alcohol/water matrix. The flavor compounds tend to be volatile in nature, which fulfills one of the main requirements of GC. General detectors, such as the flame ionization detector (FID), or more information-rich detectors, such as the mass selective detector (MSD), can be used. Additionally, the ability to automate the analysis makes GC a very practical tool in a QA/QC environment. In this guide, we will discuss how GC can be used to (1) monitor alcohol content in alcoholic beverages, (2) determine the volatile profile of a product, and (3) detect trace level impurities.

Analysis of Alcohols and Aldehydes in Alcoholic Beverages

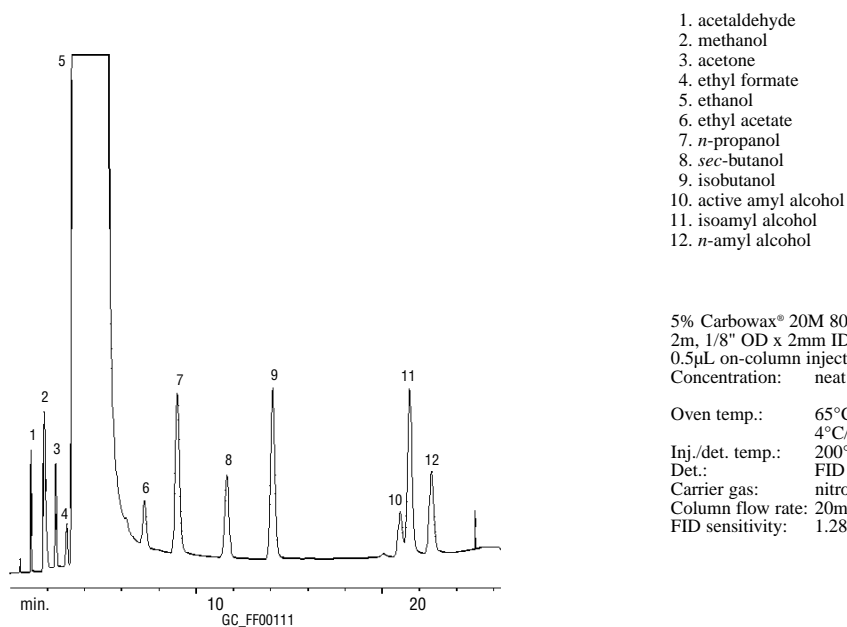
Alcoholic beverages contain a wide range of volatile compounds, including alcohols and short-chain aldehydes. Gas chromatography can be used to analyze these compounds without preliminary extractions. For example, AOAC International has published methods for the analysis of fusel oils, methanol, ethanol, and higher alcohols by GC.¹ Traditionally, packed columns prepared from glass tubing have been used for alcoholic beverage analysis, but these are prone to breakage and can adsorb some of the more reactive compounds. Restek's Silcosteel®-treated CarboBlack™ columns are made from stainless steel which has been treated to provide it with a deactivated silica surface. This conditioning significantly improves inertness and flexibility, relative to traditional glass packed columns.

CarboBlack™ packed columns can be used to quantify the various alcohols in alcoholic beverages. For example, ethanol can be monitored to determine the proof value of the beverage, while methanol and isopropanol can be quantified to determine the levels of denaturants present.² While poor methanol peak shapes often are associated with columns of limited sample capacity, a CarboBlack™ B packed column with 5% Carbowax® 20M provides an excellent peak shape for methanol, and completely resolves methanol from ethanol, as shown in Figure 1. In addition, the two predominant fusel oils, active amyl alcohol and isoamyl alcohol, can be resolved and monitored by using this column.

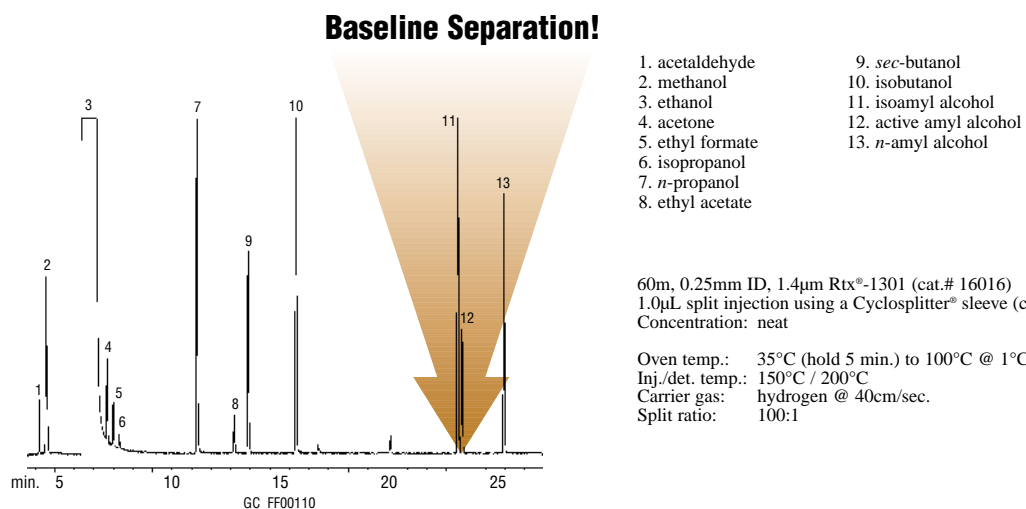
Alcohols and aldehydes in alcoholic beverages also can be monitored by capillary GC. Since capillary columns offer efficient separations, capillary GC is especially useful in analyses of structurally similar compounds, such as the fusel alcohols. The unique polarity of the Rtx®-1301 stationary phase ensures excellent resolution of a range of alcohols and fusel oils. An example of a rum analysis is shown in Figure 2.

Figure 1

Difficult-to-monitor alcoholic beverage components methanol, active amyl alcohol, and isoamyl alcohol can be quantified from a packed column analysis, using 5% Carbowax® on CarboBlack™ B.

**Figure 2**

An Rtx®-1301 capillary column offers excellent resolution of alcohols and fusel oils.



Additional Restek Literature

Performance information on six polyethylene glycol (PEG) columns—free on request.



Lit. Cat. #59890

Flavor Compounds in Distilled Liquor Products

Distilled liquor products contain a wide range of volatile and non-volatile compounds in an ethanol/water matrix. The most abundant fusel alcohols and esters can be determined by simple split injection, which also minimizes the amount of matrix ethanol and water transferred to the column. However, many trace-level fatty acids and their esters, which often are used to indicate product quality in alcoholic beverages such as whiskey and rum, cannot be determined by this approach. Capillary gas chromatography is a powerful tool for the analysis of these compounds, but the large ranges in volatilities and acidities can make it difficult to quantify all of the components in a single chromatographic separation. In addition, because the concentrations can vary widely, a splitless injection technique with some type of preconcentration step often is necessary. One example of this is large volume injection (LVI) with a venting step, which can be optimized to remove most of the matrix ethanol and water. Since some water will enter the chromatographic column, a stabilized phase, such as the Stabilwax®-DA phase, should be used.

By using a bonded polyethylene glycol (PEG) capillary column, flavor compounds in distilled liquor products can be quantified in a single splitless injection. A Stabilwax®-DA column was selected for this application, to improve peak shape and reproducibility for acidic components. An acidic functionality added to the PEG stationary phase reduces adsorption of acidic components and significantly reduces peak tailing. An optimized configuration of 30m, 0.18mm ID, and 0.18µm film thickness minimizes analysis times.

To optimize the chromatographic conditions for this analysis, we used a test mixture containing acids, esters, and flavor compounds typically found in alcoholic beverages (Figure 3). A computer modeling program, ezGC™, was used to optimize the column configuration, temperature program, and inlet flow for this system.

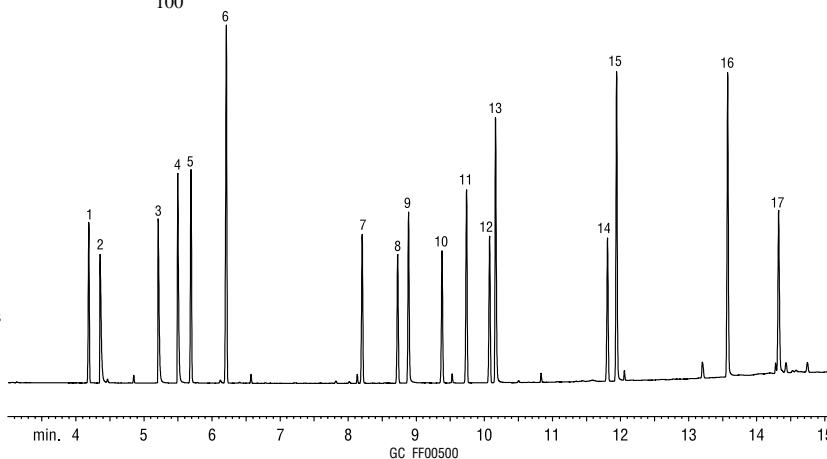
To test the applicability of this column in these dimensions, the critical pair of caproic acid and ethyl laurate was studied. These components can be very difficult to resolve on standard Carbowax®-type columns, especially if peak tailing or broadening occurs, or if one component is present at a significantly higher concentration. The Stabilwax®-DA column achieves baseline resolution of these two compounds in a reasonable analysis time (Figure 4).

Figure 3

Acids, esters, and flavor compounds typically found in alcoholic beverages are well resolved on a Stabilwax®-DA column.

Peak List	Conc. (ppm)		
1. ethyl octanoate	100	14. ethyl palmitate	50
2. acetic acid	100	15. decanoic acid	100
3. propionic acid	100	16. dodecanoic acid	100
4. isobutyric acid	100	17. vanillin	100
5. decanol 3	50		
6. ethyl decanoate	50		
7. ethyl laurate	50		
8. <i>cis</i> -lactone	100		
9. 2-phenylethanol	50		
10. <i>trans</i> -lactone	100		
11. methyl myristate	50		
12. ethyl myristate	50		
13. octanoic acid	100		

Stabilwax®-DA 30m, 0.18mm ID, 0.18µm (cat.# 550752)
 Inj.: 1µL splitless (hold 0.5 min.) at conc. shown in peak list, in ethyl acetate, 4mm ID splitless liner w/wool (cat.# 20814-202.1)
 Inj. temp.: 240°C
 Carrier gas: hydrogen
 Make-up gas: nitrogen
 Linear velocity: 28psi @ 240°C
 Oven temp.: 70°C to 240°C at 12°C/min. (hold 3 min.)
 Det.: FID



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Because alcoholic beverage samples often are injected via splitless mode, it was important to ascertain the stability of the Stabilwax®-DA column when exposed to aqueous injections. We verified stability by performing a splitless injection of the alcoholic beverage test mix, followed by five 1µL injections of water. We repeated this process 10 times, then made a final injection of the test mix. The chromatogram for the final test mix injection is shown in Figure 5. Even after repeated splitless injections of 100% water, there is very little degradation in the peak shapes for the test mix components. Over the course of the study, the variation in the peak retention times was 0.08-0.22% RSD. These data include retention times for the polar free fatty acids, which can be difficult to analyze under ideal conditions. The excellent stability of this stationary phase is demonstrated by the reproducibility of the retention times.

Figure 4

A Stabilwax®-DA column resolves the caproic acid / ethyl laurate critical pair to baseline.

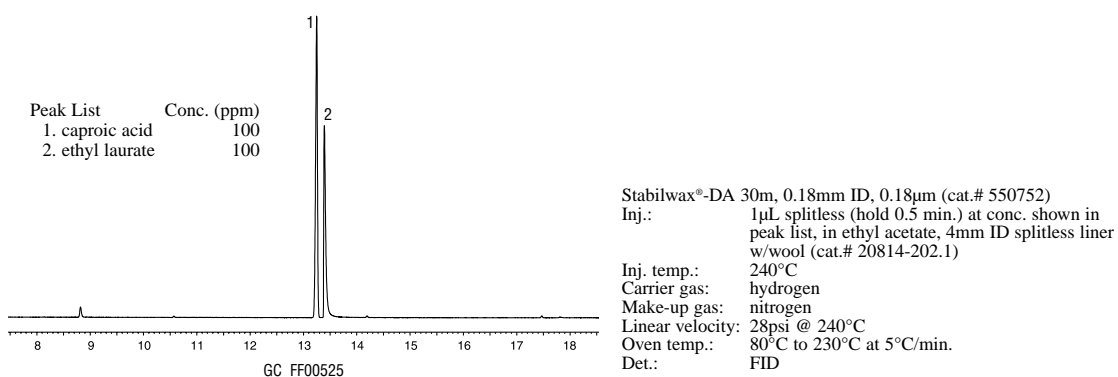
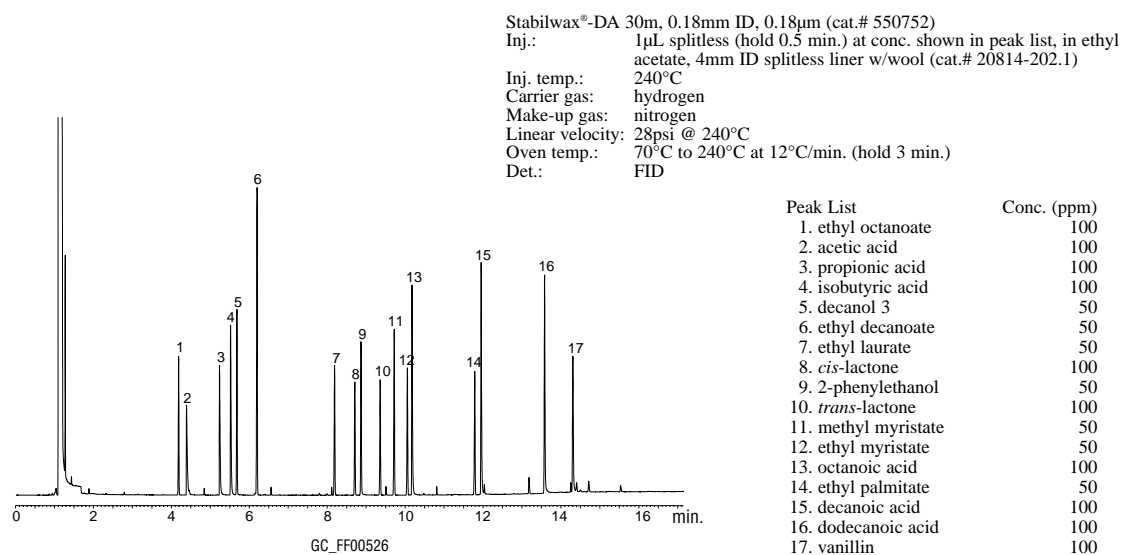


Figure 5

Stabilwax®-DA columns are well named: repeated injections of water produce very little degradation in the peak shapes for alcoholic beverage test mix components.

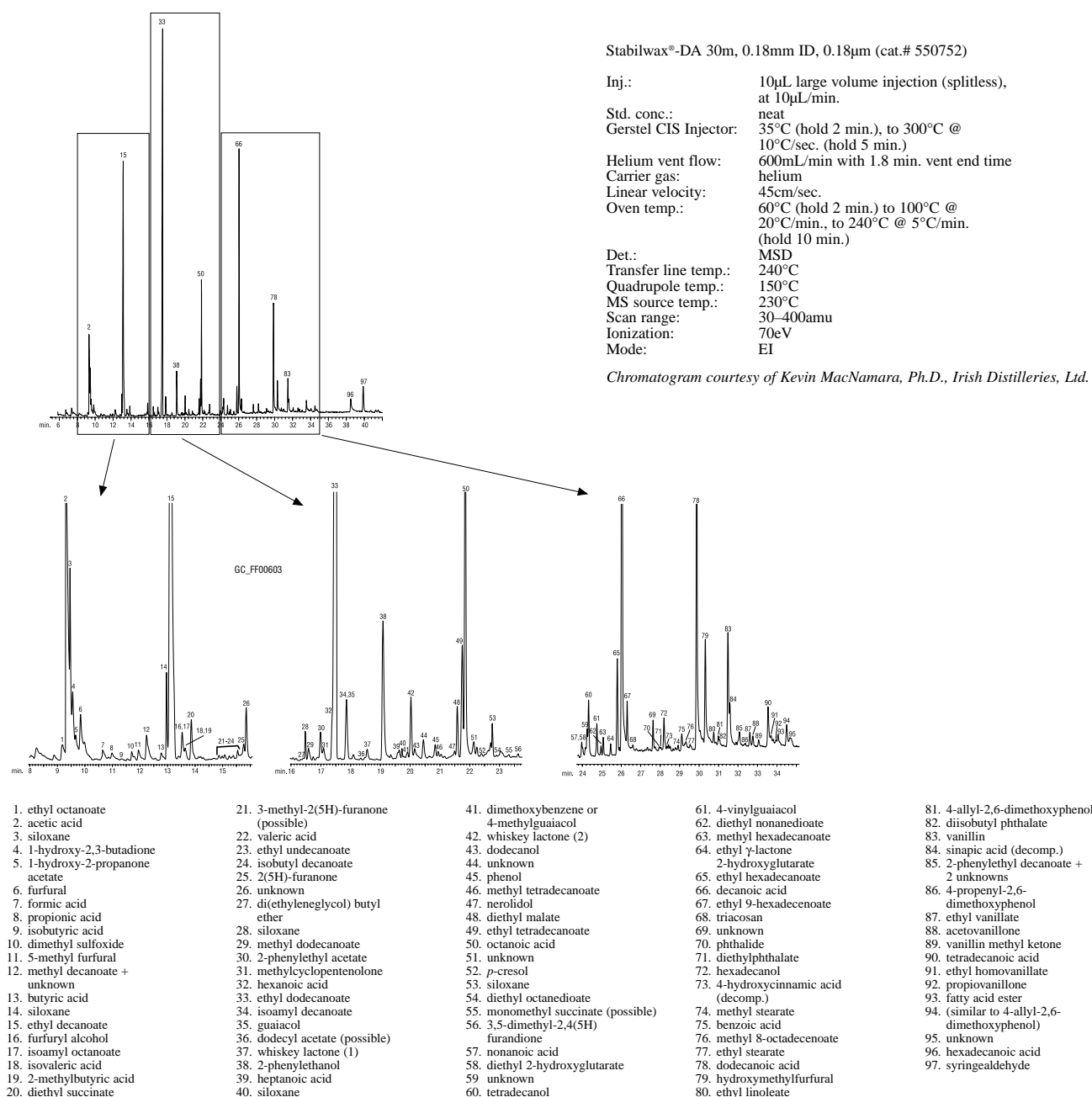


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Large volume injections (LVI) can be used to determine flavor compounds in alcoholic beverages such as malt whiskeys and grappas. Whiskey is distilled from a fermented mash of grain, such as corn, rye, barley, or wheat. The whiskey is aged in barrels or casks, and it is during the aging process that whiskey obtains its characteristic color, flavor, and aroma. Factors that influence the flavor of the final product include the characteristics of the grain, the recipe, and how the whiskey is distilled. The flavor profiles of whiskeys contain hundreds of compounds, including fatty acids, esters, alcohols, and aldehydes, in a wide range of concentrations. An example of a malt whiskey profile, determined by GC/MS, is shown in Figure 6.

Figure 6

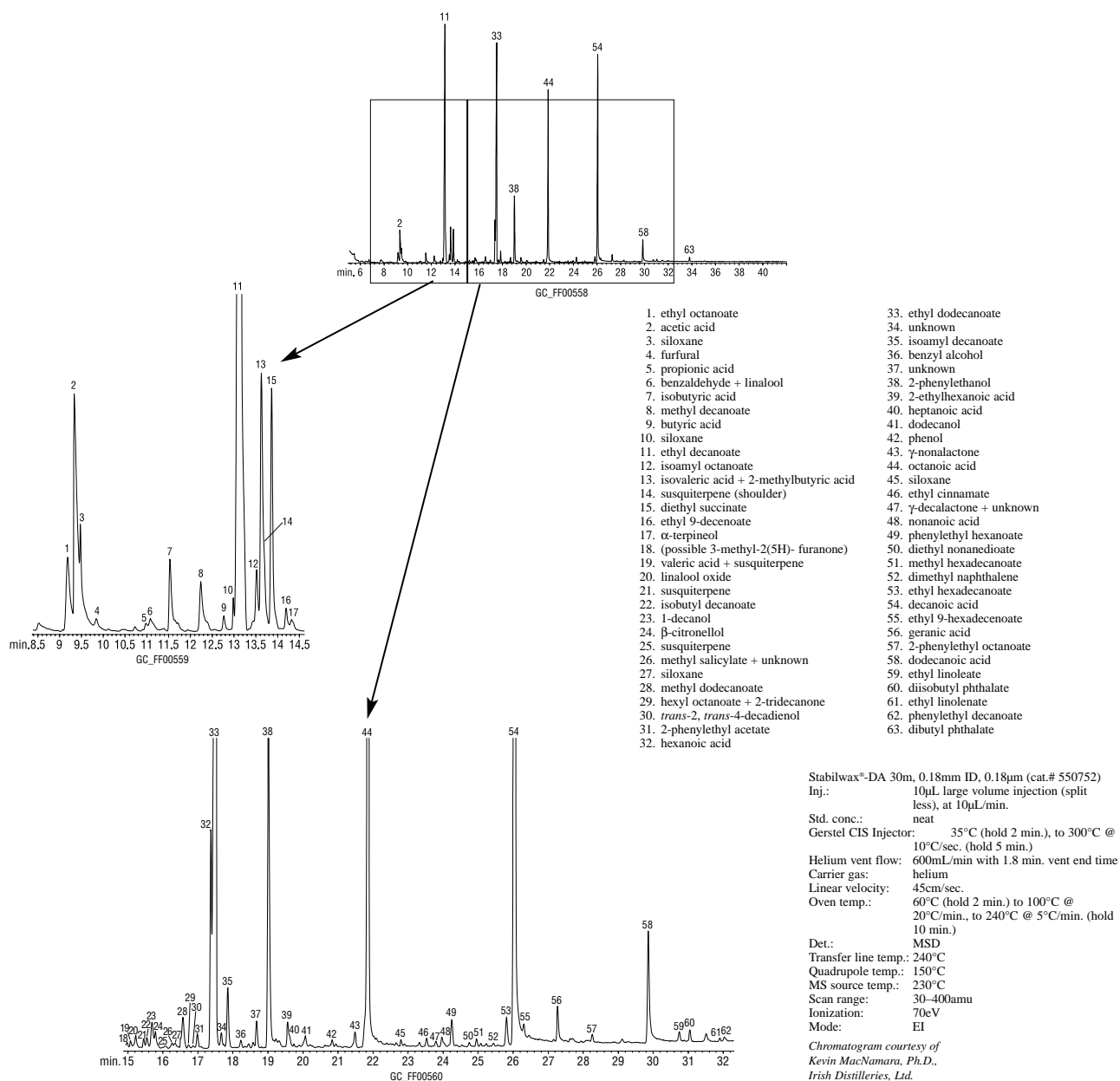
Malt whiskey profile, determined by GC/MS, using a Stabilwax®-DA column and a large volume injection technique.



Grappa is the spirit produced from grape marc, or the skins of the grapes after they have been pressed during wine production. Grape marc is fermented and distilled either directly or by water vapor. Grappas generally do not require the same amount of aging as other alcoholic beverages, although, for example, Italian law requires at least six months of aging. Flavored grappas can be produced by adding ingredients such as herbs and fruits. Flavor profiles of grappas contain hundreds of compounds at a wide range of concentrations. The chromatographic profile of an example grappa is shown in Figure 7.

Figure 7

Grappa profile, determined by GC/MS, using a Stabilwax®-DA column and a large volume injection.



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HOT tech tip

Sample, Transfer, and Analyze Sulfur Compounds at Parts-per-Billion Levels

Our exclusive Sulfinert™ process is the next generation of metals passivation treatments, developed specifically for deactivating metal surfaces that contact organo-sulfur compounds. Untreated stainless steel adsorbs or reacts with hydrogen sulfide, mercaptans, and other active sulfur-containing compounds. Applied to a stainless steel surface, a Sulfinert™ layer prevents these compounds, and other active compounds (e.g., amines), from contacting the reactive metal surface. Combine custom-deactivated sample storage and transfer components with stock Sulfinert™-treated parts to passivate your entire system, and obtain highly accurate information about sulfur compounds in your samples.

Additional Important Features

Durable and flexible - will not crack or flake.
Stable to 400°C.
No memory effects, as seen with polymeric surfaces.

Determining Trace Sulfur Compounds In Beer

Trace sulfur compounds that are generated during the fermentation process can affect the taste and aroma of malted products such as beers. Several common volatile sulfur compounds might be present in beer at ppb or ppm levels (Table 1).

Table 1

Volatile sulfur-containing compounds found in beer at ppm to ppb levels.

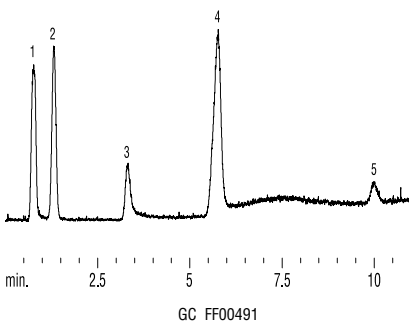
hydrogen sulfide	ethyl mercaptan	dimethyl disulfide
isopropyl mercaptan	t-butyl mercaptan	isobutyl mercaptan
carbonyl sulfide	sulfur dioxide	carbon disulfide
methyl ethyl sulfide	sec-butyl mercaptan	n-butyl mercaptan
methyl mercaptan	dimethyl sulfide	t-amyl mercaptan
n-propyl mercaptan	diethyl sulfide	

Accurate measurement of reactive sulfur compounds at these levels requires a highly inert chromatographic system. Restek's Rt-XLSulfur™ micropacked column contains a modified divinyl benzene polymer packed into Sulfinert™ tubing, and is specifically designed for monitoring ppb levels of active sulfur compounds. The Rt-XLSulfur™ column exhibits low bleed and thermal stability to 300°C. This column provides excellent resolution of hydrogen sulfide, carbonyl sulfide and sulfur dioxide.

Sample introduction into the column is a critical step in obtaining accurate analytical results for sulfur compounds. In this application, a beer headspace sample was introduced onto the column using a Valco six-port sampling valve fitted with a 1mL sample loop. The valve, sample loop, and all other surfaces in the sample pathway were deactivated using our Sulfinert™ deactivation process. The use of Sulfinert™-treated hardware is critical to achieving a 20ppb detection level for sulfur dioxide and the other target sulfur compounds (Figure 8).

Figure 8

Low levels of reactive sulfur compounds in CO₂ (i.e., 20ppb) easily can be detected using an Rt-XLSulfur™ micropacked column and a Sulfinert™ treated sample pathway.



1. hydrogen sulfide
2. carbonyl sulfide
3. methyl mercaptan
4. ethyl mercaptan and/or dimethyl sulfide
5. dimethyl disulfide

1m, 0.75mm ID Sulfinert™ tubing
Rt-XLSulfur™ 100/120 mesh (cat.# 19806)
Conc.: sulfur standard @ 20ppb each in CO₂
Inj.: 1cc sample loop, 6-port Valco® valve
Carrier gas: helium
Flow rate: 10mL/min. @ ambient temp.
Oven temp.: 60°C to 260°C @ 15°C/min. (hold 5 min.)
Det. sensitivity: SCD, attn. x 1
Det. temp.: 800°C

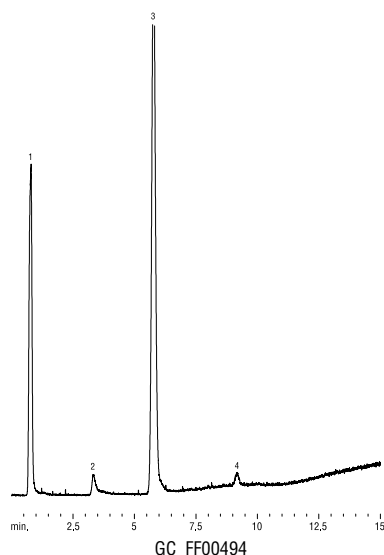
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We evaluated the effectiveness of the Rt-XLSulfur™ column by measuring trace sulfur compounds in one domestic (US) and two imported brands of beer. The results from headspace sampling of these products demonstrate the capability of the RT-XLSulfur™ column and the Sulfinert™ deactivated GC system to easily detect sulfur compounds at the 20ppb level (Figure 9).

Figure 9

ppb levels of hydrogen sulfide, dimethyl sulfide, and/or ethyl mercaptan and methyl mercaptan in beer.

Domestic Beer

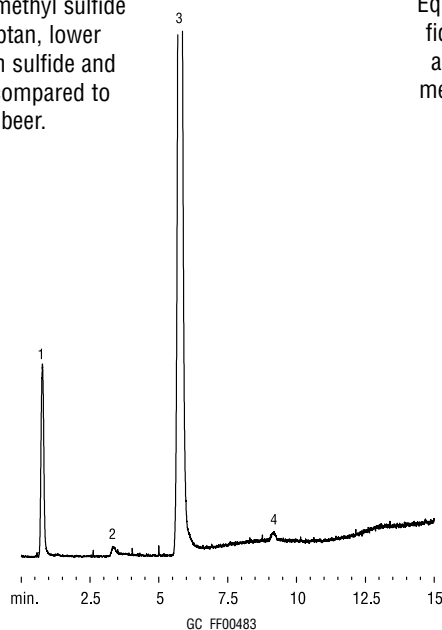


1. hydrogen sulfide
2. methyl mercaptan
3. dimethyl sulfide and/or ethyl mercaptan
4. unknown

1m, 0.75mm ID Sulfinert™ tubing
 Rt-XLSulfur™ 100/120 mesh (cat.# 19806)
 Conc.: headspace of a domestic (US) or imported beer sample
 Inj.: 1cc sample loop, 6-port Valco® valve
 Carrier gas: helium
 Flow rate: 10mL/ min. @ ambient temp.
 Oven temp.: 60°C to 260°C @ 15°C/min. (hold 5 min.)
 Det. sensitivity: SCD, attn. x 1
 Det. temp.: 800°C

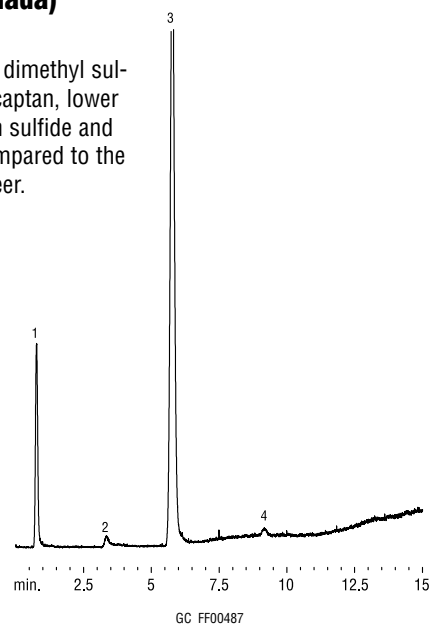
Imported Beer (Mexico)

Higher amounts of dimethyl sulfide and/or ethyl mercaptan, lower amounts of hydrogen sulfide and methyl mercaptan, compared to the domestic beer.



Imported Beer (Canada)

Equivalent amounts of dimethyl sulfide and/or ethyl mercaptan, lower amounts of hydrogen sulfide and methyl mercaptan, compared to the domestic beer.



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Summary

Gas chromatography is a simple, sensitive way to characterize the volatile compounds in alcoholic beverage products. Alcohols and aldehydes in alcoholic beverages can be analyzed by packed column GC or capillary GC, depending on the target analytes and their concentrations. Capillary GC provides very efficient separations, thereby resolving closely-related compounds, but the higher capacity of packed column GC systems sometimes makes it easier to detect trace levels of alcohols and short-chain aldehydes in the presence of high levels of ethanol. Ultimately, the choice of technique will depend on the needs of the analyst and the equipment available.

A Stabilwax®-DA capillary column is an excellent choice for analyses of acids, esters, and other flavor components in alcoholic beverage products. This highly stable column has been optimized for analyses of acidic compounds, making it possible to analyze a wide range of compounds. Large volume injection (LVI) techniques accommodate a wide range of concentrations in a single run. As shown in this guide, analytes at higher concentrations, such as alcohols and esters, and trace level flavor compounds can be analyzed simultaneously. The venting step during the large volume injection can be optimized to remove most of the ethanol/water matrix.

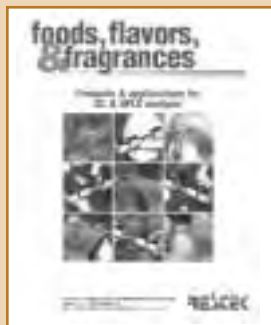
Low levels of reactive sulfur compounds in malted beverages also can be monitored reliably by gas chromatography. The combination of an Rt-XLSulfur™ micropacked column and a Sulfinert™ deactivated sample introduction system provides a state-of-the-art, robust, sampling and analysis approach for monitoring trace levels of volatile sulfur compounds in beer. This system also can be used to detect sulfur compounds in carbon dioxide used for artificial carbonation of carbonated beverages, such as soda waters and soft drinks. For information about this application, and example chromatograms, visit the following page on our website: www.restekcorp.com/advntage/d01four.htm

References

1. AOAC *Official Methods of Analysis* (2000), 17th edition, AOAC International.
2. Deman, *Principles of Food Chemistry* (1990), 2nd edition, Van Nostrand Reinhold, New York.

Additional Restek Literature

Foods Flavors Fragrances minicatalog



Lit. Cat. #59260

Preservatives by HPLC



Lit. Cat. #59398

Genuine Restek Replacement Parts



Lit. Cat. #59627C

Flavor Volatiles in Alcoholic Beverages



Lit. Cat. #59579

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Capillary Columns for Alcoholic Beverage Analysis

Ordering Information | Rtx®-1301 (G43) Capillary GC Columns (Fused Silica)

(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	temp. limits*	15-Meter	30-Meter	60-Meter
0.25mm	0.10	-20 to 280°C	16005	16008	16011
	0.25	-20 to 280°C	16020	16023	16026
	0.50	-20 to 270°C	16035	16038	16041
	1.00	-20 to 260°C	16050	16053	16056
0.32mm	1.40	-20 to 240°C			16016
	0.10	-20 to 280°C	16006	16009	16012
	0.25	-20 to 280°C	16021	16024	16027
	0.50	-20 to 270°C	16036	16039	16042
0.53mm	1.00	-20 to 260°C	16051	16054	16057
	1.50	-20 to 250°C	16066	16069	16072
	0.10	-20 to 280°C	16007	16010	16013
	0.25	-20 to 280°C	16022	16025	16028
	0.50	-20 to 270°C	16037	16040	16043
	1.00	-20 to 260°C	16052	16055	16058
	1.50	-20 to 250°C	16067	16070	16073
	3.00	-20 to 240°C	16082	16085	16088

Ordering Information | Stabilwax®-DA Capillary GC Columns (Fused Silica)

(Crossbond® Carbowax® for acidic compounds)

ID	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.18mm	0.18	40 to 250°C		550752	
0.25mm	0.10	40 to 250°C	11005	11008	11011
	0.25	40 to 250°C	11020	11023	11026
	0.50	40 to 250°C	11035	11038	11041
0.32mm	0.10	40 to 250°C	11006	11009	11012
	0.25	40 to 250°C	11021	11024	11027
	0.50	40 to 250°C	11036	11039	11042
	1.00	40 to 240/250°C	11051	11054	11057
0.53mm	0.10	40 to 250°C	11007	11010	11013
	0.25	40 to 250°C	11022	11025	11028
	0.50	40 to 250°C	11037	11040	11043
	1.00	40 to 240/250°C	11052	11055	11058
	1.50	40 to 230/240°C	11062	11065	11068

CarboBlack™ Solid Supports

Graphitized carbon black offers unique selectivity and very little adsorption for alcohol analyses. Two CarboBlack supports are available, CarboBlack™ B and CarboBlack™ C. CarboBlack™ B support, with its higher surface area, can support up to a 10% loading of a non-silicone liquid phase. CarboBlack™ C support can hold up to a 1% loading of a non-silicone liquid phase.

Ordering Information | CarboBlack™ Packed Columns

On CarboBlack™ B	Mesh	Stainless Steel Tubing				SilcoSmooth™ Tubing			
		L (ft.)	OD (in.)	ID (mm)	cat.#*	L (m)	OD (in.)	ID (mm)	cat.#*
5% Carbowax® 20M	80/120	—	—	—	—	2	1/8	2	80105-
5% Carbowax® 20M	60/80	6	1/8	2.1	88012-	1.8	1/8	2	80106-
6.6% Carbowax® 20M	80/120	6	1/8	2.1	80451-	2	1/8	2	80107-

* Please include configuration suffix number when ordering.

Leak Detective™ II Leak Detector*

- Affordable thermal conductivity leak detector—every analyst can have one.
- Compact, ergonomic design is easy to hold and operate with one hand.
- Helium, hydrogen, and nitrogen can be detected at 1x10⁻⁴cc/sec. or at an absolute concentration as low as 100ppm.**
- Fast results—responds in less than 2 seconds to trace leaks of gases with thermal conductivities different than air.
- Micro-chip design improves sensitivity and response timover previous models.
- Auto zeroing with the touch of a button.
- Battery-operated for increased portability (one 9-volt).



Description	qty.	cat.#
Leak Detective™ II Leak Detector	ea.	20413

*Never use liquid leak detectors on a capillary system because liquids can be drawn into the system.

**Caution: NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possibly hazardous conditions.

Configurations

	General Configuration Suffix -800
	Agilent 5880, 5890, 5987, 6890: Suffix -810
	Varian 3700, Vista Series, FID: Suffix -820
	PE 900-3920 Sigma 1,2,3: Suffix -830
	PE Auto System 8300, 8400, 8700 (Not On-Column): Suffix -840

See our catalog for custom configurations

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Micropacked Columns

- Higher efficiency than packed columns.
- Higher capacity than capillary columns.
- Made from inert, flexible Silcosteel® tubing.

Micropacked columns are inexpensive, rugged, and easy to install and to operate. With our inert Silcosteel® treatment, micropacked columns are a powerful tool for solving many difficult application problems. Because the Silcosteel® coating is thin, the column can be flexed and coiled without any fear of damage to the inert surface.

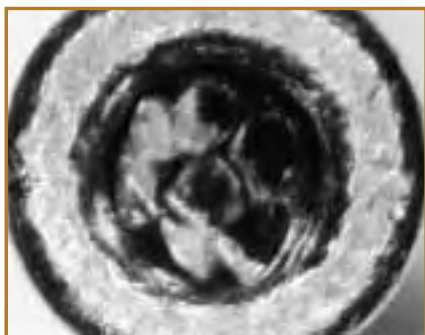
Micropacked columns fit packed or capillary injection systems. 1mm ID, (1/16-inch OD) micropacked columns improve efficiency of packed column instruments, without the expense of converting to a capillary injection system. 0.75mm ID (0.95mm OD) micropacked columns install easily into a capillary injector, using slightly larger ferrules. Micropacked columns operate at flows exceeding 10cc/min., for trouble-free operation. Packed with 100/120 mesh particles.

Ordering Information | Rt-XLSulfur™ Micropacked Columns

Purchase installation kit separately.

OD	ID (mm)	1-Meter	2-Meter
1/16"	1.0mm	19804	19805
0.95mm	0.75mm	19806	19807

HOT tech tip



A common problem with micropacked columns is the integrity of the end plug. Glass wool is difficult to insert into an opening less than 1mm wide and can be dislodged easily by carrier gas pressure surges that occur during valve switching. Restek's chemists insert braided wire into the column bore, then make a small crimp near the column outlet. End plugs are Silcosteel®-treated to ensure that the sample contacts only inert surfaces.

Ordering Information | Micropacked Columns Installation Kits

	for 0.75mm ID col.	for 1mm ID col.	for 2mm ID col.
For valve applications	21062	21065	21067
For split applications	21063	—	—
For all Agilent GCs	21064	—	—
For direct injections	—	21066	—

Headspace Vials



6.0mL Headspace Vial

Headspace Autosampler Vials

Description	100-pk.	1000-pk.
6mL Clear Vial	21166	21167
10mL Clear Vial, Flat Bottom	24683	24684
10mL Clear Vial, Rounded Bottom	21164	21165
20mL Clear Vial, Flat Bottom	24685	24686
20mL Clear Vial, Rounded Bottom	21162	21163
27mL Clear Vial	21160	21161



Silver Seal with
PTFE/Gray Butyl
Rubber Septum

20mm Aluminum Seals w/Septa, Assembled

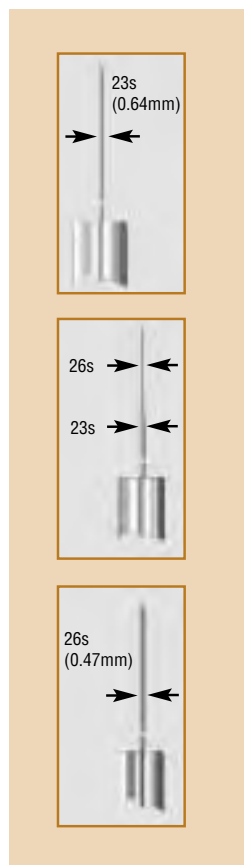
Description	100-pk.	1000-pk.
Silver Seal w/ PTFE/Gray Butyl Rubber	21761	21762
Silver Seal w/ PTFE/Silicone	21763	21764
Pressure Release Silver Seal w/ PTFE/Gray Butyl Rubber Septum <125°C	21765	21766
Pressure Release Silver Seal w/ PTFE/Silicone Septum >125°C	21767	21768

Syringes

Restek offers complementing lines of syringes from Hamilton & SGE.

- Hamilton: The historical leader in precision fluid measuring devices for over 40 years, with a commitment to precision, quality, and accuracy.
- SGE: Over 25 years of providing a comprehensive range of analytical syringes unsurpassed in design, quality, and performance.

Needle Gauge for Agilent 7673 Syringes



23s—Single Gauge Needle

- The most popular gauge for Agilent 7673.
- Stocked for same-day shipment.
- Best for Merlin Microseal® septum and standard septum-equipped GCs.
- Packed column injection ports.
- Split/splitless injection ports.

SuperfleX™ Flexible Plunger Syringe

Agilent 7673 Syringe

Gas-Tight Luer-lock Syringe

23s-26s—Dual Gauge (tapered) Needle

- Durability of a 23s gauge needle.
- Ability of a 26s gauge needle to perform split/splitless and on-column injections.

SGE Removable Needle for Agilent 7673 Autosampler

26s—Single Gauge Needle

- On-column injection ports.
- Split/splitless injection ports.

Hamilton 10µL, Autosampler Cemented Needle for Agilent 7673 Autosampler

Drawings reproduced with permission from Hamilton.

Hamilton Syringes

Volume	Needle Term.	Needle Gauge	Needle Length	Point Style	Hamilton		Restek	
					Model	cat.#	qty.	cat.#
5µL	ASN*	23s	1.71"	Agilent	75	87990	6-pk.	20170
5µL	ASN	26s	1.71"	Agilent	75	87989	6-pk.	21230
5µL	ASN	23s-26s	1.71"	Agilent	75	87994	6-pk.	24594
10µL	ASN	23s	1.71"	Agilent	701	80390**	6-pk.	20169
10µL	ASN	26s	1.71"	Agilent	701	80389	6-pk.	24599
10µL	ASN	23s-26s	1.71"	Agilent	701	80391	6-pk.	24600

* Autosampler cemented needle.

** Designated by Agilent as #80397.

SGE Syringes

Volume	Needle Term.	Needle Gauge	Needle Length	Point Style	SGE		Restek	
					Model	cat.#	qty.	cat.#
5µL	F*	23	42mm	Cone	SK-5F-HP-0.63	001814	6-pk.	24783
5µL	F	26	42mm	Cone	SK-5F-HP-0.47	001804	6-pk.	24782
5µL	F	23-26s	42mm	Cone	SK-5F-HP-0.63/0.47	001822	6-pk.	21214
10µL	F	23	42mm	Cone	SK-10F-HP-0.63	002814	6-pk.	24787
10µL	F	26	42mm	Cone	SK-10F-HP-0.47	002804	6-pk.	24786
10µL	F	23-26s	42mm	Cone	SK-10F-HP-.063/0.47	002822	6-pk.	21215

* Fixed needle.

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Siltek™ Deactivation—The Next Generation

- Maximizes the inertness of the sample pathway.
- Minimizes breakdown.
- Low bleed.
- Thermally stable.
- “Clean and green”—manufactured without the use of harmful organic solvents.

Restek offers the next generation of deactivation. The Siltek™ deactivation process (patent pending) produces a highly-inert glass surface, which features high temperature stability, extreme durability, and low bleed. Try Siltek™ liners, guard columns, wool, and connectors for better recovery of sample analytes.

For Siltek™ inlet liners, add the corresponding suffix number to your liner catalog number.







Siltek™ Inlet Liners

qty.	Siltek™	Siltek™ with Siltek™ wool	Siltek™ with CarboFrit™
each	-214.1	-213.1	-216.1
5-pk.	-214.5	-213.5	-216.5
25-pk.	-214.25	-213.25	-216.25




Benefits of wool-packed Precision™ Liners

- Wool minimizes vaporization and helps wipe the needle during injection.
- No guessing where the wool should be placed; easy to change wool.
- Wool stays in position during pressure pulses in the inlet during an injection.
- 100% deactivation ensures inertness.*

* Not Siltek™ deactivation.

Precision™ Liners	ID**/OD & Length (mm)	ea.	5-pk.	25-pk.
 Agilent 4mm Split Precision™ Liner	4.0 ID 6.3 OD x 78.5	21022	21023	20979
 Varian 1078/1079 Split Precision™ Liner	3.4 ID 5.0 OD x 54	21024	21025	
 Shimadzu 17A Split Precision™ Liner	3.5 ID 5.0 OD x 95	21020	21021	
 Varian 1075/1077 Split Precision™ Liner	4.0 ID 6.3 OD x 72	21030	21031	
 Thermo Finnigan 5mm Split Precision™ Liner	5.0 ID 8.0 OD x 105	21028	21029	
 PerkinElmer Auto SYS Split Precision™ Liner	4.0 ID 6.2 OD x 92.1	21026	21027	

Inlet Liners for APEX ProSep™ 800 & ProSep™ 800 Plus GCs

	Benefits/Uses:	ID**/OD & Length (mm)	Similar to APEX part #	cat.# ea.
 Mega IV (4.0mm ID)	injections <125µL	4.0 ID 6.0 OD x 243	L-00410	21075
 Micro I (1.0mm ID)	injections <5µL	1.0 ID 6.0 OD x 243	L-00110	21073
 MIDI II (2.0mm ID)	injections <25µL	2.0 ID 6.0 OD x 243	L-00210	21074

**Nominal ID at syringe needle expulsion point.

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Vespel® Ring Inlet Seals for Agilent 5890/6890 and 6850 GCs

- Easy-to-use, patent-pending design saves time.
- Vespel® material seals the first time, every time.
- Very little torque is required to make a seal—reduces operator variability.
- Lower leak rate versus OEM metal inlet seals—reduces detector noise.
- Increases column lifetime by preventing oxygen from leaking into the carrier gas.
- Soft sealing area reduces wear on the critical seal of the injection port base.

Vespel® ring ensures a leak-tight seal the first time, every time.



0.8mm ID Vespel® Ring Inlet Seal	2-pk.	10-pk.
Gold-Plated	21562	21563
Silcosteel®	21564	21565
Stainless Steel	21560	21561
1.2mm ID Vespel® Ring Inlet Seal	2-pk.	10-pk.
Gold-Plated	21568	21569
Silcosteel®	21570	21571
Stainless Steel	21566	21567

Washers included.

Replacement Inlet Seals for Agilent 5890/6890/6850 Split/Splitless Injection Ports

- Special grade of stainless steel that is softer and deforms more easily, ensuring a completely leak-tight seal.
- Increases column lifetime because oxygen cannot leak into the carrier gas.
- Reduced noise benefits high-sensitivity detectors (e.g., ECDs, MSDs).
- Silcosteel® seal offers the inertness of glass.



Single-Column Installation, 0.8mm Opening*		0.25/0.32mm ID Dual-Column Installation, 1.2mm Opening		0.53mm ID Dual-Column Installation 1/16-inch Opening	
2-pk.	10-pk.	2-pk.	10-pk.	2-pk.	10-pk.
Stainless Steel Inlet Seal					
21315	21316	20390	20391	20392	20393
Gold-Plated Inlet Seal					
21317	21318	21305	21306	—	—
Silcosteel® Inlet Seal					
21319	21320	21307	21308	—	—

**0.8mm ID stainless steel inlet seal is equivalent to Agilent part #18740-20880.*

0.8mm ID gold-plated inlet seal is equivalent to Agilent part #18740-20885.

Note: All seals include washers.

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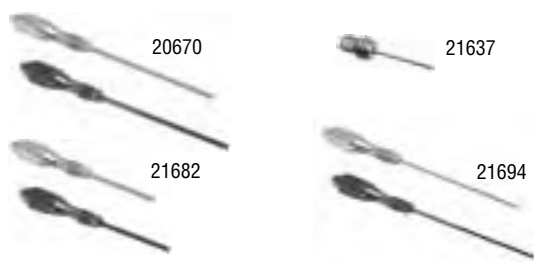
Replacement FID Jets

- Standard Version: Engineered with a fluted tip to guide the capillary column into the jet.
- High-Performance Version: Identical to the standard version, except that it has been Silcosteel®-treated. Extremely inert, use with active compounds.

Capillary Adaptable FID Jet for Agilent 5890/6890/6850 GCs (0.011-inch ID tip)

(Similar to Agilent part # 19244-80560.)

Description	qty.	cat.#	qty.	cat.#
Standard	ea.	20670	3-pk.	20671
High-Performance Silcosteel®	ea.	20672	3-pk.	20673



Capillary Dedicated FID Jet for Agilent 6890/6850 GCs

(Similar to Agilent part # G1531-80560.)

Description	qty.	cat.#	qty.	cat.#
Standard	ea.	21621	3-pk.	21682
High-Performance Silcosteel®	ea.	21620	3-pk.	21683

Capillary FID Jet for Agilent 5880 GCs

(Similar to Agilent part # 19301-80500.)

Description	qty.	cat.#
Standard	ea.	21637
	ea.	21638

Packed Column FID Jets for Agilent 5890/6890/6850 GCs

0.018-Inch ID

(Similar to Agilent part # 18710-20119.)

	qty.	cat.#	qty.	cat.#
Standard	ea.	21694	3-pk.	21695
	ea.	21696	3-pk.	21697

0.030-Inch ID

(Similar to Agilent part # 18789-80070.)

	qty.	cat.#	qty.	cat.#
Standard	ea.	21688	3-pk.	21689
	ea.	21686	3-pk.	21687

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A Technical Guide for Static Headspace Analysis Using GC



Inside:

*Basic Principles of Headspace
Analysis*

Instrumentation

*System Optimization
(Troubleshooting)*

Headspace Applications

*Recommended Headspace Analysis
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Static headspace gas chromatography (GC) is a technique used for the concentration and analysis of volatile organic compounds. This technique is relatively simple and can provide sensitivity similar to dynamic purge and trap analysis. The popularity of this technique has grown and has gained worldwide acceptance for analyses of alcohols in blood and residual solvents in pharmaceutical products. Other common applications include industrial analyses of monomers in polymers and plastic, flavor compounds in beverages and food products, and fragrances in perfumes and cosmetics.

Sample matrices like blood, plastic, and cosmetics contain high molecular weight, non-volatile material that can remain in the GC system and result in poor analytical performance. Many laboratory analysts use extensive sample preparation techniques to extract and concentrate the compounds of interest from this unwanted non-volatile material. These extraction and concentration techniques can become time consuming and costly. Static headspace analysis avoids this time and cost by directly sampling the volatile headspace from the container in which the sample is placed.

Because of the diversities in the industry and related products, this guide attempts to cover only the basic principles of static headspace and demonstrate how to apply them to achieve optimum chromatographic results. With an understanding of these principles, various instrumentation will then be reviewed to help build upon this knowledge and identify the benefits and potential problems associated with each mode of sample transfer. Information from the *Basic Principles* and *Instrumentation* sections of this guide can then be brought together and applied to the conditions and methodology of common analyses. Like most applications, a variety of problems may arise in which the *System Optimization* section will help to identify these problems and offer techniques to help resolve them.

Time and money are two of the many reasons why an analyst would use static headspace analysis. Other reasons may include ease of operation and the ability to assay a variety of sample matrices.



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Basic Principles of Headspace Analysis

Most consumer products and biological samples are composed of a wide variety of compounds that differ in molecular weight, polarity, and volatility. For complex samples like these, headspace sampling is the fastest and cleanest method for analyzing volatile organic compounds. A headspace sample is normally prepared in a vial containing the sample, the dilution solvent, a matrix modifier, and the headspace (see **Figure 1**). Volatile components from complex sample mixtures can be extracted from non-volatile sample components and isolated in the headspace or vapor portion of a sample vial. An aliquot of the vapor in the headspace is delivered to a GC system for separation of all of the volatile components.

In order to achieve the best performance when using headspace/GC, careful attention should be used in sample preparation and instrument setup. Key issues to address when setting up headspace/GC systems include minimizing system dead volume, maintaining inert sample flow paths, and achieving efficient sample transfer. These issues, as well as other instrument setup-related topics, are addressed later in the *System Optimization* section of this guide.

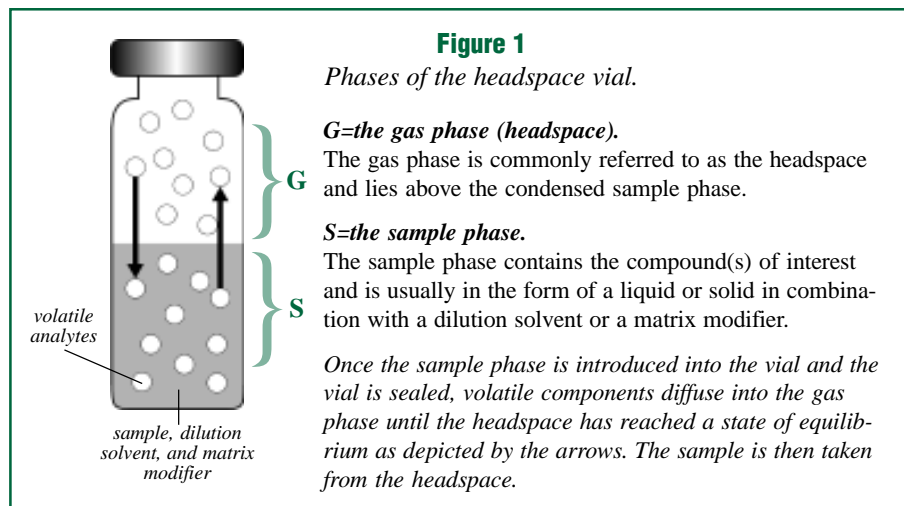


Figure 2
K and β are important variables in headspace analysis.

Equation 1
Partition Coefficient (K) = C_g/C_s

Equation 2
Phase Ratio (β) = V_g/V_s

C_s =concentration of analyte in sample phase
 C_g =concentration of analyte in gas phase
 V_s =volume of sample phase
 V_g =volume of gas phase

Partition Coefficient

Samples must be prepared to maximize the concentration of the volatile components in the headspace, and minimize unwanted contamination from other compounds in the sample matrix. To help determine the concentration of an analyte in the headspace, you will need to calculate the partition coefficient (K), which is defined as the equilibrium distribution of an analyte between the sample phase and the gas phase (**Figure 2**).

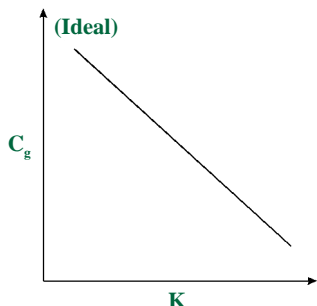
Compounds that have low K values will tend to partition more readily into the gas phase, and have relatively high responses and low limits of detection (**Figure 3**). An example of this would be hexane in water: at 40°C, hexane has a K value of 0.14 in an air-water system. Compounds that have high K values will tend to partition less readily into the gas phase and have relatively low response and high limits of detection. An example of this would be ethanol in water: at 40°C, ethanol has a K value of 1355 in an air-water system. Partition coefficient values for other common compounds are shown in **Table I**.

Table I
K Values of Common Solvents in Air-Water Systems at 40°C

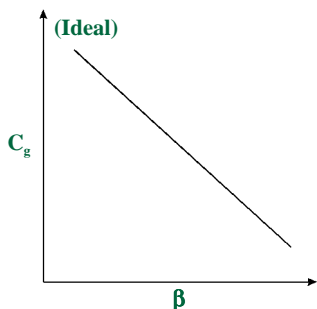
Solvent	K Value
cyclohexane	0.077
n-hexane	0.14
tetrachloroethylene	1.48
1,1,1-trichloromethane	1.65
o-xylene	2.44
toluene	2.82
benzene	2.90
dichloromethane	5.65
n-butyl acetate	31.4
ethyl acetate	62.4
methyl ethyl ketone	139.5
n-butanol	647
isopropanol	825
ethanol	1355
dioxane	1618

Figure 3

Sensitivity is increased when K is minimized.

**Figure 4**

Sensitivity is increased when β is minimized.



K can be lowered by changing the temperature at which the vial is equilibrated or by changing the composition of the sample matrix. In the case of ethanol, K can be lowered from 1355 to 328 by raising the temperature of the vial from 40°C to 80°C. It can be lowered even further by introducing inorganic salt into the aqueous sample matrix. High salt concentrations in aqueous samples decrease the solubility of polar organic volatiles in the sample matrix and promote their transfer into the headspace, resulting in lower K values. However, the magnitude of the salting-out effect on K is not the same for all compounds. Compounds with K values that are already relatively low will experience very little change in partition coefficient after adding a salt to an aqueous sample matrix. Generally, volatile polar compounds in polar matrices (aqueous samples) will experience the largest shifts in K and have higher responses after the addition of salt to the sample matrix. **Table II** lists most of the common salts used for salting-out procedures.

Table II

Common salts used to decrease matrix effects.

ammonium chloride
ammonium sulfate
sodium chloride
sodium citrate
sodium sulfate
potassium carbonate

Phase Ratio

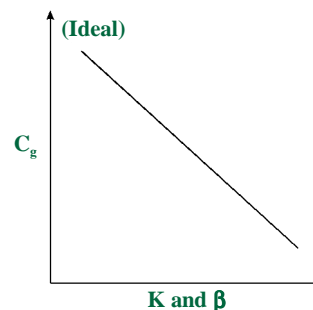
The phase ratio (β) is defined as the relative volume of the headspace compared to volume of the sample in the sample vial (**Figure 2**). Lower values for β (i.e., larger sample size) will yield higher responses for volatile compounds (**Figure 4**). However, decreasing the β value will not always yield the increase in response needed to improve sensitivity. When β is decreased by increasing the sample size, compounds with high K values partition less into the headspace compared to compounds with low K values, and yield correspondingly smaller changes in C_g . Samples that contain compounds with high K values need to be optimized to provide the lowest K value before changes are made in the phase ratio.

Combining K and β

Partition coefficients and phase ratios work together to determine the final concentration of volatile compounds in the headspace of sample vials. The concentration of volatile compounds in the gas phase can be expressed as $C_g = C_o / (K + \beta)$ (where C_g is the concentration of volatile analytes in the gas phase and C_o is the original concentration of volatile analytes in the sample). Striving for the lowest values for both K and β will result in higher concentrations of volatile analytes in the gas phase and, therefore, better sensitivity (**Figure 5**).

Figure 5

Lower K and β result in higher C_g and better sensitivity.



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Derivatization/Reaction Headspace

Derivatization is another technique that can be used to increase sensitivity and chromatographic performance for specific compounds. Compounds such as acids, alcohols, and amines are difficult to analyze because of the presence of reactive hydrogens. When attempting to analyze these types of compounds, they can react with the surface of the injection port or the analytical column and result in tailing peaks and low response. In addition, they may be highly soluble in the sample phase, causing very poor partitioning into the headspace and low response. Derivatization can improve their volatility, as well as reduce the potential for surface adsorption once they enter the GC system.

Common derivatization techniques used in reaction headspace/GC are esterification, acetylation, silylation, and alkylation. Any of these derivatization techniques can be performed using the sample vial as the reaction vessel (see **Table III** for a list of commonly used reagents). Although derivatization may improve chromatographic performance and volatility for some compounds, derivatization reactions may introduce other problems into the analytical scheme. Derivatization reagents as well as the by-products from derivatization reaction may be volatile and can partition into the headspace along with derivatized compounds. These extra volatile compounds may pose problems by eluting with similar retention times as the compounds of interest, causing either partial or complete coelutions.

Derivatization reactions also are typically run at elevated temperatures. Pressures inside the sample vial may exceed the pressure handling capabilities of the vial or the septa. Specially designed septa are available that allow excess pressure to be vented during derivatization reactions.

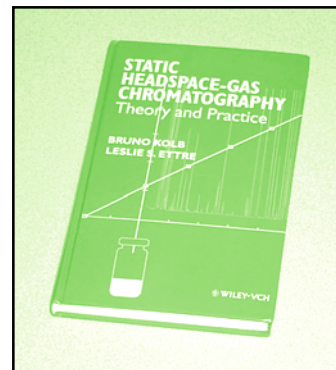
Table III

Common reagents used to derivatize compounds of interest.

Compound of Interest	Derivatizing Reagent	Resulting Derivative
fatty acids	methanol with boron trifluoride	esterification
glycerol	acetic anhydride with sodium carbonate	acetylation

For more information on derivatization, please refer to the "Handbook of Analytical Derivatization Reactions" by Daniel R. Knapp or to the text at right.

For more information on headspace analysis, check out the textbook,
Static Headspace-Gas Chromatography, Theory and Practice
by Bruno Kolb and Leslie S. Ettre.



Headspace Sample Size

In addition to working with K , β , and derivatization reactions, sensitivity also can be improved by simply increasing the size of the headspace sample that is withdrawn from the sample vial and transferred to the GC. Increasing the sample size also means that the amount of time it takes to transfer the sample to the column will increase in proportion to the column volumetric flow rate. Sample size can be increased only to the point that increases in peak width, as a result of longer sample transfer times, will not affect chromatographic separations. Larger sample sizes and longer transfer times can be offset by using cryogenic cooling and sample refocusing at the head of the column.

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Instrumentation

Gas-Tight Syringe Injection

Use of a gas-tight syringe autosampling system is one of three common techniques (gas-tight syringe, balanced pressure, and pressure loop) used to transfer a headspace sample. Most of the autosampling units can retrofit to a standard GC with a split/splitless injection port, making them relatively simple to use and understand. These systems do not require the use of special configurations or special instrumentation other than the autosampler itself. The gas-tight syringe autosampler is beneficial for use with diverse samples because of the variety of sampler configurations and method options available.

The gas-tight syringe technique operates by initially thermostating the sample in an incubation oven at a given temperature and for a given time until it has reached a state of equilibrium (**Figure 6, Step 1**). Once the sample has reached an equilibrium, an aliquot is taken from the headspace using the gas-tight syringe (**Figure 6, Step 2**), and the aliquot is injected into the GC as if it were a liquid sample injection (**Figure 6, Step 3**).

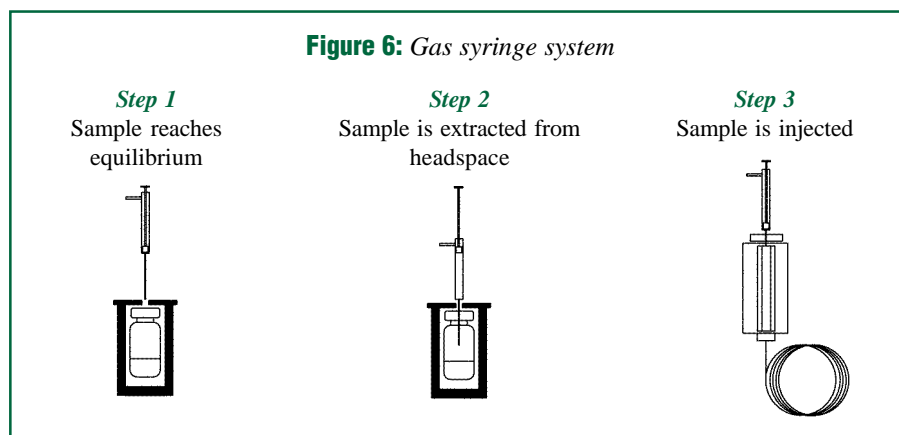
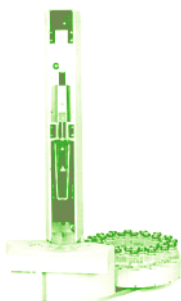


Figure 7

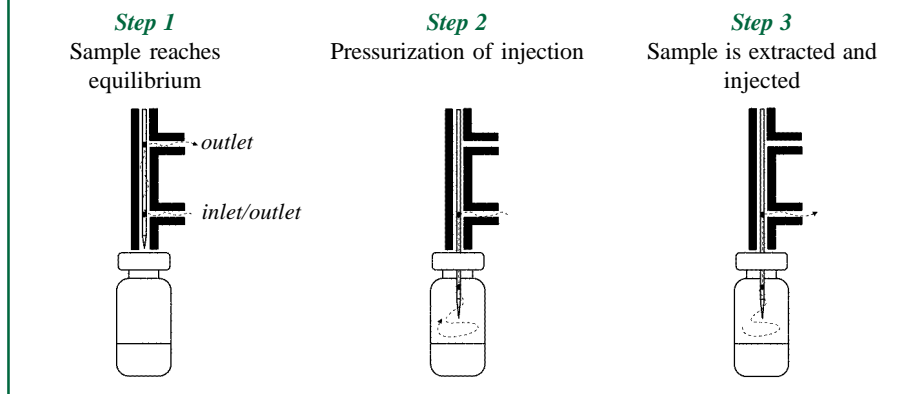
Gas-tight syringe autosampler
TRACE HS850



Several concerns exist regarding this technique. Because the sample is being transferred from a heated oven, the syringe also must be heated to ensure that the sample will not recondense in the syringe. Many manufacturers have taken this into consideration and their samplers now come with a heated syringe assembly. There also are reproducibility issues because of possible sample loss. As the sample is transferred from the vial to the injection port, some of it may be lost because of the pressure differences between the vial and atmospheric conditions. Beyond these concerns, the gas-tight syringe technique is simple to use, can retrofit into a variety of GC systems, and is best suited for diverse samples. Examples of manufacturers and models of the gas-tight syringe units are: the ThermoQuest TRACE™ HS2000 and HS850 (**Figure 7**) Headspace Autosamplers and the Leap Technologies CTC COMBI PAL Sampler.

Balanced-Pressure System

Another common technique is the balanced-pressure system, which is capable of generating results with a high degree of repeatability. It uses a seamless injection directly from the vial into the carrier gas stream without additional moving parts other than a valve and a needle. The balanced-pressure system, like other techniques, uses an incubation oven to thermostat the vial so the sample reaches equilibrium (**Figure 8, Step 1**). During these initial steps, a needle is inserted into the vial and then is pressurized with a carrier gas (**Figure 8, Step 2**). After the vial is pressurized and equilibrium has been reached, the valve is switched for a specific amount of time to redirect the sample into the transfer line and onto the column (**Figure 8, Step 3**).

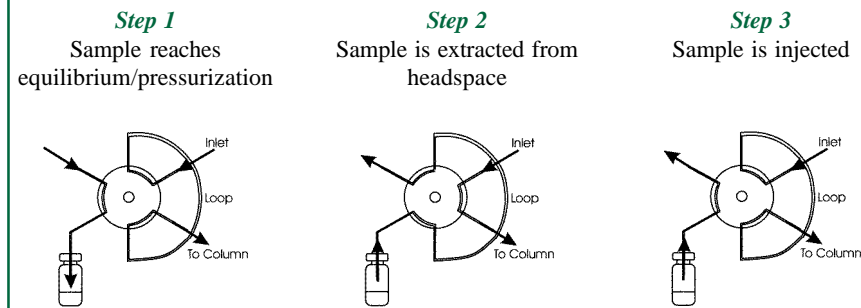
Figure 8: Balanced-pressure system

Because this technique uses a theoretical amount of time to inject the sample, the absolute volume of the sample is unknown. However, this technique is highly reproducible because the number of moving parts are minimized, which decreases the chance for compound adsorption and loss via leaks. An example of a balanced-pressure system is the HS 40XL manufactured by Perkin-Elmer (**Figure 9**).

Figure 9
Balanced-pressure autosampler Perkin-Elmer HS 40XL

Pressure-Loop System

The last common injection technique discussed in this guide is the pressure-loop system. Unlike balanced-pressure, the pressure-loop system uses a known amount of sample. This technique typically uses a six-port valve, and initially thermostats and pressurizes the vial as in the previously described techniques (**Figure 10, Step 1**). After pressurization, the valve is turned and the loop is filled with the sample (**Figure 10, Step 2**). After the loop has been filled, the valve is turned again to redirect the gas flow and flush the sample into the transfer line leading to the analytical column (**Figure 10, Step 3**).

Figure 10: Pressure-loop system

The pressure-loop system has several advantages and disadvantages. One of the advantages of this system is that the loop can be thermostatted to high temperatures, which helps to lessen adsorption of higher molecular weight and sensitive compounds. The fixed volume of the sample loop also helps to improve run-to-run reproducibility. A disadvantage of a pressure-loop system is that it may cause ghost peaks because of sample carryover from a previous analysis.¹ Several makes and models of pressure-loop systems include the OI Model 4632 (**Figure 11**), Varian Genesis, Tekmar 7000HT, and the HP 7694E.

Figure 11
Pressure-loop system OI Model 4632

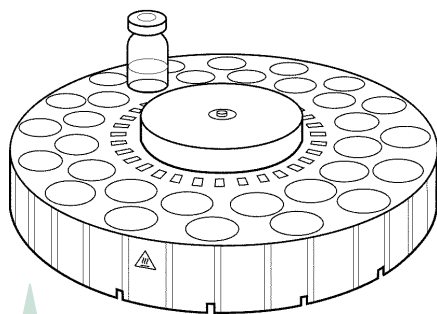
System Optimization (Troubleshooting)

Chromatographic performance in Headspace/GC is greatly influenced by how the sample is introduced into the analytical column. Variables that affect sample preparation and transfer of the sample from the headspace unit to the analytical column must be optimized to obtain reproducible and efficient separations. Key issues to address when setting up headspace/GC systems include minimizing system dead volume, maintaining inert sample flow paths, and achieving efficient sample transfer. This section will explain how to optimize areas that are critical in addressing these issues and providing good chromatographic performance.

Sample Preparation

Samples for headspace/GC must be prepared in such a manner as to maximize the concentration of the volatile sample components in the headspace while minimizing unwanted contamination from other compounds in the sample matrix. Sample matrices such as biological samples, plastics, and cosmetics can contain high molecular weight, volatile material that can be transferred to the GC system. Water from the sample matrix also can cause problems by recondensing in the transfer line. Incomplete or inefficient transfer of high molecular weight compounds or water vapor from sample matrices can produce adsorptive areas in the transfer line or injection port that can lead to split peaks, tailing peaks, or irreproducible responses or retention times. To minimize matrix problems and prevent water condensation from aqueous samples, use a higher transfer line temperature (~125°C–150°C).

High-concentration samples need to be prepared appropriately to obtain optimal chromatography. High-concentration samples can produce ghost peaks in subsequent analyses due to carryover of sample from previous injections. Sample carryover can be minimized by using higher transfer line and injection port temperatures, but some samples may need to be diluted and re-analyzed to obtain reliable results. Additionally, we recommend injecting standards and samples in order from low to high concentrations to help minimize carryover. When sample carryover or ghost peaks are evident, you may need to bake-out the column at its maximum operating temperature and elevate the transfer line temperature in order to remove all of the residual sample. If high-concentration samples are anticipated in a sequence of samples, running a blank after the suspected samples will reduce carryover contamination of following ones. It is good lab practice to handle standards and method blanks the same way samples are handled to make any vial or sample preparation problems easier to identify.



Always use pre-cleaned vials for sample preparation and storage.

Sample Vial

Sample vials should be selected to match the type and size of the sample being analyzed. Always use pre-cleaned vials for sample preparation and storage. Vials that are not properly cleaned prior to packaging or that absorb contaminants during shipping can produce unknown chromatographic peaks, or “ghost peaks.” Ghost peaks that are the result of vial contamination can be identified by running method blanks and zero standards during the system calibration sequence.

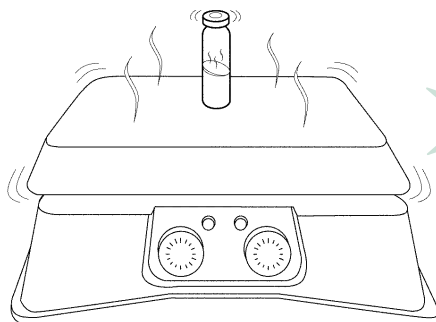
The septa used to seal the headspace vials also can be a source for contaminants, which can bleed into the headspace of the vial during equilibration. These contaminants can appear as single peaks or multiple peak patterns. Some septa are available with a Teflon® face to eliminate bleed from the rubber portion of the septa. These septa should not be re-used. Once the Teflon® face has been punctured by a syringe, contaminants from the rubber portion of the septa can migrate into the headspace and show up as unidentified peaks. Again, the use of method blanks can help to determine the source of contaminants.

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Sample Vial Heater and Mixer

Once the sample is placed inside a clean, non-contaminating vial and the vial is sealed, volatile compounds from the sample will partition into the headspace until a state of equilibrium is reached. The rate at which volatile compounds partition out of the sample matrix and into the headspace, as well as the equilibrium concentration of volatile compounds in the headspace depends on several parameters (see also *Introduction* of this guide).

Temperature, time, and mixing can be used to improve the transfer of volatile analytes from the sample into the headspace of the vial. Adjusting the temperature of the sample will change the solubility of the analyte in the sample matrix and can be used to drive the equilibrium in favor of the gas phase. Sufficient time must be built into the sample cycle in order to achieve a constant state of equilibrium. Some sample matrices require longer equilibration times due to physical characteristics like high viscosity. Shaking or vibrating the vial during heating can assist in achieving equilibrium more quickly by exposing more sample surface area for the transfer of volatile analytes to the headspace.



Shaking or vibrating the vial during heating can assist in achieving equilibrium.



Sampling

There are several techniques used to transfer samples from the vial to the GC. When using a *gas-tight syringe* for sampling, heat the syringe to a temperature comparable to the sample vial temperature. This minimizes pressure differences and condensation problems. To prevent carryover from inside the syringe, flush the syringe after each injection. Because gas-tight syringe samplers inject through the GC injection port septum, ensure the septum is well maintained to decrease the possibility of a leak.

For *balanced-pressure sampling* instruments, analysts should consider the inertness and efficiency of the components that make up the sample pathway inside the autosampler. If sensitive compounds are being analyzed, an inert pathway should be used to decrease possible adsorption. Materials such as stainless steel, nickel, Silcosteel® and Teflon® coatings, or KEL-F® parts can be used to minimize sample adsorption and peak tailing. Transfer line internal diameter should be as narrow as possible to help maintain narrow sample band widths and symmetrical peak shapes (see the following optimization of transfer lines for more information). Analysts also should ensure that balanced-pressure instruments are leak-free and operate with the least amount of dead volume in the sample flow path. This will help obtain optimal peak shape and sensitivity.

When using *pressure-loop sampling* instruments, the same concerns apply as with gas-tight syringe and balanced-pressure systems. Inert sample pathways and low dead volume systems will yield the best chromatographic performance. In pressure-loop systems, a gas sampling valve with a sample loop is used to transfer the sample from the headspace unit to the GC. Adequate purging of the sample valve and loop will guard against sample carryover. If low response or broad peaks are observed, it may be necessary to increase the sample vial pressure to ensure that the sample loop is being completely filled with headspace sample. If there are extraneous peaks present due to carryover of matrix contaminants, increase the sample valve temperature to prevent sample carryover, condensation, and contamination.

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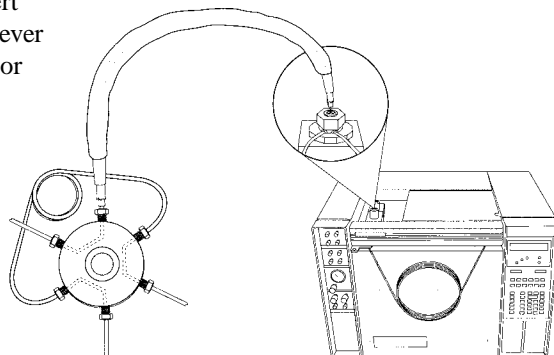
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Transfer Line

After the headspace sample is withdrawn from the vial, it is ready to be transferred to the GC. In balanced-pressure and pressure-loop systems a short piece of tubing called a transfer line is used to transfer the sample from the autosampler to the GC. Transfer line material must be chosen that suits the sample analytes. Many different materials can be used as transfer line tubing, including stainless steel, nickel, fused silica, and Silcosteel®- or Siltek™-coated tubing. Stainless steel provides a strong, flexible tubing material, but can be adsorptive towards more active analytes such as alcohols, diols, and amines. Nickel and Silcosteel® tubing are highly inert towards active compounds and provide ruggedness similar to stainless steel. Fused silica and Siltek™ tubing are extremely inert towards active compounds, however they are not as rugged as nickel or Silcosteel® tubing.

Use an inert transfer line when optimizing pressure-loop systems.

The internal diameter of the transfer line should be chosen depending on the internal diameter of the analytical column, the column flow rate, and the flow rate delivered from the autosampler. To eliminate tubing dead volume, use the smallest diameter tubing possible. For example, compound residence time in a 1.0mm ID transfer line is 3.6 times greater than in the same length of 0.53mm ID tubing. Reducing the residence time of the headspace sample in the transfer line helps to minimize band broadening. Therefore, the flow rate should be set as high as possible to quickly move the sample cloud through the tubing and minimize any dead volume effects.



Transfer line temperature should be set depending on the analytes of interest and the sample matrix. Typical transfer line temperatures range from 80°C to 125°C. To minimize matrix problems and prevent water condensation from aqueous samples, use a higher transfer line temperature (~125°C to 150°C).

Restek's technical service is here to help. If you still have questions after reviewing this guide, please call us at 800-356-1688 or 814 353-1300, ext. 4, or call your local Restek representative.

Injection Port Interface

The quality of the connection of the transfer line to the analytical column greatly affects sample bandwidth. In most cases, the transfer line has a smaller internal diameter than the injection port liner, and the vaporized headspace sample carrying the compounds of interest will be diluted into a larger volume of carrier gas when the sample elutes from the transfer line into the inlet liner. This can lead to broader peaks, tailing peaks, lower sensitivity, and loss of resolution. Because headspace samples are already in a gaseous state (vapor cloud) when they enter the injection port, there is no need to use a large buffer volume in the liner to allow for sample expansion as when analyzing liquid samples. Using injection port liners that have smaller internal diameters and lower buffer volumes will help maintain a narrow bandwidth as samples move from the end of the transfer line to the head of the analytical column. 1.0mm ID deactivated injection port liners are recommended for most headspace applications to achieve the lowest injection port dead volume.

If band-broadening due to excess dead volume in the system is still a problem, peak shape may be improved by refocusing sample analytes at the analytical column head. Highly volatile compounds can be trapped at the column head and refocused into a narrow bandwidth by reducing the initial oven temperature below the boiling point of compounds of interest. After the sample is completely transferred to the column, the oven temperature can be increased to move the compounds through the column.

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Headspace Applications

Blood Alcohol Analysis

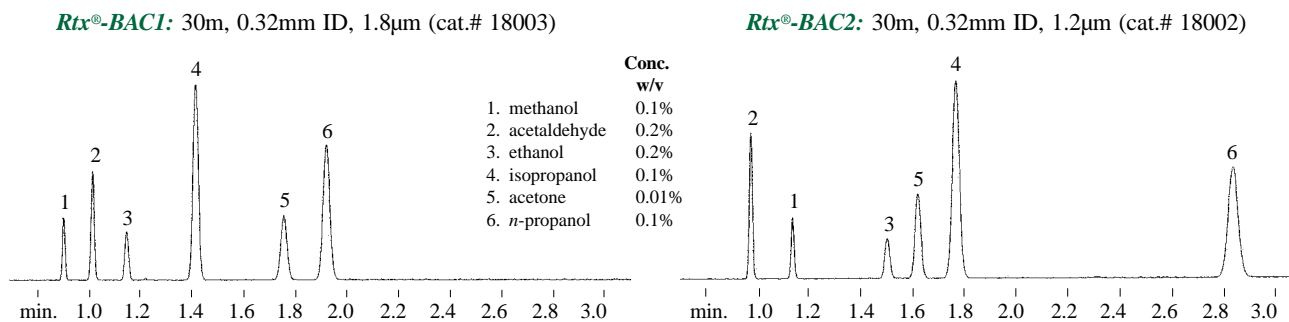
Analysis time and resolution are two critical factors when developing a GC assay for ethanol. Analysis time for each sample should be as short as possible while still maintaining baseline resolution for all analytes. Isothermal analysis is the method of choice because it eliminates the cool-down period between temperature-programmed runs. Overall analysis time can be reduced in isothermal analysis by raising the oven temperature or by increasing carrier gas flow rate. However, in attempting to shorten the analysis time, either by increasing the flow rate or raising the temperature, many traditional capillary column stationary phases fail to provide adequate resolution of all the components commonly tested during blood alcohol analysis. Current advances have aided in the design of two novel capillary column stationary phases to meet all of these requirements—the Rtx®-BAC1 and the Rtx®-BAC2 columns.

Quantitation Technique for Blood Alcohol Analysis (Internal Standard)

The internal standard technique uses one or more designated compounds at known concentrations spiked into the sample. The response of the compounds of interest are then compared to the results of the internal standard. There are several advantages to this technique. Multiple injections of the standard are not necessary for concentration calculations; small changes in injection volumes or detector response over time can be determined.

Figure 12

Achieve baseline resolution of all blood alcohol components in less than 3 minutes using the Rtx®-BAC1 and Rtx®-BAC2 columns and a Perkin-Elmer HS 40 headspace autosampler.



Dual-column analysis using a two-hole ferrule. 1.0mL headspace sample of a blood alcohol mix.

Oven temp.: 40°C isothermal; Inj. temp.: 200°C; Carrier gas: He; Sample equilibration temp.: 70°C; Sample equilibration time: 15 min.; Vial pressure: 30psi; Vial pressurization time: 0.15 min.; Vial sampling time: 0.01 min.; Transfer line: 0.32mm ID FS Hydroguard™ tubing; Transfer line temp.: 200°C; Injection port sleeve: 2mm ID; Split flow: 20mL/min.

A balanced pressure sampling unit was used to transport the sample to the GC. This type of sampling works better with columns that require higher head pressure (smaller ID) to improve flow efficiencies. 0.32mm ID analytical columns were chosen for this application because of their higher operating pressure. Optimal column performance during headspace analysis depends on GC/headspace system set up. Band broadening can occur if there is excess dead volume in the sample flow path between the sample valve and the head of the column. Low volume inlet liners or interfaces in the injection port should be used to reduce the amount of excess volume at the exit end of the transfer line. A 2mm ID liner was used in this analysis to reduce dead volume and maintain narrow peak widths. High carrier gas flow rates through the transfer line also can be used to maintain narrow sample bandwidths and speed up sample transfer to the column head. A flow of 40mL-per-minute was used to optimize the analysis on the Perkin-Elmer HS 40 system.

Simulated blood alcohol samples were prepared and analyzed using a modification of a procedure published by Christmore et al.² n-Propanol was used as the internal standard and was prepared at a concentration of 0.03g/dL in 1.0M ammonium sulfate as a diluent. Five milliliters of diluent were added to 1mL of sample in a 20mL headspace vial (Figure 12).

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These conditions (PE Auto SYS GC and HS 40 headspace autosampler), combined with unique columns (Rtx®-BAC1 and Rtx®-BAC2), provided excellent accuracy and precision in the analysis of blood alcohol with complete resolution in less than 3 minutes. Calibration curves were constructed using concentrations ranging from 0.01% to 0.5% ethanol. Correlation coefficients above 0.999 were easily obtained for all compounds. Response factor repeatability was less than $\pm 1\%$ standard deviation while analyzing six samples at a concentration of 0.2% ethanol. Based on our experimentation, a system detection limit of 0.001% ethanol should be achievable while maintaining a minimum signal-to-noise ratio of 10. For more information on this analysis request, request cat.# 59548.

Quantitation Technique for USP <467> (External Standard)

This technique uses a separate sample (standard) that has the compounds of interest at known concentrations in the same matrix. This technique is advantageous if various samples are being analyzed, and all compounds of interest can be assayed using a single set external standards.

USP <467>

A new test for the gas chromatographic (GC) analysis of Organic Volatile Impurities (OVI) in pharmaceutical products was published in the Third Supplement to the US Pharmacopoeia (USP) XXII-NF XVII, which became effective November 15, 1990.

Since its original appearance in the USP, this testing protocol has undergone many revisions and additions.¹⁻⁶ The most recent of which was published as USP 24, effective January 1, 2000.⁷ The biggest change was to the limit test concentrations, which now match the European Pharmacopoeia (EP) concentrations and the ICH guidelines for the five USP <467>-regulated solvents.^{8,9}

Limit Test Concentrations for USP <467>

benzene	New 2ppm
chloroform	60ppm
1,4-dioxane	380ppm
methylene chloride	600ppm
trichloroethene	80ppm

USP issued an in-process revision announcing that the limit test for benzene is not required unless a specific limit for benzene is included in the individual drug monograph.¹⁰ The revision was needed because Methods I and V were unable to detect benzene at 2ppm. Currently, Method IV is the only method that detects benzene at 2ppm. It is anticipated that USP will make more revisions to benzene detection limits during 2000.

USP also has clarified that a 5m phenyl-methyl guard column is not needed for the Method IV, headspace analysis.¹⁰

Figure 13 shows an analysis using Method IV at the revised concentrations, the method-specified sample preparation procedure, a G43 analytical column, and no guard column.

USP made changes in 1997 to overcome the difficulties resulting from unregulated solvents coeluting with regulated solvents, and thereby causing over-representation of their concentrations using GC/flame ionization detection (FID) methods.¹¹ GC/mass spectrometry (MS) or a second, validated column having a different stationary phase may be used to confirm the presence of the coeluting unregulated solvent and report the correct concentration of regulated solvent. For more information on this analysis request, request cat.# 59577A.

Table IV

*Organic Volatile Impurities (OVI)
methods and corresponding
chromatographic systems.*

Method IV - Static Headspace

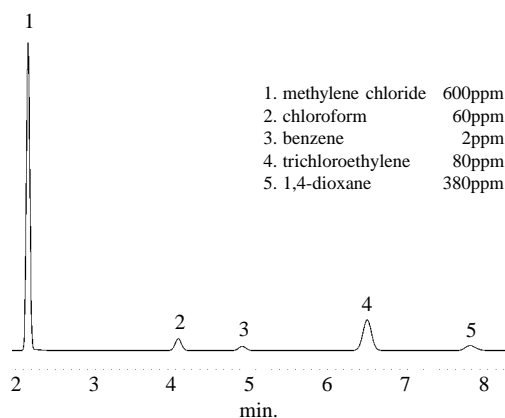
6% cyanopropylphenyl/94%
dimethylpolysiloxane (G43)
30m, 0.53mm ID, 3.0 μ m
(Rtx®-G43 column, cat.# 16085-126)

Method for Coated Tablets - Static Headspace

0.2% polyethylene glycol, MW 1500 (G39)
on graphitized carbon (S7)
(0.2% Carbowax® 1500 on 80/100
CarboBlack™ C packed column, cat. # 80122)

Figure 13

The Rtx®-G43 column provides the resolution and detection limits needed for USP 24th edition <467> revised limit test concentrations in USP Method IV.



Sample Preparation: 100µL of cat.# 36007 in 5mL distilled water, 1 gram sodium sulfate in a 20mL headspace vial.

30m, 0.53mm ID, 3.0µm Rtx®-G43 (cat.# 16085)

Oven temp.: 40°C (hold 20 min.) to 240°C @ 35°C/min. (hold 20 min.);

Inj. temp: 140°C, 1mm split sleeve (cat.# 20916);

Det. temp.: 260°C;

FID sensitivity: 1.25 x 10⁻¹¹ AFS;

Carrier gas: helium, 3.5psi constant pressure, 35cm/sec. set @ 40°C; **Split ratio:** 2:1; ThermoQuest HS 2000 Headspace Autosampler Vial 80°C, 60 min. shaker on.

Comments in the September/October 1992 Pharmacopoeial Forum³ propose the use of dimethyl sulfoxide as the solvent for stock standard, but this has not been approved as of the date of this publication.

In regards to this proposal, an investigation was conducted to determine if there were significant changes in results if dimethyl sulfoxide was used as the diluent for stock standard. Similar RSDs can be obtained when stock solutions are diluted in dimethyl sulfoxide as opposed to solutions made with water (**Table V**).

Table V

Percent RSD for stock solutions in water vs. DMSO

Stock Solvent	Methylene Chloride	Chloroform	Benzene	1,1,1-Trichlorethylene	1,4-Dioxane
Water	10.19	10.64	14.52	16.75	18.61
Water w/ Sample	8.29	9.25	11.3	13.38	15.26
DMSO	7.37	8.59	8.18	8.01	14.43
DMSO w/ Sample	7.25	7.54	8.8	8.67	7.37

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Quantitation Techniques for European Pharmacopoeia

(External Standard)

This technique uses a separate sample (standard) that has the compounds of interest at known concentrations in the same matrix. This technique is advantageous if various samples are being analyzed, and all compounds of interest can be assayed using a single set external standards.

(Standard Addition)

The standard addition technique uses known amounts of the compounds of interest and adds it to the existing sample. The original concentration of the compounds of interest are then calculated using linear regression.

Peak List for Figure 15

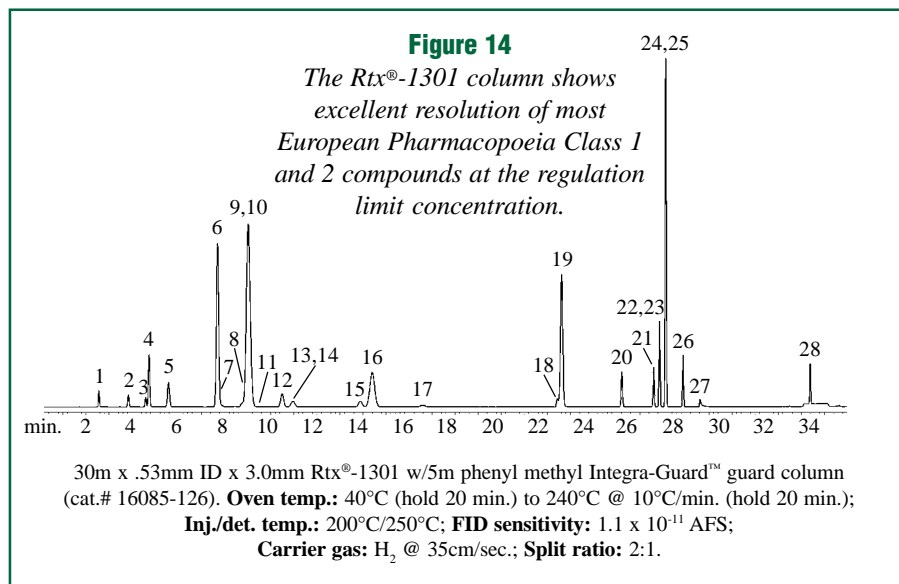
Headspace injection of 28 Class 1 and Class 2 residual solvents for pharmaceutical processing. Prepared at the regulatory limit concentration. Samples shaken and heated at 80°C for 15 minutes, 1mL headspace injection.

1. methanol
2. 1,1-dichloroethene
3. acetonitrile
4. methylene chloride (dichloromethane)
5. hexane (C6)
6. *cis*-1,2-dichloroethene
7. nitromethane
8. chloroform
9. cyclohexane
10. 1,1,1-trichloroethane
11. carbon tetrachloride
12. benzene
13. 1,2-dimethoxyethane
14. 1,2-dichloroethane
15. trichloroethylene (1,1,2-trichloroethene)
16. methylcyclohexane
17. 1,4-dioxane
18. pyridine
19. toluene
20. 2-hexanone
21. chlorobenzene
22. DMF
23. ethylbenzene
24. *m*-xylene
25. *p*-xylene
26. *o*-xylene
27. N,N-dimethylacetamide
28. 1,2,3,4-tetrahydronaphthalene

European Pharmacopoeia Tests

The International Conference on Harmonization (ICH) has proposed a set of guidelines for residue solvent testing in pharmaceutical formulation and the European Pharmacopoeia (EP) was the first to revise their regulations.^{7,8} However, these guidelines are challenging, containing over 60 compounds of regulatory interest to manufacturers of active substances, excipients, and medicinal products. The EP methods also allow testing limits based on either a concentration limit in a product, or calculated from the maximum daily dosage of the product and the permissible daily exposure limit of the solvent. These technical challenges affect the sampling method and capillary column needed to ensure precise and accurate results.

The recommended primary capillary column for EP residual solvent testing is the Rtx®-1301. The Rtx®-1301 column shows excellent resolution of most EP Class 1 and Class 2 compounds at the regulation limit concentration (**Figure 14**). Restek also offers Stabilwax® columns, the recommended confirmational column for European Pharmacopoeia residual solvent testing. For more information on this analysis request, request cat.# 59107.



Guide References

1. M.S. Bergren and D.W. Foust, "Comments on USP General Chapter, Organic Volatile Impurities <467>," and Associated Monograph Proposals," *Pharmacopoeial Forum*, May/June 1991, Vol. 17, No. 3, pp. 1963-1968.
2. J.A. Krasowski, H. Dinh, T.J. O'Hanlon, R.F. Lindauer, "Comments on Organic Volatile Impurities, Method I, <467>," *Pharmacopoeial Forum*, May/June 1991, Vol. 17, No. 3, pp. 1969-1972.
3. *Pharmacopoeial Forum*, March/April 1991, Vol. 17, No. 2, p. 1653.
4. Fifth Supplement, USP-NF, Organic Volatile Impurities <467>, Nov. 15, 1991, pp. 2706-2708.
5. "Organic Volatile Impurities <467>," *Pharmacopoeial Forum*, May-June 1993, Vol. 19, No. 3, pp. 5335-5337.
6. *Pharmacopoeial Forum*, September/October 1992, Vol. 18, No. 5, p. 4028.
7. USP 24/NF 19, <467> Organic Volatile Impurities, (1877-1878).
8. "ICH Harmonized Tripartite Guideline, Impurities: Guideline for Residual Solvents," *The Fourth International Conference on Harmonization*, July 17, 1997.
9. European Pharmacopoeia, Supplement 1999, pp. 14-15, 208.
10. *Pharmacopoeial Forum*, November - December 1999, Vol. 25, Number 6, (9223 - 9224).
11. Sixth Supplement, USP-NF, Organic Volatile Impurities <467>, May 15, 1997, pp. 3766-3768.

These references are not available from Restek.

Recommended Static Headspace Analysis Products:

Capillary Columns

Rtx®-BAC1 Capillary Columns

ID (mm)	df (µm)	30-Meter
0.32	1.80	18003
0.53	3.00	18001

Rtx®-G27 Integra-Guard™ Column

ID (mm)	df (µm)	30-Meter
0.53	5.00	10279-126

Rtx®-5 Capillary Columns

ID (mm)	df (µm)	30-Meter
0.32	3.00	10284
0.53	5.00	10279

Stabilwax® Capillary Columns

ID (mm)	df (µm)	30-Meter
0.32	0.25	10624
0.53	0.50	10640
0.53	1.00	10655

Rtx®-BAC2 Capillary Columns

ID (mm)	df (µm)	30-Meter
0.32	1.20	18002
0.53	2.00	18000

Rtx®-G43 Integra-Guard™ Column

ID (mm)	df (µm)	30-Meter
0.53	3.00	16085-126

Rtx®-1301 Capillary Columns

ID (mm)	df (µm)	30-Meter
0.32	1.50	16069
0.53	3.00	16085

Guard Columns

Fused Silica Guard Columns

ID (mm)	OD (mm)	5-Meter
0.32	0.45 ± 0.04	10044
0.53	0.69 ± 0.05	10045

Integra-Guard™ Guard Columns

ID (mm)	OD (mm)	Length	Suffix
0.32	0.45 ± 0.04	5m	-125
0.32	0.45 ± 0.04	10m	-128
0.53	0.69 ± 0.05	5m	-126
0.53	0.69 ± 0.05	10m	-129

For technical support, call
800-356-1688, ext. 4
 (814-353-1300, ext. 4)

or call your local
 Restek representative.

Press-Tight® Connectors

Universal Press-Tight® Connectors

- Ideal for connecting guard columns to analytical columns.
- Repair broken columns.
- Connect column outlets to transfer lines.

5-pk.	25-pk.	100-pk.
20400	20401	20402

Universal 'Y' Press-Tight® Connectors

- Split sample flow onto two columns.
- Split a single column flow into two different detectors.
- Perform confirmational analysis with a single injection.

each	3-pk.
20405	20406

Universal Angled Siltek™-Deactivated Press-Tight® Connectors

- Siltek™ deactivation for inert pathways to maintain sample integrity.
- Ideal for connecting guard columns to analytical columns.
- Designed at an angle approximating the radius of a capillary column.

5-pk.	25-pk.	100-pk.
20482	20483	20484

Universal Angled Press-Tight® Connectors

- Ideal for connecting guard columns to analytical columns.
- Designed at an angle approximating the radius of a capillary column.
- Reduces strain on column-end connections.

5-pk.	25-pk.	100-pk.
20446	20447	20448

Universal Angled 'Y' Press-Tight® Connectors

- Alleviates column-end connection strain.
- Inlet and outlet ends conform to the column radius.
- Perform confirmational analysis with a single injection.

each	3-pk.
20403	20404

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Analytical Reference Materials

USP <467> Reference Material Mixes

USP <467> Calibration Mix #2

benzene	100µg/mL
chloroform	50
1,4-dioxane	100
methylene chloride	500
trichloroethene	100

Prepared in methanol, 1mL/ampul

Ea.: cat.# 36002	10-pk.: cat.# 36102
-------------------------	----------------------------

USP <467> Calibration Mix #4

benzene	2µg/mL
chloroform	60
1,4-dioxane	380
methylene chloride	600
trichloroethene	80

Prepared in methanol, 1mL/ampul

Ea.: cat.# 36006	10-pk.: cat.# 36106
-------------------------	----------------------------

USP <467> Calibration Mix #3

benzene	100µg/mL
chloroform	50
1,4-dioxane	100
methylene chloride	500
trichloroethene	100

Prepared in DMSO, 1mL/ampul

Ea.: cat.# 36004	10-pk.: cat.# 36104
-------------------------	----------------------------

USP <467> Calibration Mix #5

benzene	2µg/mL
chloroform	60
1,4-dioxane	380
methylene chloride	600
trichloroethene	80

Prepared in DMSO, 1mL/ampul

Ea.: cat.# 36007	10-pk.: cat.# 36107
-------------------------	----------------------------

European Pharmacopoeia/ICH Reference Material Mixes

Class 1 Mix

benzene	2µg/mL
carbon tetrachloride	4
1,2-dichloroethane	5
1,1-dichloroethene	8
1,1,1-trichloroethane	1500

Prepared in water:dimethylsulfoxide 90:10, 1mL/ampul

Each	5-pk.	10-pk.
36228	36228-510	36328

Class 2 Mix A

chlorobenzene	360µg/mL
cyclohexane	3880
cis-1,2-dichloroethene	1870
dichloromethane	600
ethylbenzene	369
hexane	290
methylcyclohexane	1180
N,N-dimethylformamide	880
toluene	890
1,1,2-trichloroethene	80
m-xylene	1302
o-xylene	195
p-xylene	304

Prepared in water:dimethylsulfoxide 90:10, 1mL/ampul

Each	5-pk.	10-pk.
36229	36229-510	36329

Class 2 Mix B

acetonitrile	410µg/mL
chloroform	60
1,2-dimethoxyethane	100
N,N-dimethylacetamide	1090
1,4-dioxane	380
1,2,3,4-tetrahydronaphthalene (tetraline)	100
2-hexanone	50
methanol	3000
nitromethane	50
pyridine	200

Prepared in water:dimethylsulfoxide 90:10, 1mL/ampul

Each	5-pk.	10-pk.
36230	36230-510	36330

Class 2 Mix C

2-ethoxyethanol	160µg/mL
ethylene glycol	620
formamide	220
2-methoxyethanol	50
N-methylpyrrolidone	4840
sulfolane	160

Prepared in water, 1mL/ampul

Each	5-pk.	10-pk.
36231	36231-510	36331

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GC Accessories

Headspace Autosampler Vials

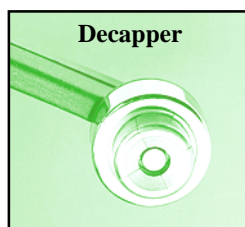
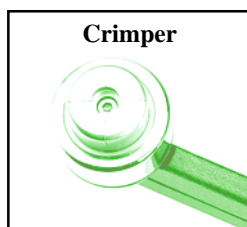
Description	100-pk.	1000-pk.
6mL Clear Vial	21166	21167
10mL Clear Vial, Flat Bottom	24683	24684
10mL Clear Vial, Rounded Bottom	21164	21165
20mL Clear Vial, Flat Bottom	24685	24686
20mL Clear Vial, Rounded Bottom	21162	21163
27mL Clear Vial	21160	21161

20mm Aluminum Seals w/Septa, Assembled

Description	100-pk.	1000-pk.
Silver Seal w/ PTFE/Gray Butyl Rubber	21761	21762
Silver Seal w/ PTFE/Silicone	21763	21764
Pressure Release, Silver Seal w/ PTFE/ Gray Butyl Rubber Septa <125°C	21765	21766
Pressure Release, Silver Seal w/ PTFE/ Silicone Septa >125°C	21767	21768

Aluminum Seal Crimper and Decapper

The crimper is adjustable for optimized sealing performance. It also is comfortable enough for pro- longed use. For chromatographers who need to save, transfer, or dispose of their samples, we provide a decapper that allows the user to remove a crimp-top cap safely and easily. If you haven't used an aluminum seal decapper, order one today!



Size	Crimper	Decapper
8mm	21735	21736
11mm	21170	21171
13mm	21739	21740
20mm	21737	21738

Thermolite® Septa

Septum Diameter	25-pk.	50-pk.	100-pk.
9.5mm (3/8")	20359	20360	20361
10mm	20378	20379	20380
11mm (7/16")	20363	20364	20365
12.5mm (1/2")	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Plug	20372	20373	20374

To request a FREE sample of Thermolite® septa, call 800-356-1688, ext. 5, or your local Restek representative.



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Capillary Ferrules*(for 1/16" compression-type fittings)*

Ferrule ID (mm)	Fits Column ID (mm)	Graphite 50-pk.	Vespal®/ Graphite 50-pk.
0.4	0.25	20227	20229
0.5	0.32	20228	20231
0.8	0.53	20224	20230

Two-Hole Ferrules*(for 1/16" compression-type fittings)*





Ferrule ID (mm)	Fits Column ID (mm)	Graphite 5-pk.	Vespal®/ Graphite 5-pk.
0.4	0.25	20235	20241
0.5	0.32	20235	20242
0.8	0.53	20245	20246

Graphite Ferrules for M4 Fittings*(for QCQ Fisons 8000 & TRACE 2000)*





Ferrule ID (mm)	Fits Column ID (mm)	Graphite 2-pk.	Graphite 10-pk.
0.4	0.18–0.25	20280	20281
0.5	0.32	20282	20283
0.8	0.50 & 0.53	20284	20285

Please request the Inlet Supplies Guide (#59893) for a comprehensive list of Restek's inlet supplies.


**Inlet Liners for HP/Finnigan GCs**

Liner	ID/OD/length	ea.	5-pk.	25-pk.
 2mm Splitless	2.0 x 6.5 x 78.5	20712	20713	20714
 Gooseneck Splitless (2mm)	2.0 x 6.5 x 78.5	20795	20796	20797
 Recessed Gooseneck (2mm)	2.0 x 6.5 x 78.5	20980	20981	20982
 1mm Split	1.0 x 6.3 x 78.5	20972	20973	—


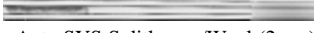
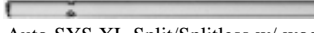
Inlet Liners for Varian GCs

Liner	ID/OD/length	ea.	5-pk.	25-pk.
 1mm Split	1.0 x 6.3 x 72	20970	20971	—
 2mm Splitless	2.0 x 6.3 x 74	20721	20722	20723
 Open 0.5mm ID	0.5 x 5.0 x 54	20992	20993	—
 Open 0.75mm ID	0.75 x 5.0 x 54	21714	21715	21716


Inlet Liners for Shimadzu GCs

Liner	ID/OD/length	ea.	5-pk.	25-pk.
 17A 1mm Split	1.0 x 5.0 x 94	20976	20977	20978


Inlet Liners for Perkin-Elmer GCs

Liner	ID/OD/length	ea.	5-pk.	25-pk.
 2mm Splitless	2.0 x 5.0 x 100	20730	20731	20732
 Auto SYS Splitless w/Wool (2mm)	2.0 x 6.2 x 92.1	20829	20830	20831
 Auto SYS XL Split/Splitless w/ wool	2.0 x 4.0 x 81.2	21717	21718	—

Inlet Liners for CE Instruments/ThermoQuest GCs**for 5000 and 6000 GCs**

Liner	ID/OD/length	ea.	5-pk.	25-pk.
 2mm Splitless	2.0 x 5.5 x 79.5	20811	20812	20813

for TRACE and 8000 GCs

Liner	ID/OD/length	ea.	5-pk.	25-pk.
 1mm Split	1.0 x 8.0 x 105	20916	20917	—

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Hewlett-Packard 1/16-Inch Capillary Inlet Adaptor Fitting Kit

Restek has specially engineered a high-precision, 1/16-inch fitting that uses standard size, two-hole capillary ferrules. The fitting kit comes with everything needed for dual-column confirmational analysis using 0.25 and 0.32mm ID capillary columns (two-hole ferrules must be ordered separately).

Capillary Inlet Adaptor Fitting Kit (for 0.25/0.32mm ID columns): cat.# 20633

Replacement Inlet Seal (1.2mm hole): cat.# 20390, (2-pk.); cat.# 20391, (10-pk.)



Hewlett-Packard 1/8-Inch Capillary Inlet Adaptor Fitting Kit

Restek has specially engineered a high-precision, 1/8-inch fitting that uses standard 1/8-inch, two-hole capillary ferrules. The fitting kit comes with everything needed for installation.

1/8-inch Capillary Inlet Adaptor Fitting Kit (for 0.53mm ID columns): cat.# 20645

Replacement Inlet Seal (1/16-inch hole): cat.# 20392, (2-pk.); cat.# 20393, (10-pk.)



Low-Volume Injector for Hewlett-Packard 5890 Septum Packed Purge Port

Includes a 1/16-inch nut, a 1/16-inch ferrule, a base nut and 1/4-inch Vespel®/graphite ferrule, a 1/16-inch capillary nut, a 5-pack of low-bleed plug septa, and a special low-mass septa nut. Order appropriate capillary ferrules separately.

Description

LVI for HP 5890 Septum Packed Purge Port

Kit

cat.# 21698



Low-Volume Injector for Hewlett-Packard and Varian GCs

Includes a 1/16-inch nut, a 1/16-inch ferrule, a base nut and 1/4-inch Vespel®/graphite ferrule, a 1/16-inch capillary nut, a 5-pack of low-bleed plug septa, and a special low-mass septum nut. Order appropriate capillary ferrules separately.

Description

LVI for HP Split/Splitless GC inlets

Kit

cat.# 21692

LVI for Varian Split/Splitless GC inlets

cat.# 21693



Restek Leak Detective™ Electronic Leak Detector

The Leak Detective™ responds in less than 2 seconds to trace leaks of gases with thermal conductivities different than air. Helium or hydrogen can be detected at 3×10^{-4} cc/sec.* or at an absolute concentration as low as 200ppm. Leaks are indicated by an audible alarm, as well as by an LED readout. (Batteries and AC adaptor included.)

*Caution: not designed for determining leaks of combustible gases.

Description

Restek Leak Detective™ Electronic Leak Detector (110 volts)

Each

cat.# 21607

Restek Leak Detective™ Electronic Leak Detector (220 volts)

cat.# 21609



Restek Veri-Flow 500 Electronic Flowmeter

- Calculates linear velocity based on column ID.
- Measures N₂, He, H₂, 5% Ar/Me, and Air.
- Measures split flow and mass flow.
- Has pulse-free operation that will not interfere with EPCs.
- Reads flow accurately from 5 to 500mL/min.

Description

Restek Veri-Flow 500 Electronic Flowmeter (110 volts)

Each

cat.# 21643

Restek Veri-Flow 500 Electronic Flowmeter (220 volts)

cat.# 21645



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We do what it takes to meet & satisfy our customer's needs!

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Restek's highly trained customer service team looks forward to working with you. We are here when you need to place an order, track a package, check the status of an open order, or request a price quote. We will suggest time- and money-saving options and are dedicated to getting your products to you *fast*. Because we know how busy you are, we will do whatever it takes to simplify your work. That's what having the best customer service in the business is all about! Our extended hours are 8:00 a.m. to 6:00 p.m. EST, Monday thru Friday.



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Our newly-formed Chromatography Information Services (CIS) team focuses its resources on innovating ways to teach the art and science of chromatography. This knowledge management group is dedicated to exploring and implementing ways of capturing, organizing, and disseminating chromatographic knowledge, experience, and wisdom to our internal and external customers worldwide. CIS enhances Restek's vision of becoming *"the first company chromatographers think of whenever they have a separation need or chromatographic question."*

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Selection Guide for Polar WAX GC Column Phases.....



Inside:

**Information on
Six Different PEG
Columns**

**Polyethylene Glycol
Stationary Phase
Selection Guide**

**Applications for
Each Polyethylene
Glycol Column**

RESTEK
CORPORATION



"Among the most widely used capillary columns today are the polar polyethylene glycol (PEG) columns. While PEG columns offer a unique selectivity for a wide variety of analyses, their uses have been limited because of short column lifetimes, restricted operating ranges, and poor inertness.

Restek Corporation has been providing chromatographers with superior capillary columns since 1985. And, our PEG columns are just another example of how we've improved on existing technology to solve customers' problems. The columns in this guide are durable, function at a wide range of temperatures, and are highly inert.

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Stabilwax®-DA Columns	5
Stabilwax®-DB Columns	6
Rtx®-Wax Columns	7
FAMEWAX™ Columns	9
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We have designed six different PEG capillary GC column phases — Stabilwax®, Stabilwax®-DA, Stabilwax®-DB, FAMEWAX™, Rtx®-Wax, and MXT®-Wax columns — each an innovative improvement over existing, competitor PEG columns. In addition to the wide variety of PEG columns, we offer many custom column materials and configurations. We will try to match any column written into a method, or engineer a new custom PEG phase if your needs are special. If you cannot find what you are looking for, call our technical support at 800-356-1688, ext. 4."

——— **Rick Crago**, Product Line Manager

Mark Lawrence & Tom Gurecki
Mark and Tom lead the manufacturing efforts for all of the phases included in this publication. Their goal is to bring you the most consistent columns, while continuing to expand the product offering.



Polyethylene Glycol Stationary Phase Selection Guide

Restek Phase	Temp. Limits	Cross-Reference	Typical Analyses	Benefits
Stabilwax® MXT®-WAX	40 to 260°C	Supelcowax™-10, HP-Innowax, Carbowax®, CP wax 52 CB	FAMES, Flavors, Essential Oils, Solvents, BTEX	Resists oxidation, extended lifetimes, high thermal stability.
Stabilwax®-DA	40 to 260°C	Nukol™, SP™-1000, HP-FFAP, AT-1000, BP 21, CP Wax 58 CB, 007 FFAP, DB-HAP	Free Fatty Acids	Provides efficiency and inertness for acidic compounds.
Stabilwax®-DB	40 to 220°C	CAM, Carbowax® Amine, CP Wax 51	Amines	Provides efficiency and inertness for basic compounds.
Rtx®-Wax	20 to 250°C	DB-Wax, HP-Wax, AT-Wax, 007-Wax	Flavors, Essential Oils, Polar Solvents, BTEX	Increased efficiency and inertness over conventional wax columns, lower minimum operating temperature.
FAMEWAX™	20 to 250°C	Omegawax™	Fatty Acid Methy Esters	Specifically designed for fast, efficient analysis of FAMES.

Stabilwax® Columns for Solvents, FAMES, Xylene Isomers, and Flavors

- Increased column lifetime.
- Remarkably low bleed.
- Solvent rinsable.

Stabilwax® columns are durable, all-purpose PEG columns. FAMES, flavors, acids, essential oils, amines, solvents, xylene isomers, BTEX, and EPA Method 603 compounds (acrolein and acrylonitrile) are all easily analyzed on these rugged columns.

Restek's research chemists developed a polar-deactivated surface that tightly binds the Carbowax® polymer, thus increasing the polymer's thermal stability over competitive columns. Next, they incorporated antioxidant features into the polymer backbone to resist damage from an influx of trace oxygen, the most common cause of Carbowax® column failure. Finally, they fine-tuned the bonding mechanisms to result in a column that can be rejuvenated by solvent-washing.

Benefits of Low Bleed

The low bleed at higher temperatures that is provided by a Stabilwax® column ensures accurate identification and quantitation of higher-boiling components, minimal detector contamination, and extended column lifetime. The Stabilwax® column is thermally stable to 250°C. Only a minimal bleed level is evident at this temperature. In fact, compared to similar PEGs from other manufacturers, the Stabilwax® exhibits the **lowest** bleed profile at 250°C (**Figure 1**).

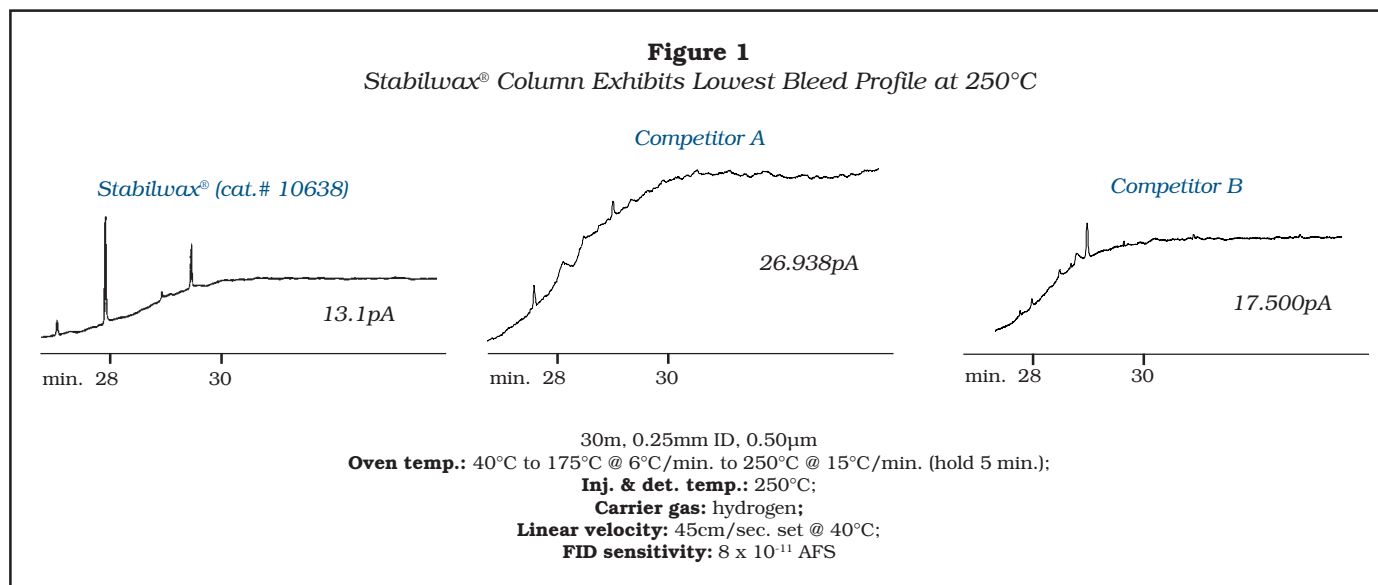


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Stabilwax® Columns for Complex Mixtures

This PEG column also provides an affinity toward and separation of many polar compounds found in complex samples such as spearmint oil and industrial solvent mixtures. (**Figures 2 and 3**)

Figure 2

Flavor Components of Native Spearmint Oil are Baseline Resolved on a Stabilwax® Column

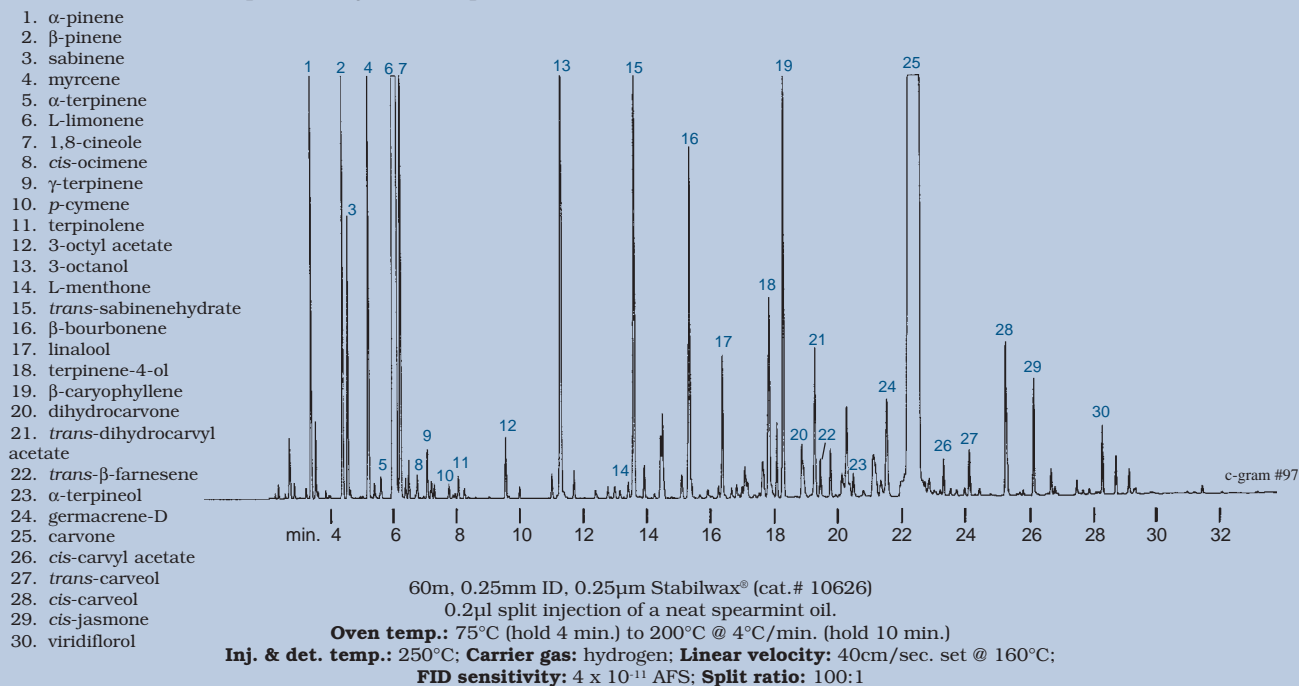
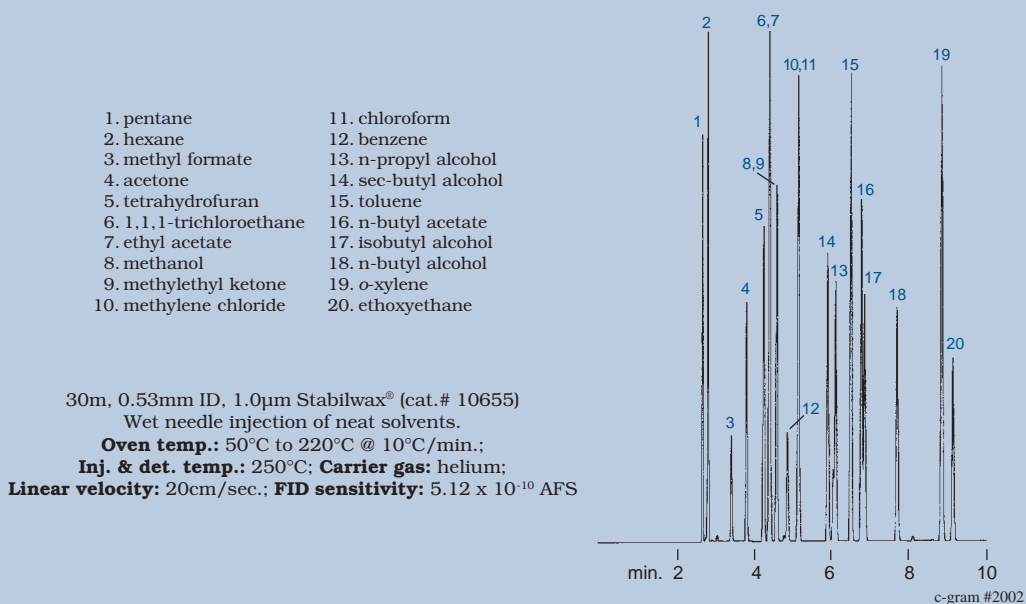


Figure 3

Industrial Solvents Separated on a Stabilwax® Column in Under 10 minutes



Stabilwax®-DA Columns for Acidic Compounds

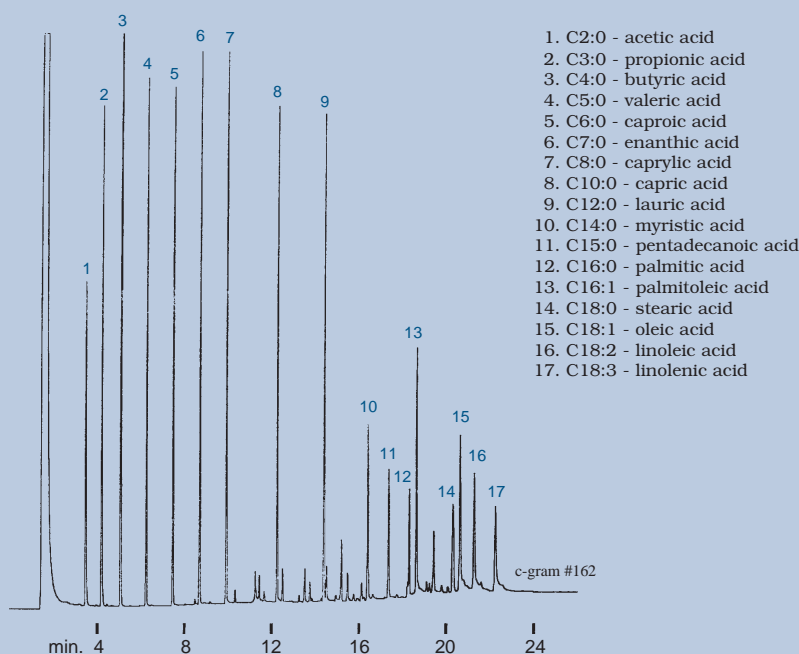
- Ideal for free fatty acids analysis.
- Thermally stable to 250°C.
- Solvent rinsable.
- Resolves saturated and unsaturated free fatty acids.
- No derivatization needed of free fatty acids.
- Inertness increases sample capacity.
- Achieves good peak shapes.

The analysis of volatile free fatty acids has been difficult on common siloxane stationary phases because of analyte adsorption and low sample capacity. Although PEG stationary phases exhibit improved capacity and better selectivity for many free acids, peak tailing still can occur. Stabilwax®-DA is a bonded PEG stationary phase that has been specially deactivated to analyze acidic compounds. The resulting inertness significantly reduces adsorption and increases sample capacity of volatile-free acids, thereby improving peak shape (**Figure 4**). It also eliminates the need to derivatize free fatty acids from C1 to C20, which simplifies sample preparation. Restek's deactivation and innovative Crossbond® procedure produces columns that last longer and give better peak shapes with high molecular weight acids than our competitors.

Restek's Crossbond® Procedure produces columns with lower bleed, longer lifetime, solvent rinsability, and you can use direct aqueous injections.

Figure 4

Stabilwax®-DA Column Gives Good Peak Shapes for Free Fatty Acids



30m, 0.53mm ID, 0.25µm Stabilwax®-DA (cat.# 11025)
0.5µl direct injection of a 5mg/ml standard.

Oven temp.: 100°C (hold 2 min.) to 250°C @ 8°C/min.;

Inj. & det. temp.: 280°C; **Carrier gas:** helium;

Linear velocity: 40cm/sec. (flow rate: 5.2cc/min.); **FID sensitivity:** 8 x 10⁻¹¹ AFS

Special deactivation improves separations and simplifies sample preparation.

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at www.restekcorp.com
for detailed product
information and to
order free literature.**

Unbreakable Metal Capillary Columns



For harsh environments or special applications where fused silica tubing may not be the ideal material, Restek offers MXT® capillary columns. MXT® columns are made from unbreakable, thin-wall stainless steel that has been treated with our Silcosteel® process. This process deposits a sub-micron layer of flexible fused silica on the inside of the tubing, giving it the same inertness as an Rtx® column, but with the ruggedness of stainless steel. MXT® columns are caged in small diameter coils that are ideal for portable or process GCs.

MXT®-Wax columns combine the unique selectivity of the PEG stationary phases with the rugged durability of stainless steel tubing. These columns are ideal for use in the process and portable GC industries, where column breakage is a significant problem.

**See page 12 for
ordering information.**

Stabilwax®-DB Columns for Basic Compounds

- Bonded phase reduces column bleed.
- Thermally stable to 220°C.
- Eliminates need for derivatization.
- No column priming required.
- Reproducible amine analyses.
- Stringently tested for analysis of difficult basic compounds.

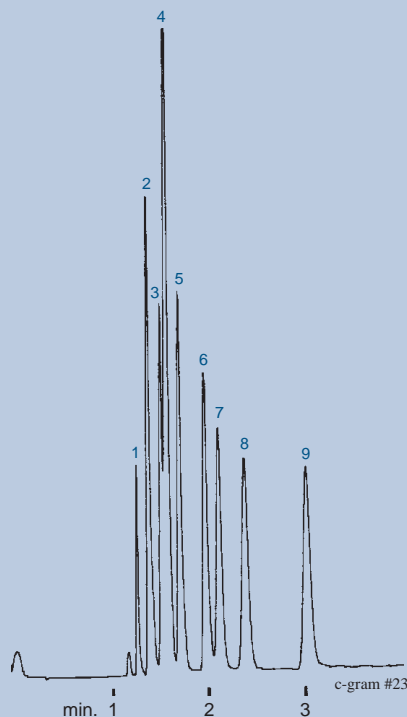
This column was developed to reduce adsorption and improve response of basic compounds without the need for column priming. Analyses that previously required derivatization, or another analytical technique such as HPLC, can now be performed on the Stabilwax®-DB column. In addition to the stringent Grob test for efficiency, bleed, and inertness (excluding oxygenated compounds), the Stabilwax®-DB column must also analyze a mixture that contains triamine, diamine, and propylamine. This additional test ensures that every Stabilwax®-DB column is basic enough to analyze these difficult amine compounds.

Some basic compounds, such as amines, are prone to adsorption in the GC system, including the analytical column. The Stabilwax®-DB column is specially treated for better recovery and quantitation of amines (**Figure 5**).

Figure 5

*Improved Recovery and Quantitation of Primary Amines
(low MW) on a Stabilwax®-DB Column*

1. trimethylamine
2. dimethylamine
3. ethylamine
4. methylamine
5. isopropylamine
6. n-propylamine
7. tert-butylamine
8. diethylamine
9. sec-butylamine



30m, 0.53mm ID, 1.0µm Stabilwax®-DB (cat.# 10855)
1.0µl direct injection of low molecular weight amines in water.
Oven temp.: 45°C isothermal; **Inj. & det. temp.:** 250°C; **Carrier gas:** hydrogen;
Linear velocity: 40cm/sec. (flow rate: 5cc/min.); **FID sensitivity:** 1 x 10⁻¹¹ AFS

Rtx®-Wax Columns for Difficult Active Compounds

- Minimum temperature of 20°C for analysis of volatiles.
- Excellent efficiency and inertness for aldehydes.
- Fast BTEX analyses.
- Thermally stable to 250°C.

The Rtx®-Wax column features a bonded, highly inert PEG stationary phase that provides excellent selectivity for compounds ranging in volatility, molecular weight, and polarity, and can even resolve aromatic isomers. It has a low minimum operating temperature (20°C) for analysis of volatile compounds. Crossbond® cross-linking, or bonding of the stationary phase, provides thermal stability at higher temperatures for analysis of higher boiling compounds. Compared to other available Carbowax® columns, the crossbonded Rtx®-Wax column provides a vast operational temperature range, excellent efficiency, selectivity, and superior inertness. These advantages make it a logical improvement for all Carbowax® applications.

Benefits of Low Temperature Analyses

Many PEG columns undergo a solid-liquid phase transition at temperatures below 40°C, which results in loss of efficiency, reduced sample capacity, and poor retention time reproducibility. The new Rtx®-Wax column can sustain 20°C for limited periods before solidification of the stationary phase occurs. This is especially advantageous for applications involving purge-and-trap and headspace analyses. Volatile components can be cold-trapped onto the column and elute as sharp, symmetrical, well-resolved peaks because column efficiency is still maintained at this low temperature. **Figures 6A and 6B** demonstrate the difference in PEG column performance at 20°C. The Rtx®-Wax column provides better column efficiency and resolution of the analytes, which demonstrates that it is far more suitable for low temperature analyses.

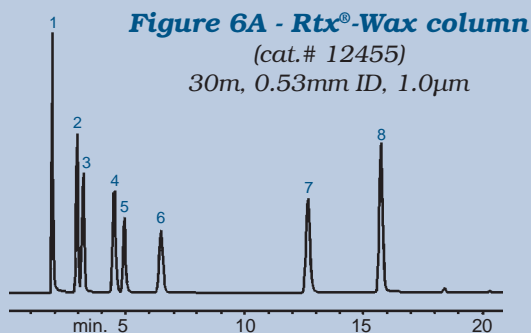
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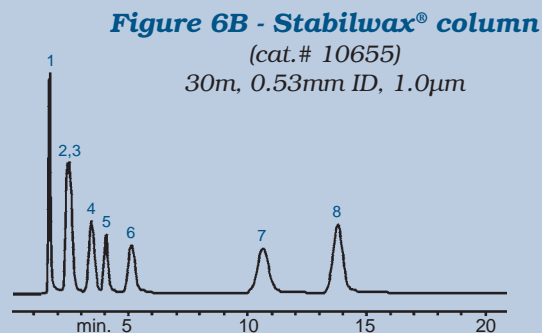
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Figures 6A and 6B
Rtx®-Wax Columns Maintain Efficiency at 20°C



1. acetaldehyde
2. methanol
3. acetone
4. ethyl formate
5. ethanol
6. ethyl acetate
7. n-propanol
8. isobutanol



0.1µl split injection of volatile solvents.
Oven temp.: 20°C (hold 10 min.) to 100°C @ 5°C/min.;
Inj. & det. temp.: 200°C; Carrier gas: helium; Linear velocity: 40cm/sec. set @ 20°C;
FID sensitivity: 8 x 10⁻¹¹ AFS; Split ratio: 10:1

Excellent Inertness for Aldehydes

Most PEG columns can effectively analyze alcohols, esters, and acids, but some exhibit peak-tailing with aldehydes. The Rtx®-Wax column is inert to aldehydes as well because the stationary phase undergoes an extensive purification procedure. Peak tailing of some straight-chain aldehydes is apparent on a typical bonded PEG column, but is absent on the highly inert Rtx®-Wax column (**Figures 7A and 7B**).

Figures 7A and 7B

Rtx®-Wax Column Exhibits Excellent Symmetrical Peak Shape for Aldehydes

1. ethanal
2. propanal
3. butanal
4. pentanal
5. hexanal
6. heptanal
7. octanal
8. nonanal
9. decanal
10. undecanal

Figure 7A - Rtx®-Wax Column

(cat. # 12438)

30m, 0.25mm ID, 0.50µm

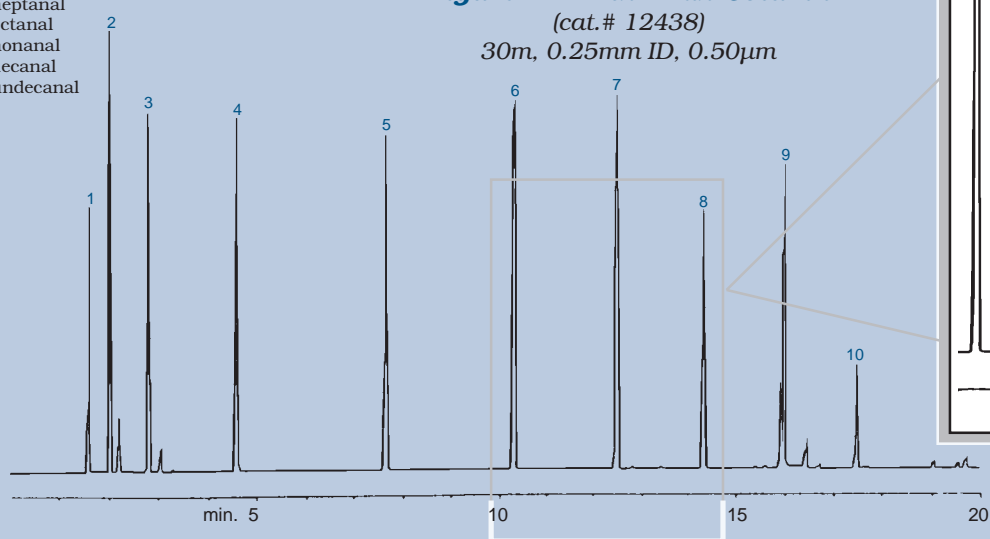
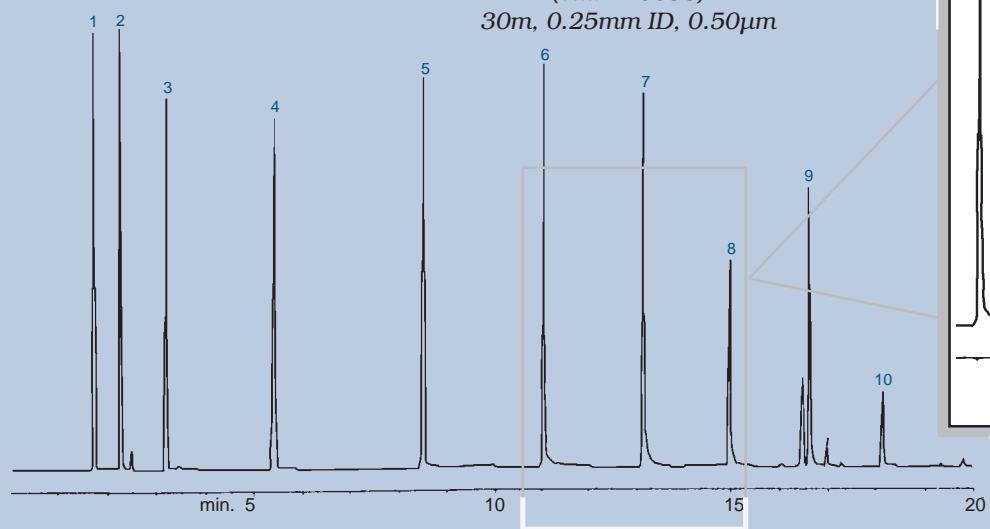


Figure 7B - Stabilwax® Column

(cat. # 10638)

30m, 0.25mm ID, 0.50µm



split injection of C2-C11 aldehyde mixture.

on-column concentration: 250ng;

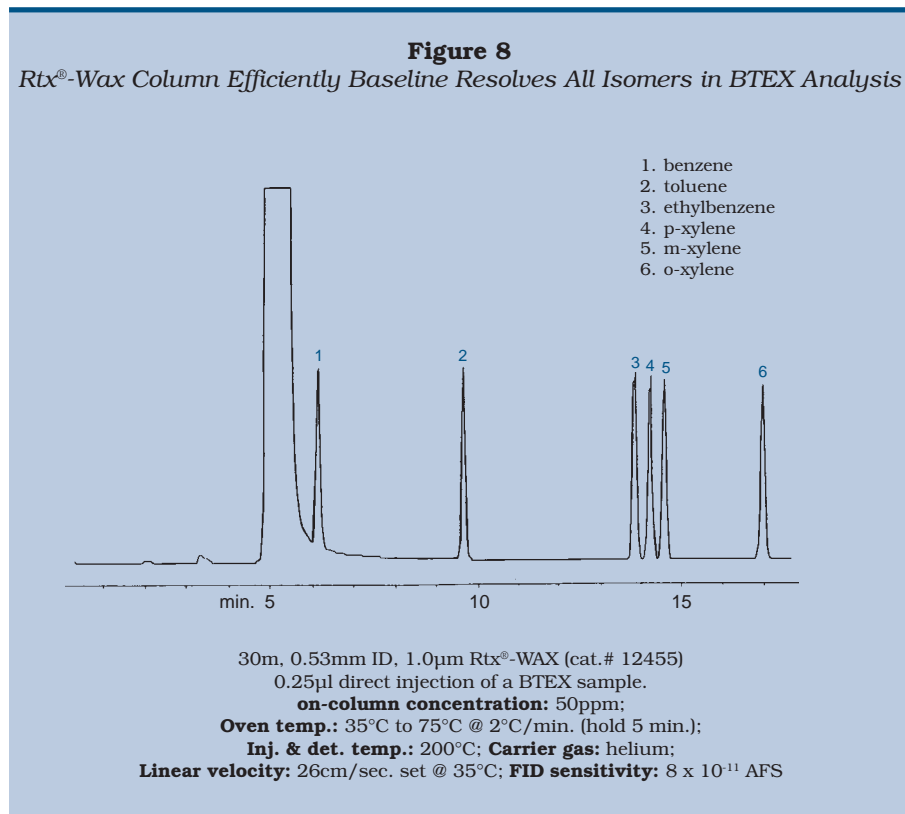
Oven temp.: 40°C (hold 5 min.) to 200°C @ 10°C/min.;

Inj. & det. temp.: 200°C; Carrier gas: hydrogen; Linear velocity: 35cm/sec. set @ 40°C;

FID sensitivity: 8×10^{-11} AFS; Split ratio: 100:1

All Components Resolved for BTEX isomers

The Rtx®-Wax column offers the same selectivity as other Carbowax® columns for isomers of substituted aromatics. This is useful for BTEX analyses requiring the quantitation of the individual xylene isomers. **Figure 8** illustrates that all components in the BTEX analysis are resolved. The meta-, para-, and ortho-xylene are baseline resolved in just 17 minutes.



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FAMEWAX™ Columns for FAME Analysis

- Fast, efficient analyses.
- Thermally stable to 250°C.
- Meets AOCS and AOAC method requirements.

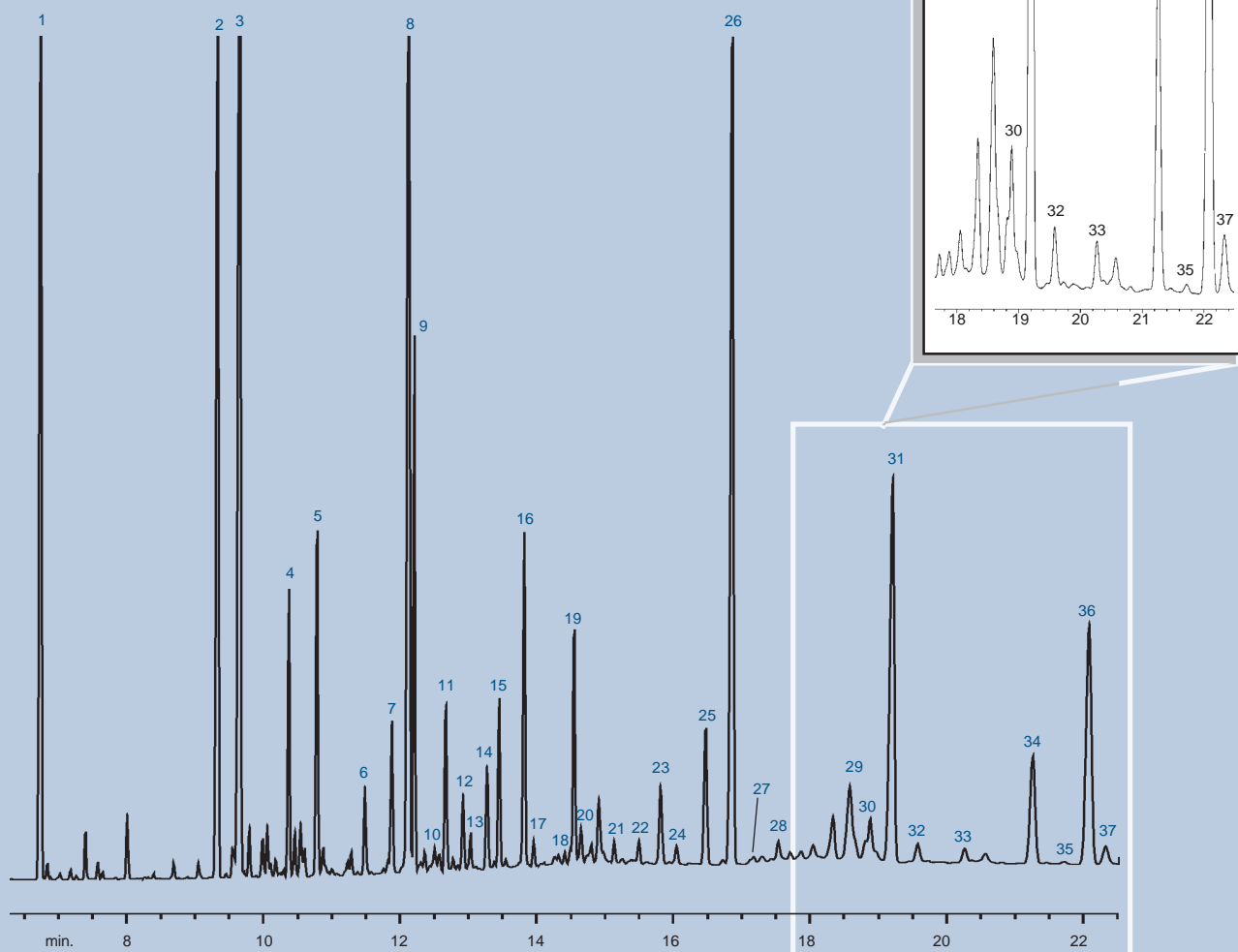
The FAMEWAX™ column provides superb stationary phase consistency, excellent column efficiency, and thermal stability to 250°C. The elution order of complex polyunsaturated fatty acid methyl esters (FAMES) is comparable to that on other Carbowax® columns, but baseline resolution is achieved in significantly less time.

Capillary column performance requirements for PUFA analysis are specified in American Oil Chemists Society (AOCS) and Association of Official Analytical Chemists (AOAC) methods. The AOCS Method CE 1b-89 *FAMES analysis by Capillary GLC* requires baseline resolution of C21:5n3 and C23:0 (internal standard [IS]), and C24:0 and C22:6n3 (DHA). The AOAC Official Method #991.39 *Fatty Acids in Encapsulated Fish Oils and Fish Oil Methyl and Ethyl Esters* requires the same elution pattern as Carbowax® 20M and the additional resolution of C23:0(IS) from C22:4n6.

FAMEWAX™ columns meet all the criteria listed in the methods in significantly less time, with faster flow and temperature program rates than other Carbowax® columns. The menhaden oil PUFA analysis on a FAMEWAX™ column in **Figure 9** shows that C21:5n3 and C23:0 (IS) are well resolved, as are C24:0, C22:6n3 (DHA), and C24:1n9, with a total analysis time of only 22 minutes. The same analysis on another PEG column typically used for FAMES analysis shows peaks C21:5n3 and C23:0 are not baseline resolved, nor are C22:6n3 and C24:1n9. To achieve resolution of these components on this other column, the program rate must be decreased to 2°C or 3°C/minutes, which increases the analysis time by 59%!

Figure 9
FAMEWAX™ Columns Meet the Resolution Criteria of AOCS and AOAC
Methods and Cut Analysis Time By Up To 50%

For peak identifications, please
refer to Figure 10.



30m, 0.25mm ID, 0.25µm Famewax™ (cat.# 12497).
0.8µl split injection of menhaden oil PUFA with C23:0 (IS).
On-column concentration: 100-150ng.

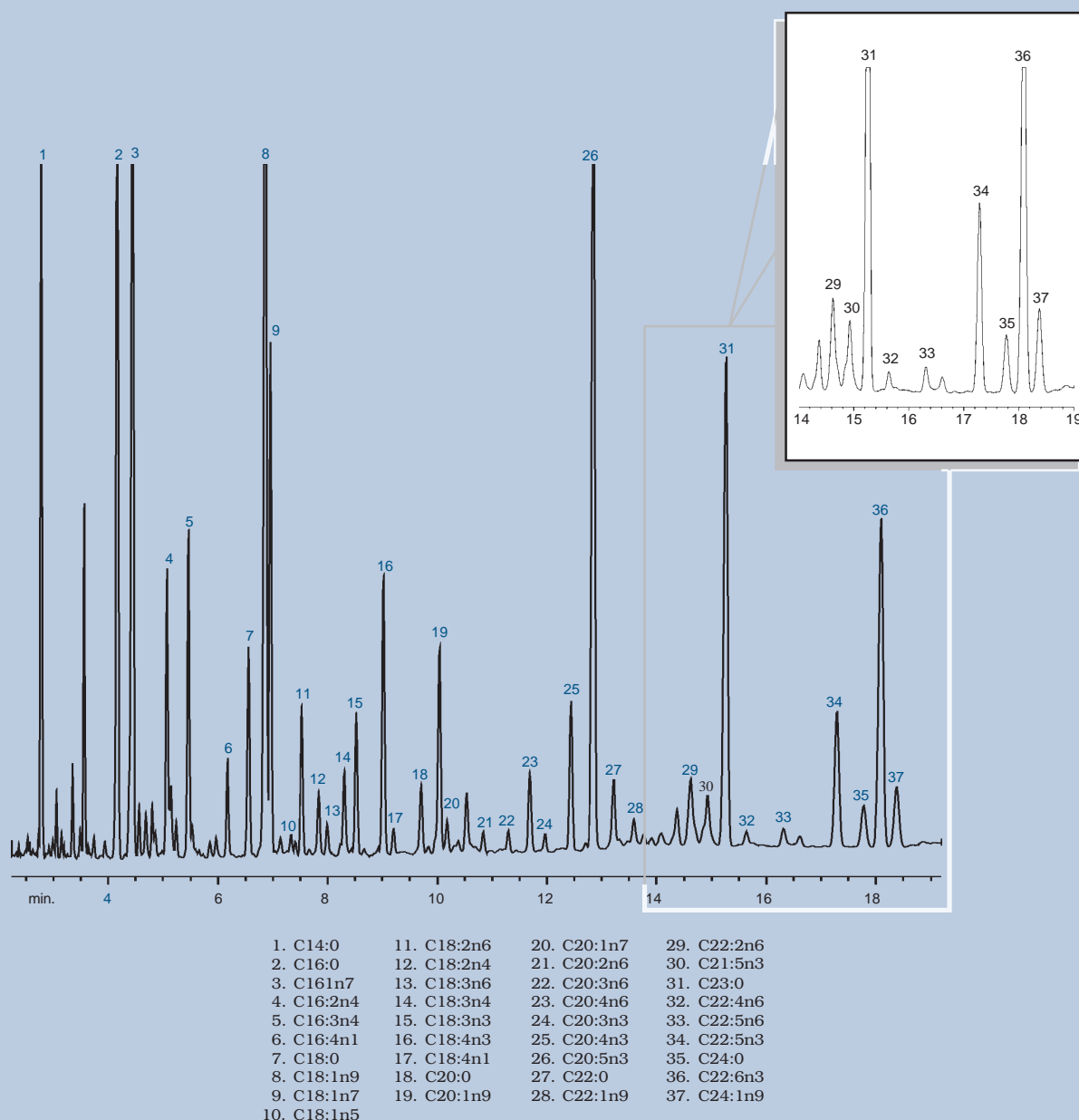
Oven temp.: 120°C to 220°C @ 7°C/min. (hold 20 min.);

Inj./det. temp.: 220°C; **Carrier gas:** hydrogen; **Linear velocity:** 60cm/sec. @ 120°C;

FID sensitivity: 8×10^{-11} AFS **Split ratio:** 50:1.

The 0.32mm ID FAMEWAX™ column also meets the resolution criteria for PUFA analysis in less time. Plus, the larger inner diameter columns provide 4 to 5 times more sample capacity (400–500ng vs. 50–100ng) to minimize overloading of more concentrated samples without losing column efficiency. **Figure 10** shows an analysis of menhaden oil FAMES completed in less than 19 minutes on a 30m, 0.32mm ID, 0.25µm FAMEWAX™ column.

Figure 10
0.32mm ID FAMEWAX™ Columns Allow More Concentrated Samples to be Analyzed in Less Than 20 Minutes



30m, 0.32mm ID, 0.25µm FAMEWAX™ (cat.# 12498). 0.5µl each split injection of menhaden oil PUFA & Omegawax® test mix.

On-column concentration: 200-350ng.

Oven temp.: 190°C (hold 4 min.) to 225°C @ 4°C/min. (hold 15 min.); **Inj./det. temp.:** 225°C/250°C;

Carrier gas: helium; **Linear velocity:** 35cm/sec. @ 190°C;

FID sensitivity: 8×10^{-11} AFS; **Split ratio:** 50:1.

Product List

Stabilwax® (Fused Silica) Crossbond® Carbowax® - provides oxidation resistance; Stable to 250°C

ID	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.25mm	0.10	40 to 250/260°C	10605	10608	10611
	0.25	40 to 250/260°C	10620	10623	10626
	0.50	40 to 250/260°C	10635	10638	10641
0.32mm	0.10	40 to 250/260°C	10606	10609	10612
	0.25	40 to 250/260°C	10621	10624	10627
	0.50	40 to 250/260°C	10636	10639	10642
0.53mm	1.00	40 to 240/250°C	10651	10654	10657
	0.10	40 to 250/260°C	10607	10610	10613
	0.25	40 to 250/260°C	10622	10625	10628
	0.50	40 to 250/260°C	10637	10640	10643
	1.00	40 to 240/250°C	10652	10655	10658
	1.50	40 to 230/240°C	10666	10669	10672
	2.00	40 to 220/230°C	10667	10670	

Stabilwax®-DA (Fused Silica) Crossbond® Carbowax® for acidic compounds; Stable to 250°C

ID	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.25mm	0.10	40 to 250/260°C	11005	11008	11011
	0.25	40 to 250/260°C	11020	11023	11026
	0.50	40 to 250/260°C	11035	11038	11041
0.32mm	0.10	40 to 250/260°C	11006	11009	11012
	0.25	40 to 250/260°C	11021	11024	11027
	0.50	40 to 250/260°C	11036	11039	11042
0.53mm	1.00	40 to 240/250°C	11051	11054	11057
	0.10	40 to 250/260°C	11007	11010	11013
	0.25	40 to 250/260°C	11022	11025	11028
	0.50	40 to 250/260°C	11037	11040	11043
	1.00	40 to 240/250°C	11052	11055	11058
	1.50	40 to 230/240°C	11062	11065	11068

Stabilwax®-DB (Fused Silica) Crossbond® Carbowax® for amines and basic compounds; Stable to 220°C

ID	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.25mm	0.25	40 to 210/220°C	10820	10823	
	0.50	40 to 210/220°C		10838	
0.32mm	0.25	40 to 210/220°C	10821	10824	
	0.50	40 to 210/220°C		10839	
	1.00	40 to 210/220°C		10854	10857
0.53mm	0.50	40 to 210/220°C		10840	
	1.00	40 to 210/220°C	10852	10855	10858
	1.50	40 to 210/220°C		10869	

Rtx®-Wax (Fused Silica) Crossbond®; Stable to 250°C

ID	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.25mm	0.10	20 to 250°C	12405	12408	
	0.25	20 to 250°C	12420	12423	12426
	0.50	20 to 250°C	12435	12438	12441
0.32mm	0.10	20 to 250°C	12406	12409	
	0.25	20 to 250°C	12421	12424	12427
	0.50	20 to 250°C	12436	12439	12442
0.53mm	1.00	20 to 240/250°C	12451	12454	12457
	0.25	20 to 250°C	12422	12425	
	0.50	20 to 250°C	12437	12440	12443
	1.00	20 to 240/250°C	12452	12455	12458
ID	df (µm)	temp. limits	10-Meter	20-Meter	
0.10mm	0.10	20 to 250°C	41601	41602	
	0.20	20 to 240/250°C	41603	41604	

MXT®-WAX (Silcosteel®) Crossbond® Carbowax® - provides oxidation resistance; Stable to 250°C

ID	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.28mm	0.25	40 to 250°C	70621	70624	70627
	0.50	40 to 250°C	70636	70639	70642
	1.00	40 to 240°C	70651	70654	70657
0.53mm	0.25	40 to 250°C	70622	70625	70628
	0.50	40 to 250°C	70637	70640	70643
	1.00	40 to 240°C	70652	70655	70658
	1.50	40 to 230°C	70666	70669	70672
	2.00	40 to 220°C	70667	70670	

FAMEWAX™ (Fused Silica) Crossbond®; Stable to 250°C

ID	df (µm)	temp. limits	30-Meter
0.25mm	0.25	20 to 250°C	12497
0.32mm	0.25	20 to 250°C	12498
0.53mm	0.50	20 to 250°C	12499

Analytical Reference Materials

Fatty Acid Methyl Esters

Neat fatty acid methyl esters can be used to prepare specific mixtures not commercially available. These products are of the highest purity available, typically 99% by GC/FID analysis. Each compound is packaged under a nitrogen blanket to ensure product stability. A Certificate of Analysis is provided with each ampul.

Carbon Number	Compound	CAS #	quantity	cat.#
C6:0	Methyl caproate	106-70-7	100mg	35037
C7:0	Methyl heptanoate	106-73-0	100mg	35038
C8:0	Methyl caprylate	111-11-5	100mg	35039
C9:0	Methyl nonanoate	1731-84-6	100mg	35040
C10:0	Methyl caprate	110-42-9	100mg	35041
C11:0	Methyl undecanoate	1731-86-8	100mg	35042
C12:0	Methyl laurate	111-82-0	100mg	35043
C13:0	Methyl tridecanoate	1731-88-0	100mg	35044
C14:0	Methyl myristate	124-10-7	100mg	35045
C14:1 D 9 cis	Methyl myristoleate	56219-06-8	100mg	35046
C15:0	Methyl pentadecanoate	7162-64-1	100mg	35047
C16:0	Methyl palmitate	112-39-0	100mg	35048
C16:1 D 9 cis	Methyl palmitoleate	1120-25-8	100mg	35049
C17:0	Methyl heptadecanoate	1731-92-6	100mg	35050
C18:0	Methyl stearate	112-61-8	100mg	35051
C18:1 D 9 cis	Methyl oleate	112-62-9	100mg	35052
C18:2 D 9,12 cis	Methyl linoleate	112-63-0	100mg	35053
C18:3 D 9,12,15 cis	Methyl linolenate	301-00-8	100mg	35054
C19:0	Methyl nonadecanoate	1731-94-8	100mg	35055
C20:0	Methyl arachidate	1120-28-1	100mg	35056
C20:1 D 11 cis	Methyl eicosenoate	2390-09-2	100mg	35057
C20:2 D 11,14 cis	Methyl eicosadienoate	2463-02-7	100mg	35058
C20:3 D 11,14,17 cis	Methyl eicosatrienoate	55682-88-7	100mg	35059
C20:4 D 5,8,11,14 cis	Methyl arachidonate	2566-89-4	100mg	35060
C21:0	Methyl heneicosanoate	6064-90-0	100mg	35061
C22:0	Methyl behenate	929-77-1	100mg	35062
C22:1 D 13 cis	Methyl erucate	1120-34-9	100mg	35063
C24:0	Methyl lignocerate	2442-49-1	100mg	35064
C24:1 D 15 cis	Methyl nervonate	2733-88-2	100mg	35065

**For pricing & ordering information, please call
Restek's Customer Service Department at 800-356-1688, ext. 3.**

Analytical Reference Materials

Quantitative Fatty Acid Methyl Ester (FAME) Mixtures

Analysis of fatty acid profiles for food products has come to prominent attention in recent years. While these analyses have been performed for some time, the focus on healthy living has been increasing the demand for food testing. More consumers are concerned about the level of saturated and unsaturated fatty acids in the foods they eat.

To help analytical chemists identify and calibrate their instruments, we now offer several common fatty acid methyl ester mixtures. The mixtures, listed in the following chart, can be used for quantitation (AOCS Method CE 1-62) and correspond to the following types of oils:

AOCS #1: Suitable for corn, poppy seed, cotton seed, soybean, walnut, safflower, sunflower, rice, bran, and sesame.

AOCS #2: Suitable for linseed, perilla, hempseed, and rubberseed.

AOCS #3: Suitable for peanut, rapeseed, and mustard seed.

AOCS #4: Suitable for olive, teaseed, and neatsfoot.

AOCS #5: Suitable for coconut, palm kernel, babassu, and ouri-curi.

AOCS #6: Suitable for lard, beef or mutton tallow, and palm.

FAME #13: Suitable for mustard seed oil.

FAME #14: Suitable for cocoa butter.

FAME #15: Suitable for peanut oil.

Composition of each compound listed as a weight/weight % basis.

Packaged by volume to contain 50+mg of mixture.		cat. # 35022	cat. # 35023	cat. # 35024	cat. # 35025	cat. # 35026	cat. # 35027	cat. # 35028	cat. # 35029	cat. # 35030	cat. # 35031	cat. # 35032	cat. # 35033	cat. # 35034	cat. # 35035	cat. # 35036
Chain	Compound	AOCS #1	AOCS #2	AOCS #3	AOCS #4	AOCS #5	AOCS #6	NIH-A	NIH-B	NIH-C	NIH-D	NIH-E	NIH-F	FAME #13	FAME #14	FAME #15
6:00	methyl caproate															
7:00	methyl heptanoate															
8:00	methyl caprylate					7.0				1.5		6.3				
9:00	methyl nonanoate															
10:00	methyl caprate					5.0				3.0		9.1				
11:00	methyl undecanoate															
12:00	methyl laurate					48.0				6.0		12.1				
13:00	methyl tridecanoate															
14:00	methyl myristate			1.0		15.0	2.0	25.0	4.0	12.0	11.8	23.3	2.5		0.1	
15:00	methyl pentadecanoate															
16:00	methyl palmitate	6.0	7.0	4.0	11.0	7.0	30.0	10.0	40.0	19.4	23.6	49.2	4.2	3.0	26.3	10.0
16:01	methyl palmitoleate						3.0				6.9			1.0	0.4	
17:00	methyl heptadecanoate															0.3
18:00	methyl stearate	3.0	5.0	3.0	3.0	3.0	14.0	65.0	56.0	24.9	13.1		7.3	2.0	33.7	3.0
18:01	methyl oleate	35.0	18.0	45.0	80.0	12.0	41.0				44.6			20.0	34.3	50.0
18:02	methyl linoleate	50.0	36.0	15.0	6.0	3.0	7.0							15.0	3.1	30.0
18:03	methyl linolenate	3.0	34.0	3.0			3.0							10.0	0.2	
20:00	methyl arachidate	3.0		3.0						33.2			13.6	1.0	1.3	1.5
20:01	methyl eicosenoate													10.0	0.1	1.5
20:02	methyl eicosadienoate													2.0		
22:00	methyl behenate			3.0									25.4	1.0	0.2	3.0
22:01	methyl erucate			20.0										30.0		
22:02	methyl docosadienoate													2.0		
24:0	methyl lignocerate			3.0									47.0	1.0		1.0
24:1	methyl nervonate													2.0		

The NIH mixtures are composed to the original compositions specified by the National Institutes of Health per Horning, et al., *Journal of Lipid Research*, Volume 5, 20-27, 1964.

Important Information From Restek:

We certify that all raw materials used have a minimum purity of 99%. The exact composition of each mixture is determined by precise gravimetric techniques based on a weight/weight % basis and is confirmed using high resolution capillary gas chromatography. A Certificate of Analysis is supplied with each product and lists mixture composition, analysis conditions, and includes a sample chromatogram. Products are packaged by volume and are guaranteed to contain a minimum amount of 50mg/ampul.

Improper storage or handling after opening may result in degradation of the unsaturated compounds, for which we cannot be responsible. All materials must be stored under nitrogen at -18°C to prevent degradation.

Custom fatty acid methyl ester mixtures are also available. Call (800) 356-1688 for details.

Composition of each compound listed as a weight/weight % basis.

Packaged by volume to contain 50+mg of mixture.		FAME #1 cat. # 35010	FAME #2 cat. # 35011	FAME #3 cat. # 35012	FAME #4 cat. # 35013	FAME #5 cat. # 35014	FAME #6 cat. # 35015	FAME #7 cat. # 35016	FAME #8 cat. # 35017	FAME #9 cat. # 35018	FAME #10 cat. # 35019	FAME #11 cat. # 35020	FAME #12 cat. # 35021
Chain	Compound												
6:0	methyl caproate		20					20					
7:0	methyl heptanoate							20				20	
8:0	methyl caprylate		20	20				20					
9:0	methyl nonanoate							20				20	
10:0	methyl caprate		20	20				20					
11:0	methyl undecanoate								20			20	
12:0	methyl laurate		20	20					20				
13:0	methyl tridecanoate								20			20	20
14:0	methyl myristate		20	20					20				
15:0	methyl pentadecanoate								20			20	20
16:0	methyl palmitate	20		20	20					20			
16:1	methyl palmitoleate					20							
17:0	methyl heptadecanoate									20			20
18:0	methyl stearate	20			20					20	20		
18:1	methyl oleate	20				20							
18:2	methyl linoleate	20											
18:3	methyl linolenate	20											
19:0	methyl nonadecanoate									20	20		20
20:0	methyl arachidate				20		20			20	20		
20:1	methyl eicosenoate					20	20						
20:2	methyl eicosadienoate						20						
20:3	methyl homo gamma linolenate						20						
20:4	methyl arachidonate						20						
21:0	methyl heneicosanoate										20		20
22:0	methyl behenate				20						20		
22:1	methyl erucate					20							
24:0	methyl lignocerate				20								
24:1	methyl nervonate					20							

Restek's Customer Response Team

.....



(L to R) Seated: Sherri Comly, Sharon Paloskey;
Standing: Cindy Ross, Kim Holliday, Tracy Hazenstab

Satisfying your needs is our number one priority! The recent expansion of our Customer Response Team gives you a 24-hour turnaround from the time we receive a literature request until it ships out the door. We continually update our database with information about your communications, so we can meet your specific needs more easily. Customers are contacted through our follow-up program to keep them informed and to ensure that overnight shipments were delivered on time. We also schedule and organize our technical service department and act as a liaison for our outside technical sales people.

Whether your chromatography problem is simple or complex, call the Customer Response Team and we will do everything we can to help you find a solution.

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A Guide to Preparing and Analyzing Chlorinated Pesticides.....



Inside:

***Extraction Methods
for Liquid, Solid,
and Biota Samples***

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***Sample Cleanup
Methods***

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***Analysis of Chlorinated
Pesticides***

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Summary

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The analysis of chlorinated pesticides (as stated in US Environmental Protection Agency (EPA) Methods 8081, 508, and 608) and polychlorinated biphenyls (PCBs) (as stated in US EPA Methods 8081 and 8082), are some of the most common tests performed by environmental laboratories. However, many laboratories struggle with them because the samples often are highly contaminated with non-target compounds such as lipids and hydrocarbons, and the methods require rigorous quality control. Several techniques, used in combination with Restek's Rtx[®]-CLPesticides column and Rtx[®]-CLPesticides 2 column, can help you more simply perform these analyses.

The compounds addressed in these methods are listed in Table I. The table includes additional compounds typically analyzed using the same methods. Herbicide compounds (US EPA Method 8151) also are listed because many laboratories use the same instrument to analyze both pesticides and herbicides. Although the separation of herbicides is included in this guide, the extraction of them, which is significantly different than for pesticides, is not included. If you are involved in the preparation and analysis of the herbicides and would like more information, please contact Restek's technical service.

Table I

US EPA 508

aldrin	chlorneb	etridiazole
Aroclor [®] 1016*	chlorobenzilate	heptachlor
Aroclor [®] 1221*	chlorothalonil	heptachlor epoxide
Aroclor [®] 1232*	DCPA	hexachlorobenzene
Aroclor [®] 1242*	4,4'-DDD	methoxychlor
Aroclor [®] 1248*	4,4'-DDE	cis-permethrin
Aroclor [®] 1254*	4,4'-DDT	trans-permethrin
Aroclor [®] 1260*	dieldrin	propachlor
α-BHC (α-HCH)	endosulfan I	technical chlordane*
β-BHC (β-HCH)	endosulfan II	trifluralin
δ-BHC (δ-HCH)	endosulfan sulfate	toxaphene*
γ-BHC (γ-HCH, lindane)	endrin	
α-chlordane	endrin aldehyde	
γ-chlordane	endrin ketone	

US EPA 608

aldrin	δ-BHC (δ-HCH)	endrin aldehyde
Aroclor [®] 1016*	γ-BHC (γ-HCH, lindane)	endrin ketone
Aroclor [®] 1221*	4,4'-DDD	heptachlor
Aroclor [®] 1232*	4,4'-DDE	heptachlor epoxide
Aroclor [®] 1242*	4,4'-DDT	technical chlordane*
Aroclor [®] 1248*	dieldrin	toxaphene*
Aroclor [®] 1254*	endosulfan I	
Aroclor [®] 1260*	endosulfan II	
α-BHC (α-HCH)	endosulfan sulfate	
β-BHC (β-HCH)	endrin	

US EPA 8081

aldrin	α-BHC (α-HCH)	endrin aldehyde
Aroclor [®] 1016*	β-BHC (β-HCH)	endrin ketone
Aroclor [®] 1221*	δ-BHC (δ-HCH)	heptachlor
Aroclor [®] 1232*	γ-BHC (γ-HCH, lindane)	heptachlor epoxide
Aroclor [®] 1242*	α-chlordane	methoxychlor
Aroclor [®] 1248*	γ-chlordane	technical chlordane*
Aroclor [®] 1254*	dieldrin	toxaphene*
Aroclor [®] 1260*	endosulfan I	
4,4'-DDD	endosulfan II	
4,4'-DDE	endosulfan sulfate	
4,4'-DDT	endrin	

*Multi-component standards.

For tech support, call
800-356-1688, ext. 4
 (814-353-1300, ext. 4)

Table I, cont.**US EPA 8081 Additional Compounds**

alachlor	metalachlor	pentachloronitrobenzene
atrazine	metribuzin	simazine
cyanozine		

US EPA 8151 (herbicides)

acidfluorfen	DCPA	4-nitrophenol
bentazon	dicamba	pentachlorophenol
chloramben	3,5-dichlorobenzoic acid	picloram
2,4-D	dichloroprop	2,4,5-T
dalapon	dinoseb	2,4,5-TP (Silvex)
2,4-DB	MCPA	
DCAA	MCPP	

Common Surrogates

2,4-DA (herbicide)	decachlorobiphenyl	2,4,5,6-tetrachloro-m-xylene
--------------------	--------------------	------------------------------

Extraction Methods for Liquid, Solid, and Biota Samples

All of the pesticide compounds listed in Table I, except the US EPA 8151 herbicides, are extracted under neutral conditions using a variety of organic solvents. There are several sample extraction methods that can be applied, but the most common will be addressed here.

Liquid Samples

For liquid samples, you can use either separatory funnel extraction (US EPA Method 3510) or automated liquid-liquid extraction (US EPA Method 3520). In comparison, separatory funnel extraction is faster and less expensive to set up, but requires continuous attention. Automated liquid-liquid extractors can operate unattended, but are more expensive. For some methods, if analyte recovery is lower than allowed, you must re-extract the sample by separatory funnel. Alternatively, if the sample forms an emulsion to the degree that acceptable solvent recovery is not possible using a separatory funnel, then some methods require liquid-liquid extraction.

According to US EPA Method 3535, solid phase extraction (SPE) can be used to extract pesticide compounds from aqueous samples.

Separatory Funnel Extraction

For separatory funnel extraction, measure up to 1L of sample into a 2L separatory funnel, and check the pH. Adjust the pH to neutral using hydrochloric acid or sodium hydroxide, depending on the starting pH. Avoid using sulfuric acid (see *Sulfuric Acid Cleanup* on page 8). If adjustment is necessary, record on your sample tracking paperwork.

Extract the sample by adding 60mL of dichloromethane and shaking for two minutes. It is critical to shake all samples in the same manner or you may see variations in extraction efficiency—the best way to ensure consistency is to use a mechanical separatory funnel shaker. The dichloromethane settles to the bottom of the separatory funnel and then is decanted through a sodium sulfate tube into a collection vessel such as a Kuderna-Danish (KD) concentrator or into a TurboVap® or RapidVap® container if using automated concentrators. This step is repeated two more times to achieve quantitative recovery of all analytes (collect all three extractions into the same collection vessel).

Use SPE (US EPA Method 3535), separatory funnel extraction (US EPA Method 3510), or automated liquid-liquid extraction (US EPA Method 3520) for liquid samples.



SPE allows very fast extraction times and low solvent volumes; it also is easily used in the field.

For ordering information, see product listings beginning on page 18.

Removing water from the dichloromethane with sodium sulfate is critical before the extract is concentrated to final volume. Dichloromethane can hold approximately 1 mL of water per liter. If water remains in the extract, it will partition out of the extract when the volume is reduced. If this occurs, either the dichloromethane will evaporate first, leaving only water in the collection vessel, or a two-layer extract will form. In either event, the recoveries of the analytes will be lower than desired, and the presence of water will interfere with gas chromatographic (GC) analysis.

The best way to remove the water is to decant the dichloromethane extract through granular sodium sulfate held in a funnel with a high-quality grade (Whatman 541) filter paper or glass wool. Approximately 30g of sodium sulfate is sufficient for most samples. This step must not be skipped! Some methods may call for powdered sodium sulfate, but some analytes are adsorbed to the smaller particles, so only a 10-60 mesh granular sodium sulfate or equivalent should be used. It also is important that this material be free from organic contaminants, so it should be purchased as ACS pesticide residue grade in glass containers. If purchased in bulk packages where exposure to plastic is an issue, bake in a muffle furnace. To bake the sodium sulfate, spread it no more than 1-inch thick into an appropriate container and place into a muffle furnace, baking at 400°C for a minimum of two hours. After this time, place the sodium sulfate into a glass container while still hot, and cap the container to keep the material from resorbing contaminants from the atmosphere. If a muffle furnace is not available, wash the sodium sulfate or extract it with dichloromethane prior to use. This technique is extremely wasteful of solvent, making the muffle furnace preferable.

Liquid-Liquid Extraction

Liquid-liquid extraction offers unattended extraction once the samples are ready and the solvent is added. Extraction is performed under neutral conditions and the recoveries are excellent for chlorinated pesticides. Set up of the extractors should be done following manufacturer specifications. Due to the extended contact time of the organic compounds with the glass surfaces, reactive compounds could breakdown if these surfaces became contaminated. Although, with the use of proper washing procedures, this is uncommon. As with the separatory funnel technique, the use of granular sodium sulfate is important to yield a dry dichloromethane extract.

Liquid-liquid extractors are available in two versions, conventional and accelerated. The accelerated type uses a hydrophobic membrane to separate the aqueous and the organic phases, and the extraction time can be cut to $\frac{1}{3}$ or $\frac{1}{4}$ of the conventional extractor time. These membranes are expensive and it is important to analyze the cost versus the number of samples extracted to determine if there is a benefit to using this technique.

Solid Phase Extraction (SPE)

Finally, SPE also is used for the extraction of pesticide and herbicide compounds from aqueous samples (US EPA Methods 3535, 508, and 515). When using SPE, it is extremely important to follow the manufacturer's recommendations on the use of the material. There are several manufacturers of C18 tubes and disks, which are the typical media used for these compounds, and the extraction steps will vary somewhat depending on the manufacturer. In general, the biggest drawbacks with SPE are the plugging of the disk or tube with suspended solids and the breakthrough of targeted organics; therefore this extraction method works most reliably if contamination levels and solids are low. SPE allows very fast extraction times and low solvent volumes; it also is easily used in the field.

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Soil Samples

For soil samples, soxhlet or ultrasonic extraction have been the most common extraction methods; although pressurized fluid, microwave, and supercritical fluid extraction (SFE) are used as well.

Pressurized fluid extraction (US EPA Method 3545) runs unattended, but has some sample size limitations. Generally, no more than 10g of sample can be extracted without using multiple vessels, so detection limits may be compromised for certain analytical methods. It is important to take this into consideration when evaluating the use of either pressurized fluid or microwave extraction.

Although not currently cited by the US EPA, microwave extraction can be useful for automated extraction as well. Microwave extraction extracts 12 samples simultaneously, but does require slightly more operator handling than the pressurized fluid extraction instruments. The instrumentation is less expensive than the pressurized fluid instrumentation, but lack of an EPA method has limited the use of this technique in the US.

Supercritical fluid extraction has been promoted for a number of years as a "solventless" extraction technique for environmental samples. SFE was added to SW-846 as Method 3560, but its application is limited. SFE suffers from severe matrix-related variation, resulting in the need to modify the SFE conditions depending on soil type, water content, sample size, and the type of analytes. This ultimately requires additional sample preparation prior to the actual extraction. These requirements, added to the high cost of these instruments, has virtually precluded the use of SFE for environmental sample preparation.



Sonication or Soxhlet Extraction

Sonication or soxhlet extraction works well for chlorinated pesticides and PCBs. Sonication is a faster technique, but requires constant operator attention. In both techniques, problems such as contamination are attributed to either contaminated reagents, especially sodium sulfate, or poor laboratory practices being used when transferring sample extracts. Using sodium sulfate to remove water (described on page 4) is important. Mix the sample with sodium sulfate to achieve a sandy consistency prior to solvent addition. Using granular sodium sulfate is recommended because some of the pesticides will adsorb to the powdered material.

Solvent Selection

Since soil and biota samples are essentially wet particles, acetone and dichloromethane usually are used in a 1:1 combination as the extraction solvent. The acetone is needed to adequately penetrate into the soil particle so that compounds contained in the particle can be extracted. Several other solvent systems can be used for unique extractions, but generally this combination works for most applications. Use pesticide residue grade solvents for this application and run solvent assays to verify the material prior to its use. To perform a solvent assay, reduce 300 to 400mL of solvent to a final volume of 1mL, and exchange to hexane for analysis by GC/ECD (electron capture detection). The extract analysis should have no chromatographic peaks above 50% of the detection limit for any target compound.

Finally, with regards to solvent selection, it is important to note that dichloromethane will form hydrochloric acid spontaneously without a stabilizer present. There are two classes of stabilizers: stabilizers that keep hydrochloric acid from forming, and stabilizers that eliminate hydrochloric acid upon formation. Methanol and cyclohexane are used to stop hydrochloric acid from forming. If water samples are extracted with dichloromethane contain-

Soxhlet and ultrasonic extraction are the most common extraction methods for soil samples; although pressurized fluid, microwave, and supercritical fluid extraction can be used as well.

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Sample extract cleanup is probably the most important step in maintaining long-term instrument performance.

For ordering information, see product listings beginning on page 18.

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ing methanol as the stabilizer, the methanol will partition into the water, leaving an unstabilized extract. Hydrochloric acid forms quickly in unstabilized dichloromethane, and injection of an acidic solvent will result in reactivity of liners and columns. The second type of stabilizers are alkene compounds, which are used to reduce hydrochloric acid upon formation. It is desirable to use an alkene stabilizer that is low-boiling to prevent interference with early eluting target compounds.

Sample Cleanup Methods

Sample extract cleanup is probably the most important step in maintaining long-term instrument performance. Generally, when instrument problems arise, they are caused by exposure of the injection port and the column to contaminants in the sample extracts. While all of these contaminants cannot be eliminated, most can be reduced to levels where they become much less of an issue. Contained in many pesticide and PCB extracts are hydrocarbons, sulfur, phthalate esters, and lipids in the case of biota samples. Many of these compounds can be removed from the extract by one or more of the following techniques, with little additional cost or time, which usually can be recovered by an increase in instrumental stability, a decrease in instrument maintenance, and possible improvements in detection limits.

Sulfur and Lipid Contaminants: Gel Permeation Chromatography

Gel permeation chromatography (GPC) is a preparative-scale chromatographic method of separation based on molecular size. Since the target compounds are similar in molecular size, they elute as a band of material and are easily separated from lighter and heavier contaminants. For the pesticide and PCB extracts, GPC is a very efficient method for removing sulfur and lipids. GPC is the only cleanup technique cited here that requires considerable expense, and the processing time per sample is between 30 to 70 minutes. For these reasons, many laboratories choose not to use GPC. However, for soil and biota samples, GPC is the most prudent cleanup method.

Sulfur also can be eliminated using mercury or activated copper powder, but lipids are not as easily removed. Lipid content of biota extracts can be several orders of magnitude higher than that handled using SPE methods, so GPC is still a good alternative. If sample extracts with high lipid content are injected into the GC, the injection port and head of the column will quickly become contaminated, resulting in failure of continuing check standards.

US EPA Method 3640 details the requirements for GPC cleanup of extracts for pesticide and PCB analysis. If the sample is to be analyzed for PCBs only, the sulfuric acid cleanup (US EPA Method 3665) described on page 8 is more cost effective than Method 3640, but is not amenable to all the pesticides. When performing a GPC cleanup, verify the instrument retention time calibration on a daily basis or before processing the next batch of samples, whichever is less frequent. If a number of samples have been processed that contain large amounts of contamination, the front of the GPC column can become reactive. This is typically observed in the loss of 2,4,6-tribromophenol for semivolatile extracts, but it may not be as easily observed in the pesticide GPC standard. The use of a 2-3" guard column can prevent repacking of the 70g analytical column.

GPC columns also are very sensitive to slight changes in mobile phase composition (solvent variations). Because soil and biota samples typically are extracted using a solvent mixture, and dichloromethane is the lowest boiling solvent, it will evaporate first when the extract is concentrated. This leaves nearly 100% acetone in the concentration vessel. If dichloromethane is then added to adjust the extract to volume, significant amounts of acetone will be introduced into the GPC column. This will lead to "solvent shock" and the

formation of a void at the front of the column. This, in turn, will effect the retention times of the compounds eluting from the GPC column and ultimately result in the possibility of some target compounds being uncollected. Therefore, to avoid large amounts of acetone being applied to the column, it is critical that all extracts be reduced to as small a volume as possible prior to reconstitution in dichloromethane.

Polar Contaminants and Co-Extractants: Adsorbent SPE Tubes

Both Florisil® and silica adsorbents have been used since the 1960s for chromatographic cleanup and fractionation of environmental samples, especially those containing chlorinated pesticides. Florisil® is a magnesium silicate, while silica is manufactured from a sodium silicate sol. Originally tested and used in manually-packed, large open-column cleanup procedures, these adsorbents were found to be useful in retaining polar contaminants from soil and waste samples that had been extracted with organic solvents such as hexane. They are ideal for retaining co-extractants, such as phenols, that may interfere with GC analysis of pesticides, PCBs, and chlorinated hydrocarbons. Large Florisil® tubes also were used to fractionate pesticide groups based on small differences in polarity, by eluting with increasing percentages of polar solvents such as ethyl ether.¹ Testing based on this method is a standard QA procedure for pesticide-grade Florisil® adsorbent. All grades of these bulk adsorbents should be heat-activated at 130°C for 16 hours, stored in a sealed glass container, and cooled to room temperature before being manually packed into glass tubes.²

To increase laboratory efficiency and reduce the amount of solvents used for these processes, the US EPA has allowed the use of pre-packed SPE tubes containing Florisil® or silica packing. These small tubes are convenient to use, require less solvent, and still are effective. They will cleanup small volumes of pesticide-containing or chlorinated hydrocarbon-containing samples. They often are used after GPC cleanup, as recommended in SW 846. Details on the appropriate use and preparation of these cartridges is contained in SW 846 Method 3620B, 3630C, and in the CLP Pesticides Statement of Work (SOW).³

It is very important to evaluate each lot of tubes to ensure minimal background from the device itself, and to verify that the packing is at maximum activity level to maintain the expected retention capacity. These tubes are available with stainless steel or Teflon® frits to reduce interferences from phthalates, which may be extracted from typical polyethylene frit materials. Using adsorbent beds of 1g or more and slower gravity elution of the samples will minimize premature breakthrough or channeling and ensure maximum recoveries in each recovered fraction.



To increase laboratory efficiency and reduce the amount of solvents used for extract cleanup, the US EPA has allowed the use of pre-packed SPE tubes containing Florisil® or silica packing.

Refer to page 19 for SPE ordering information. For additional questions on the use of Florisil® SPE, refer to the appropriate EPA Method, request Applications Note #59562 from Restek, or call Technical Service at 800-356-1688 or 814-353-1300, ext. 4.

References

1. *J. of AOAC*, Ch. 24, 208, Vol. 49, Nov.1 (1966), p. 223
2. "Test Methods for Evaluating Solid Waste Physical/Chemical Methods (US EPA SW 846) Final Update III," December 1996. Available from the US government, Mail Stop: SSOP, Washington, DC, 20402-9328.
3. US EPA Contract Laboratory Program, *Statement of Work for Organic Analysis OLM04.0*, Exhibit D Pesticides/Aroclors.

References not available from Restek.

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Contamination from Double-Bond, Triple-Bond, or Aromatic Compounds: Sulfuric Acid Cleanup

Sulfuric acid will add to nearly any double-bond, triple-bond, or aromatic compound, producing a compound that has the general structure shown in Figure 1. The only compounds that do not undergo this reaction are those with single bonds, or compounds that are stabilized by groups, which make

the multiple bond inaccessible to sulfuric acid addition. This reaction can be used to convert nearly every compound found in pesticide and PCB extracts, from organic-soluble compounds to aqueous-soluble compounds. The resulting organic phase then can be removed and concentrated, resulting in a much less contaminated extract. It is important to note that many of the pesticides will undergo this reaction, so this cleanup can only be used for PCB analysis.

To perform sulfuric acid cleanup, place the hexane extract in a vial about 3- to 4-times the volume of the extract. Add an equal amount of 1:1 sulfuric acid, cap the vial, and shake for a few minutes. Most of the color will be transferred to the aqueous (bottom) layer as the reaction progresses. Allow the layers to separate either by standing or

centrifugation. Using a glass pipette, quantitatively remove the hexane (top) layer and transfer to a KD or a concentrator vial. If the hexane extract still has significant color, repeat the steps until no more color is exchanged into the aqueous layer. Once the extract has been transferred to the KD or concentrator vial, reduce it to final volume.

Following sulfuric acid cleanup, primarily only hydrocarbons, sulfur, some chlorinated pesticides, and PCBs will be remaining.

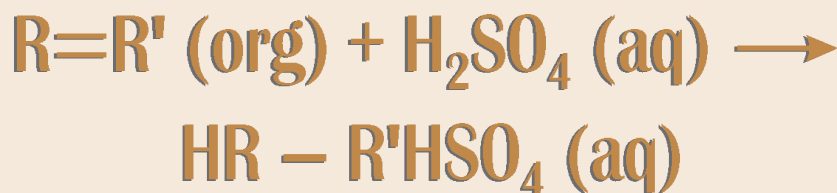
General Contaminants: Carbon Cleanup

For many years, activated charcoal has been used to separate target compounds from sample matrix interferences. Past problems have included lot-to-lot variability of the material itself, as well as manual column packing inconsistencies, which often resulted in variable elution patterns. Labs were required to test each new lot of charcoal, and then correct the required elution solvent volumes. Over the last decade, development of a new, chromatographic-grade graphitized carbon has provided a more consistent product with predictable elution behavior. This carbon material has far fewer contaminants than charcoal and also is available in commercially preppacked cartridges, which further increases performance reproducibility.

Graphitized carbon tubes have a unique elution pattern characteristic compared to Florisil®, alumina, and silica gel tubes; and have higher sample capacity in comparison to C18 tubes. In general, carbon elutes polar compounds first, then nonpolar compounds. For this reason, carbon makes a very good absorbent to remove nonpolar matrix interferences from sample extracts.

Graphitized carbon is a versatile, nonporous adsorbent, which retains or extracts a variety of compounds. The extraction system may be adjusted to retain and elute aliphatic, aromatic, polar, and nonpolar analytes. For optimal recoveries, compounds of interest should be applied in weak solvents, or

Figure 1: Sulfuric acid will add to nearly any double-bond, triple-bond, or aromatic compound, producing a compound that has this general structure:



Over the last decade, development of a new, chromatographic-grade graphitized carbon has provided a more consistent product with predictable elution behavior.

solutions with low solubility for the analytes, and eluted in strong solvents. Mixed solvent systems, including dichloromethane, often are the most effective for elution. Carbon cartridges will retain pesticides and PCBs when introduced as low volume (1mL) hexane extracts. They will retain the non-polar sample interferences and release the chlorinated pesticides using up to 20mL of a 20% dichloromethane/hexane solution*. However, caution should be taken when using graphitized carbon to clean extracts for PCB analysis because the coplanar PCB congeners BZ#77, 81, 126, and 169 are retained and do not elute using the above solvent. These congeners can be eluted using a 1:1 mixture of ethyl acetate and benzene.

**Due to the uniqueness and high capacity of graphitized carbon, all fractionation and elution volumes should be verified from lot to lot.*

Sulfur Contamination: Mercury, Activated Copper Powder Cleanup

Sulfur also is a common contaminant in pesticide and PCB extracts, and it produces a large signal on an ECD. Sulfur can be removed using GPC or one of the many cleanup procedures listed in US EPA Method 3660.

Mercury added directly to the extract vial is probably the best method for removing sulfur. (Note: Due to the hazardous nature of mercury, use caution while working with this substance.) Add a few drops of mercury to the hexane extract. The sulfur is then converted from an organic soluble species to mercury sulfide on the surface of the mercury drop, which appears as a black powder. The hexane is then pipetted off and re-vialed. Repeat this procedure until this reaction no longer occurs.

Activated copper powder also removes sulfur, but can react with some of the chlorinated pesticides if the exposure time is too long. The first compound to show signs of this reaction is usually heptachlor. The best way to use this cleanup method is to apply the activated copper powder to the top of a Florisil® SPE tube, so that the cleanup is performed as the sample passes through the cartridge, resulting in minimum exposure time. Sulfur cleanup is not amenable to the organophosphorous pesticides as several of them break down in the presence of activated copper or mercury.

Refer to page 19 for SPE tube ordering information.

Analysis of Chlorinated Pesticides

Calibration

The instrument used for the analysis of pesticides and PCBs must be calibrated prior to performing quantitative analysis. The calibration should be linear over a 16- to 100-fold concentration range. The calibration of 3 to 5 points includes analyzing a low-point standard to meet the required reporting limit, as well as a high-point standard to minimize the need for dilutions. The linearity check should contain all the pesticides being reported. The necessity to verify linearity for all target compounds is important because the different classes of pesticides (i.e., α -BHC vs. methoxychlor) will cause differences in injection port discrimination, chromatographic peak shape, or detector linearity.

Aroclor® standards are mixtures of chlorinated biphenyls, called congeners. The linearity of the PCB congeners is consistent from the monochlorinated biphenyls to decachlorobiphenyl. For some methods, running calibration curves for three Aroclor® standards covering the entire analytical range (i.e., 1242, 1254, and 1260), followed by the analysis of a single concentration for each remaining Aroclor® standard is sufficient. See pages 19-22 for common calibration standards.

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Injection Port Maintenance

The injection port is where a majority of analytical problems occur in the analysis of pesticides. The main problem is the cleanliness and inertness of the injection port with which the sample extract comes in contact. The two compounds used to check the injection port inertness are endrin and 4,4'-DDT. The breakdown components monitored for each compound are endrin aldehyde and endrin ketone, and 4,4'-DDE and 4,4'-DDD, respectively.

The breakdown of 4,4'-DDT is generally indicative of a dirty injection port caused by the analysis of oily or "dirty" sample extracts. Replacing the liner and cutting 6-12 inches off of the guard column usually is needed to bring the system back to the original state. Sample extracts causing 4,4'-DDT breakdown usually need GPC or carbon column cleanup to separate the pesticide from the sample matrix interferences.

Endrin breakdown is usually indicative of a chemical reaction taking place in the injection port. The breakdown could be caused by impurities in the carrier gas, active metal surface, a non-deactivated liner or septa particles.

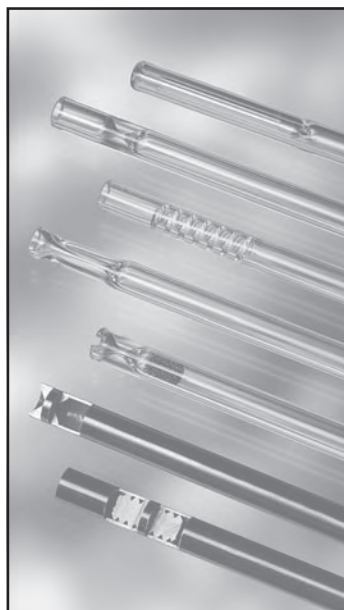
The carrier gas is usually the last troubleshooting area investigated and the hardest to eliminate. Endrin may react with a contaminate being carried into the injection port by the carrier gas. Having gas scrubbers in-line for the carrier gas will help keep this problem from occurring. In some instances, a contaminate, such as argon, in the helium carrier gas has been found when high endrin breakdown has occurred. To check for contaminated helium, use a GC/mass spectrometer (MS). For example, scan for mass 40 to check for argon contamination.

When the extract is injected into the hot injection port, the extract backflash that escapes from the top and bottom of the liner comes into contact with metal surfaces. Therefore, the metal surfaces of the injection port must be kept clean, including the inlet carrier gas line. Periodic rinsing of the carrier gas lines and swabbing out the injection port may be necessary if endrin or 4,4'-DDT breakdown increases over short periods of time or when only analyzing standards. Rinsing of the metal surfaces using solvents, or in some cases silanizing the injection port, has helped. To solvent rinse, trace the carrier gas line from the injection port back to the first connection, rinse from that point to the injection port with solvents using a syringe or HPLC pump. Do not flush solvent through any actuator valves or rubber parts, and rinse with injection port at room temperature.

Improperly deactivated injection port liners will also cause endrin breakdown. The best way to avoid this problem is to replace the liner with a newly deactivated liner when performing routine maintenance. There are two approaches to liner deactivation: perform the operation in-house or send liners out to be deactivated. Sending injection port liners to a company like Restek for cleaning and deactivating is inexpensive and keeps analysts from spending time chemically deactivating liners. There is a standard procedure for deactivating liners that includes a process of cleaning the liners in acid and deactivating with dichlorodimethylsilane. Call Restek's technical service for more information on this procedure.

Septa particles are a major cause of endrin breakdown. The septa particles will sit on top of a glass wool plug or at the bottom of the liner. Generally, filing down the burrs on the end of the syringe needle will help eliminate the coring. Another approach is to try different septa that feature reduced coring. As technologies change, new septa are investigated for their bleed characteristics and softness at different temperatures. The latest technologies on septa are available by requesting *A Guide to Minimizing Septa Problems* (lit. cat.# 59886).

The effects of chromatographic peak shape on linearity vary widely. In some cases, as with endrin aldehyde on the cyanopropyl phases (i.e., 1701 phase),



All Restek liners are deactivated for maximum inertness and minimum pesticide breakdown. Order our Inlet Supplies Catalog (lit. cat.# 55980) for a complete product listing.

the tailing of this compound is inherent with the liquid phase and does not appear to affect linearity for the limited ranges used in pesticide calibrations. However, when the tailing peaks are caused by nonvolatile contaminants deposited from sample extracts, the poor chromatography does affect linearity. The nonvolatile compounds are usually located at the front end of the column or guard column. The contaminated section of column can be removed by cutting off a piece of the inlet end of the capillary column or rinsing the column with solvent. If dirty samples are being analyzed and tailing of compounds is a problem, remove one loop of the guard column. This is usually enough to eliminate the tailing. If the analytical column is affected, the column can be rinsed with solvent to remove nonvolatile compounds. Using methylene chloride, rinse the column from back to front.

The linearity of ECDs for a 16- to 100-fold concentration range is sufficient to pass linearity requirements. Linearity for ECDs is affected by the flow rate of the make-up gas, nitrogen or argon/methane. To set the flow rate of the make-up gas, run a calibration curve including α -BHC and methoxychlor. Using response factors, calculate the percent relative standard deviation (RSD) of each compound. Set the make-up gas flow rate so the percent RSD of these two compounds is the same. An increase in make-up gas flow will improve the linearity of α -BHC but make linearity worse for methoxychlor. The remaining pesticides will exhibit linear curves once the make-up gas has been set to give good linearity for α -BHC and methoxychlor.

Because several of the pesticide compounds, most notably endrin, react with hot metal surfaces, cold on-column or direct injections are suggested. With certain GCs this becomes even more important if the sample is exposed to metal seals.

Cold On-Column Injections

In cold on-column injections, the needle is inserted directly into the column and the sample extract is deposited. On-column injections work extremely well for relatively clean samples. If contamination levels are low, and not too much nonvolatile residue is present (lipids, hydrocarbons, sulfurs, etc.) in the sample extracts, then on-column injections provide the best detectability and linearity, and narrowest peak width.

On-column injections are best suited for the analysis of water sample extracts, where analyte concentration levels are usually low and the amount of non-volatile material is relatively small. Both small and large volumes can be injected on-column, with the large-volume injections being even more sensitive to non-volatile residue. Conventional on-column injections are typically less than 1 μ L, and require the use of 0.53mm ID columns. Large-volume, on-column injections are typically 10 μ L to 100 μ L and require the use of a pre-column to eliminate the solvent. Several suppliers now offer autosamplers that permit both types of on-column injections. These systems are worth considering if you analyze relatively clean sample extracts. However, they generally only provide acceptable results for the drinking water methods (US EPA Method 500 series). If used for solid and biota extracts, the systems would require frequent maintenance.

Direct Injections

Direct injections are made by injecting the sample extract into a hot injection port liner. The extract vaporizes and the carrier gas transfers the analytes to the GC column, where they are refocused. In conventional direct injection ports using a Uniliner® glass liner, the column is connected to it by means of a press-tight seal at the bottom of the liner. This type of injection port set-up eliminates contact of analytes with the active metal surfaces below the bot-

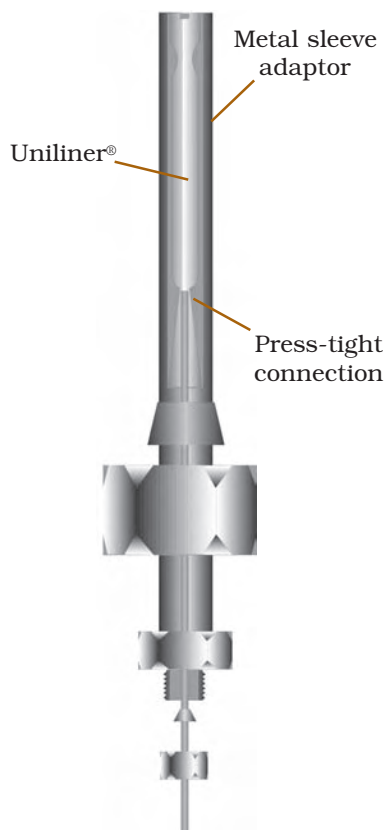
The injection port is where a majority of analytical problems occur in the analysis of pesticides. The main problem is the cleanliness and inertness of the injection port with which the sample extract comes into contact.

For tech support, call
800-356-1688, ext. 4
(814-353-1300, ext. 4)

On-column or direct injections are suggested because several of the pesticide compounds, most notably endrin, react with hot metal surfaces. With certain GCs, this becomes even more important if the sample is exposed to metal seals.

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Figure 2: A press-tight seal connects the liner to the column.



tom of the liner (Figure 2). The benefits of using this type of injection are that upwards of 3 μ L to 4 μ L of sample can be injected, and injection port discrimination is reduced.

A split/splitless injection port also may be adapted for direct injections using Uniliner® liners. In this mode of operation, the split/splitless injection port split valve (purge valve) must be turned off. Additionally, it is beneficial to make a leak-free connection (press-tight seal) between the liner and the column. Refer to the steps presented in the supplied product information sheet, or found in our catalog, to install a Press-Tight® connector. It is also helpful to use graphite ferrules for this connection because the Vespel® ferrules may cause the column connection to fracture if overtightened. Finally, before inserting the column into the connector, dip the end of the column into a vial of methanol for 30 seconds. This causes the polyimide coating on the column to swell, resulting in a better seal.

A third direct injection technique used for pesticide analysis involves large-volume injections into a cold injection port. The injected solvents and compounds are cold-trapped on the injection port liner walls. The injection port is heated to about the boiling point of the solvent, and the solvent is vented out of the system. The vent is turned off and the injection port is heated rapidly, allowing the trapped analytes to transfer from the liner to the inlet end of the analytical column.

Pesticide methods generally require a second column analysis for confirmation, to give a higher degree of confidence in reported analytes. For dual-column analyses, we recommend that these injections be made into a single injection port and split onto two columns using a glass “Y” fitting (Figure 3). Although there are alternative ways to set-up a dual-column system, this method provides the best reproducibility, while achieving the required detection limits and minimizing instrument maintenance.

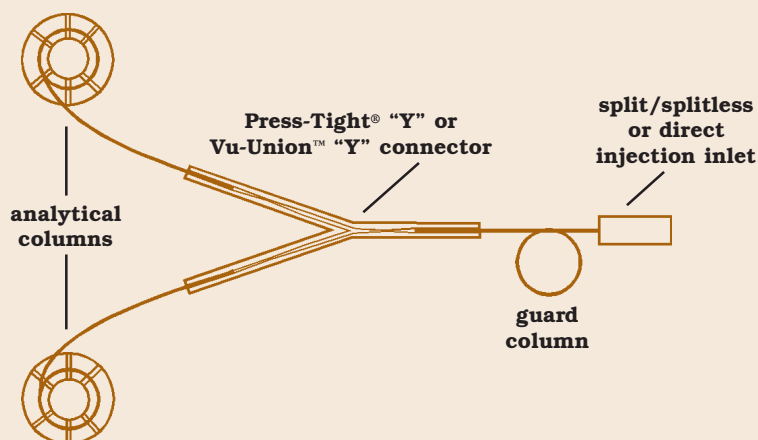
Splitless Injections

Splitless injection involves operating a split injection port with the split valve (purge valve) closed during the injection, allowing all the carrier gas to be directed into the column. The split valve remains closed for a short time (30 sec. to 2 min.) after the injection in an attempt to transfer as much of the sample extract as possible from the injection port onto the column. After this splitless hold, the purge vent is opened, and the remaining solvent and non-transferred sample are vented out of the injection port. The purge vent should have a carbon trap attached to remove any pesticides and other organic compounds before being vented into the laboratory.

Splitless injection requires optimization of the purge time to ensure that the maximum amount of analyte is transferred to the column, and minimizes the amount of solvent. Generally, the purge time is determined by maximizing the area count of the last eluting analyte. For additional information, please contact Restek's technical service and ask for the technical guide *Operating Hints for Split/Splitless Injectors* (lit. cat.#59880).

Splitless injection is prone to inertness problems because of the residence time

Figure 3: “Y” fitting provides best dual-column system connection



and exposure of the analytes, such as endrin, to the metal surfaces outside the glass liner. During injection, the vapor cloud expands outside the glass liner, exposing reactive analytes to metal surfaces. Endrin and 4,4'-DDT are used as indicator compounds for active sites. The most common active area in the injection port is the bottom of the injection port, below the liner. The vapor cloud expands past the column and comes in contact with the metal disk (inlet seal) below the liner. These inlet seals should be cleaned or replaced during routine maintenance. The use of a gold or Silcosteel®-treated inlet seal will provide greater inertness.

Resolution Discussion

For many years, environmental laboratories have struggled with various chlorinated pesticide analytical methods. Not only do the labs keep track of resolution requirements and breakdown performance criteria, but they also analyze extracts that usually contain high-boiling contaminants. While these contaminants don't always appear in the GC/ECD chromatogram, they can cause shifts in retention time, elevated baselines, and target compound breakdown. Many laboratories have used cyanopropyl capillary column stationary phases (1701 columns), which may provide the best resolution between target compounds, but have several limitations:

1. 1701-type columns are prone to on-column breakdown of DDT and methoxychlor as a result of degradation of the stationary phase. While each column can be tested for this before leaving the manufacturer, it is no guarantee that this problem will not arise after the column has been subjected to sample analyses. The problem seems to be related to the basic nature of the cyano group, and does not appear to be easily solved.
2. 1701-type columns have relatively low maximum operating temperatures, which prohibit final oven temperature ramps high enough to remove the higher-boiling oils commonly found in pesticide and PCB extracts. This procedure, commonly referred to as baking out, is used by many laboratories to eliminate or reduce the levels of heavier hydrocarbons at the end of each analysis, providing that the columns can be heated to higher levels than those used in the analysis itself.

Several phenyl/methyl phases have also been used for this analysis, including a 5% phenyl/35% phenyl/50% phenyl phase. While each of these phases has a higher maximum temperature and is less reactive, as compared to the cyanopropyl phases, they all have target compounds that coelute to some extent. The specific compounds that coelute vary based on the percent of phenyl composition, but each column has at least one coelution. This results in additional work for the laboratory, and in some cases, requires that both compounds be reported, even though only one may be present.

While using two phenyl-phase columns in a dual-column system allows baking-out of the system between analyses, the phenyl-phase columns are more prone to coelution of the chlorinated pesticides than the cyanopropyl-phase columns. This has kept the cyanopropyl-phase columns in demand for pesticide analysis, despite their limitations—until now.

The development of the Rtx®-CLPesticides and the Rtx®-CLPesticides2 columns has simplified the choice. These columns are capable of baseline resolution of the 22 common chlorinated pesticides as listed in US EPA Methods 8081, CLP, and 608. Each column is available in 0.25mm, 0.32mm, and 0.53mm IDs, and has been optimized for ECD analysis. Both feature almost zero bleed after conditioning. In addition to their separating ability, the Rtx®-CLPesticides columns can be heated to temperatures previously only tolerated by phenyl-phase columns. The maximum temperature of the Rtx®-

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**Rtx®-CLPesticides and
Rtx®-CLPesticides2 columns
baseline resolve all 22
chlorinated pesticides (US EPA
Methods 8081, CLP, and 608)
in under 25 minutes. See page
18 for ordering information.**

**Figures 6–9 are shown on
pages 16 and 17. Product
listings begin on page 18.**

CLPesticides column and the Rtx®-CLPesticides2 column is 330°C, making it similar to the 5% phenyl Rtx®-5 column.

When using cyanopropyl-phase or phenyl-phase columns, laboratories typically must calibrate using 5-point curves, injecting mix A and mix B compounds separately because the target compounds coelute. Because no coelution problems occur with the Rtx®-CLPesticides columns, the mixes can be combined. This eliminates the need for at least 5 injections during calibration of the instrument, and may free a minimum of 2.5 hours a day to analyze more samples. (The CLP method, however, mandates the separate calibration sequence—it is the only method to do so.) Restek provides the calibration standards as a single mix for laboratories wishing to use only one calibration mix in their calibration curves (see the product listings beginning on page 18 for details).

Although Rtx®-CLPesticides columns are available in all three common ID dimensions, we typically recommend using the 0.32mm ID size. This size provides the best combination of capacity and peak width (Figure 4). If your sample extracts are particularly contaminated, you may find that the 0.53mm ID columns allow for longer duration of calibration, because of the large capacity (Figure 5). Columns of 0.25mm ID provide better resolution, but cannot handle contaminated or large samples (Figure 6). In most cases, the 0.32mm ID is the size of choice for this analysis.

When configuring the column pair, use a 5m section of guard tubing to connect the glass “Y” to the injection port. This allows enough of a retention gap so that the sample is evenly split into the two columns. The best flow rates and oven programs are listed on the chromatograms, but it is possible to get a total run time as low as 16 minutes using hydrogen as a carrier gas (Figure 7). Some laboratories may not be comfortable using hydrogen, though it affords a shorter run time. In any event, both helium and hydrogen work well as a carrier gas when using Rtx®-CLPesticides columns for this analysis.

The separations of US EPA Method 508 pesticides (Figure 8) and US EPA Method 8151 herbicides (Figure 9) also are shown because these analyses typically are run on the same instrument as the chlorinated pesticides already shown. It is important to note that Rtx®-CLPesticides columns also exhibit baseline separation for these compounds, except for a few that are not commonly observed. The Rtx®-CLPesticides column and the Rtx®-CLPesticides2 column combination results in the resolution of all compounds, allowing the use of one column pair and the same instrument flow rate for many different analyses.

Summary

Although the analysis of chlorinated pesticides historically has been one of the more difficult tests performed by environmental testing laboratories, using Restek’s Rtx®-CLPesticides columns, coupled with the methods presented in this guide will make your analyses easier. Optimized sample preparation and extract cleanup, the proper injection technique, suitable analytical columns and standards, and accurate quantitation will improve your results and increase your lab’s throughput.

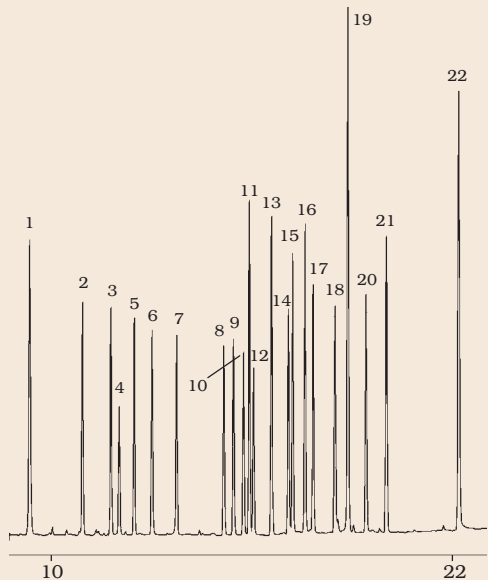
When problems occur, using proper troubleshooting and maintenance techniques can quickly reestablish system integrity. When faced with difficulties in your pesticide or PCB analysis, remember that the majority of problems occur during the sample preparation and cleanup step, or at the injection port of the GC. If you are still having difficulties with your analysis after following the steps in this guide, please contact Restek’s technical service at 800-356-1688, ext. 4, and we will be happy to help you.

Figure 4

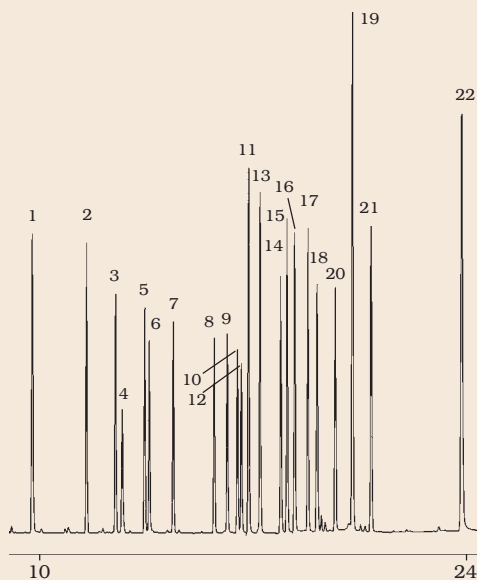
0.32mm ID columns provide best capacity and resolution of all 22 pesticides used in US EPA Method 8081.

Rtx®-CLPesticides

30m, 0.32mm ID, 0.50µm (cat.# 11139)

**Rtx®-CLPesticides2**

30m, 0.32mm ID, 0.25µm (cat.# 11324)



1. 2,4,5,6-tetrachloro-m-xylene
2. α -BHC (α -HCH)
3. γ -BHC (lindane)
4. β -BHC (β -HCH)
5. δ -BHC (δ -HCH)
6. heptachlor
7. aldrin
8. heptachlor epoxide
9. γ -chlordane
10. α -chlordane
11. 4,4'-DDE
12. endosulfan I
13. dieldrin
14. endrin
15. 4,4'-DDD
16. endosulfan II
17. 4,4'-DDT
18. endrin aldehyde
19. methoxychlor
20. endosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl

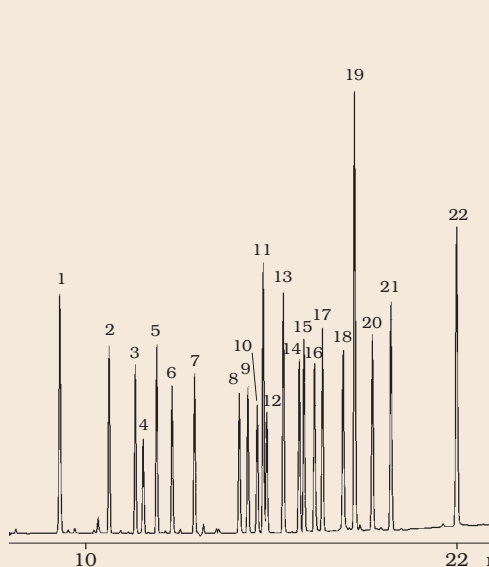
On-column concentration: 16-160pg organochloride pesticide mix AB#2 (cat.# 32292); **Oven temp.:** 120°C (hold 1 min.) to 300°C (hold 10 min.) @ 9°C/min.; **Inj. port:** Direct, Uniliner® sleeve (cat.# 20335), at 200°C; **Detector:** ECD, 300°C with Anode Purge; **Dead time:** 1.9 min.; **Head pressure:** 8.7psi (constant); **Flow rate:** 1.3mL/min. @ 120°C, Helium.

Figure 5

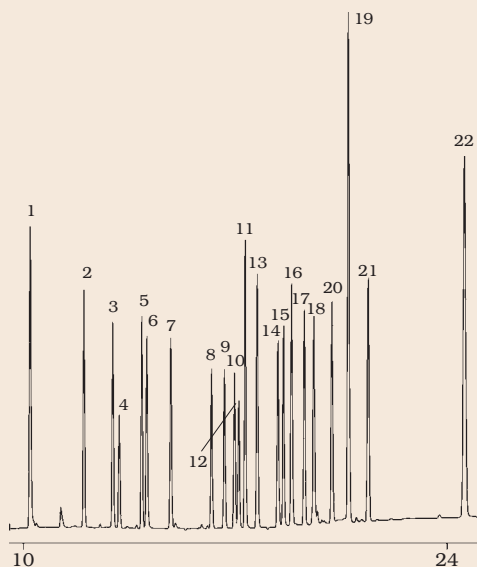
0.53mm ID columns for the best analysis of contaminated pesticide samples (US EPA Method 8081).

Rtx®-CLPesticides

30m, 0.53mm ID, 0.50µm (cat.# 11140)

**Rtx®-CLPesticides2**

30m, 0.53mm ID, 0.42µm (cat.# 11340)



1. 2,4,5,6-tetrachloro-m-xylene
2. α -BHC (α -HCH)
3. γ -BHC (lindane)
4. β -BHC (β -HCH)
5. δ -BHC (δ -HCH)
6. heptachlor
7. aldrin
8. heptachlor epoxide
9. γ -chlordane
10. α -chlordane
11. 4,4'-DDE
12. endosulfan I
13. dieldrin
14. endrin
15. 4,4'-DDD
16. endosulfan II
17. 4,4'-DDT
18. endrin aldehyde
19. methoxychlor
20. endosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl

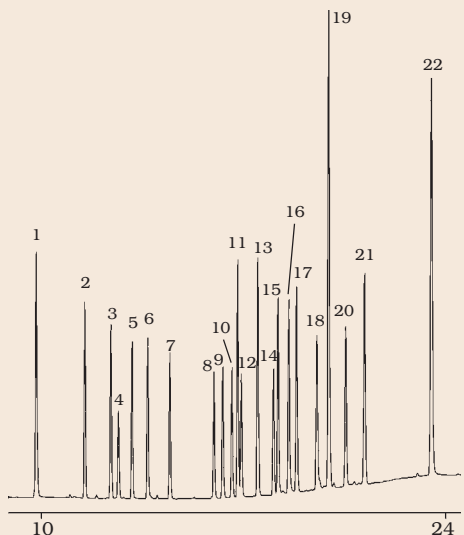
On-column concentration: 16-160pg organochloride pesticide mix AB#2 (cat.# 32292); **Oven temp.:** 120°C (hold 1 min.) to 300°C (hold 10 min.) @ 9°C/min.; **Inj. port:** Direct, Uniliner® sleeve (cat.# 20335), at 200°C; **Detector:** ECD, 300°C with Anode Purge; **Dead time:** 1.9 min.; **Head pressure:** 3psi (constant); **Flow rate:** 2.83mL/min. @ 120°C, Helium.

Figure 6

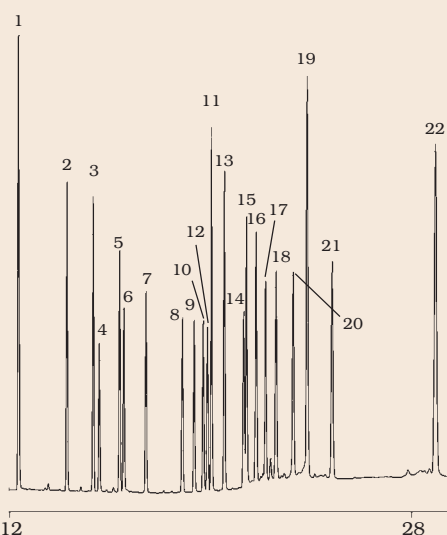
Smaller ID provides better detection limits. 0.25mm ID columns provide better signal-to-noise ratio.
(US EPA Method 8080).

Rtx®-CLPesticides

30m, 0.25mm ID, 0.25µm (cat.# 11123)

**Rtx®-CLPesticides2**

30m, 0.25mm ID, 0.20µm (cat.# 11323)



1. 2,4,5,6-tetrachloro-m-xylene
2. α -BHC (α -HCH)
3. γ -BHC (lindane)
4. β -BHC (β -HCH)
5. δ -BHC (δ -HCH)
6. heptachlor
7. aldrin
8. heptachlor epoxide
9. γ -chlordane
10. α -chlordane
11. 4,4'-DDE
12. endosulfan I
13. dieldrin
14. endrin
15. 4,4'-DDD
16. endosulfan II
17. 4,4'-DDT
18. endrin aldehyde
19. methoxychlor
20. endosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl

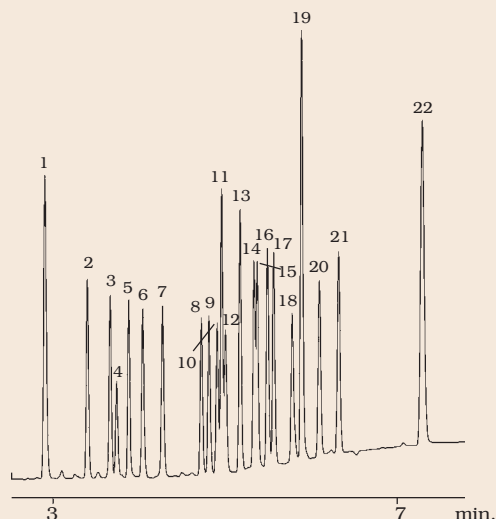
On-column concentration: 16-160pg organochloride pesticide mix AB#2 (cat.# 32292); **Oven temp.:** 120°C (hold 1 min.) to 300°C (hold 3 min.) @ 8.5°C/min.; **Inj. port:** Direct, Uniliner® sleeve (cat.# 20335), at 200°C; **Detector:** ECD, 300°C with Anode Purge; **Dead time:** 2.4 min.; **Head pressure:** 11.2psi (constant); **Flow rate:** 0.64mL/min. @ 120°C, Helium.

Figure 7

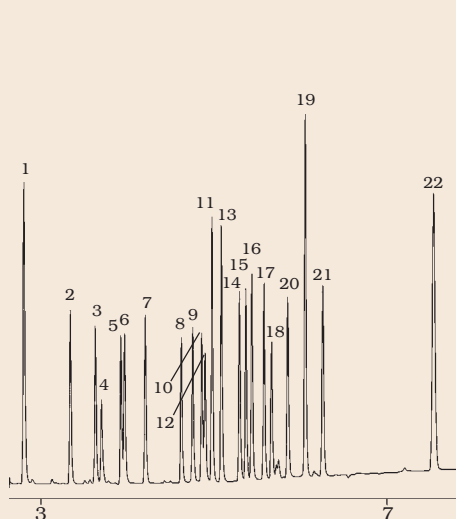
Fast screening of pesticides using hydrogen gas flow with 15m columns.

Rtx®-CLPesticides

15m, 0.32mm ID, 0.50µm (cat.# 11136)

**Rtx®-CLPesticides2**

15m, 0.32mm ID, 0.25µm (cat.# 11321)



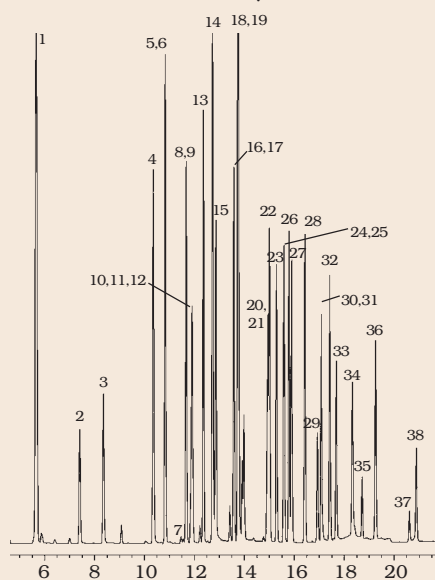
1. 2,4,5,6-tetrachloro-m-xylene
2. α -BHC (α -HCH)
3. γ -BHC (lindane)
4. β -BHC (β -HCH)
5. δ -BHC (δ -HCH)
6. heptachlor
7. aldrin
8. heptachlor epoxide
9. γ -chlordane
10. α -chlordane
11. 4,4'-DDE
12. endosulfan I
13. dieldrin
14. endrin
15. 4,4'-DDD
16. endosulfan II
17. 4,4'-DDT
18. endrin aldehyde
19. methoxychlor
20. endosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl

On-column concentration: 16-160pg organochloride pesticide mix AB#2 (cat.# 32292); **Oven temp.:** 115°C to 280°C (hold 2 min.) @ 29°C/min.; **Inj. port:** Direct, Uniliner® sleeve (cat.# 20335), at 200°C; **Detector:** ECD, 300°C with Anode Purge; **Dead time:** 0.73 min.; **Head pressure:** 4.5psi (constant); **Flow rate:** 1.33mL/min. @ 120°C, Hydrogen.

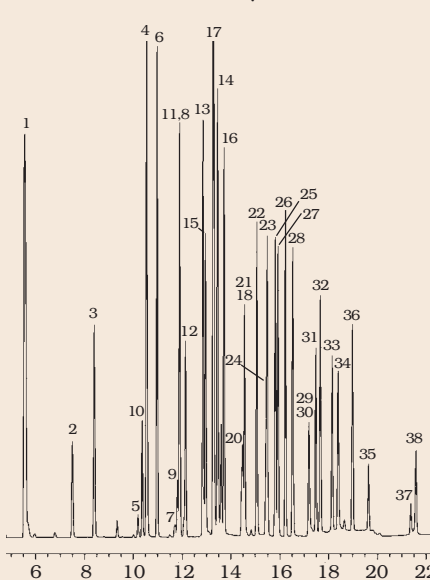
Figure 8

0.32mm ID Rtx®-CLPesticides and Rtx®-CLPesticides2 columns provide good separation of Method 508.1 pesticides.

Rtx®-CLPesticides
30m, 0.32mm ID, 0.50µm (cat.# 11139)



Rtx®-CLPesticides2
30m, 0.32mm ID, 0.25µm (cat.# 11324)



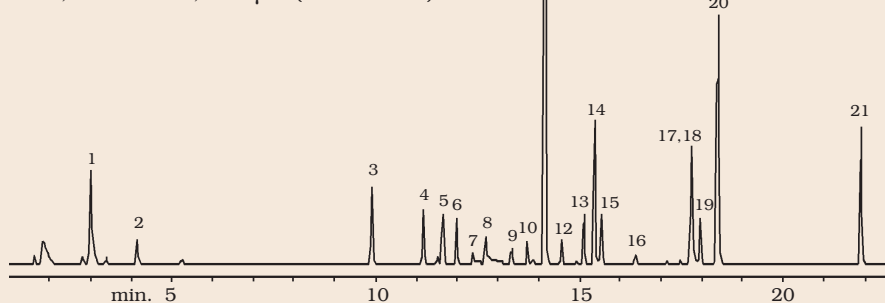
On-column concentration: see peak ID list; **Oven temp.:** 120°C (hold 1 min.) to 300°C (hold 10 min.) @ 9°C/min.; **Inj. port:** Direct, Uniliner® sleeve (cat.# 20335), at 200°C; **Detector:** ECD, 300°C with Anode Purge; **Dead time:** 1.7 min.; **Head pressure:** 12psi (constant); **Flow rate:** 1.4mL/min. @ 120°C, Helium.

1.	hexachloropentadiene	1000 pg/µL
2.	etridiazole	500
3.	chlorneb	500
4.	hexachlorobenzene	1000
5.	propachlor	100
6.	α-BHC (α-HCH)	500
7.	simazine	100
8.	γ-BHC (γ-HCH)	5000
9.	atrazine	1000
10.	trifluralin	100
11.	pentachloronitrobenzene	100
12.	β-BHC (β-HCH)	100
13.	δ-BHC (δ-HCH)	100
14.	metribuzin	100
15.	heptachlor	1000
16.	aldrin	1000
17.	chlorothalonil	1000
18.	4,4'-dibromobiphenyl	1000
19.	alachlor	1000
20.	metalachlor	100
21.	DCPA (decthal)	100
22.	heptachlor epoxide	5000
23.	γ-chlordane	100
24.	cyanozine	100
25.	α-chlordane	100
26.	4,4'-DDE	100
27.	endosulfan I	100
28.	dieldrin	500
29.	endrin	100
30.	chlorobenzilate	100
31.	4,4'-DDD	100
32.	endosulfan II	100
33.	4,4'-DDT	100
34.	endrin aldehyde	100
35.	methoxychlor	100
36.	endosulfan sulfate	100
37.	cis-permethrin	1000
38.	trans-permethrin	1000

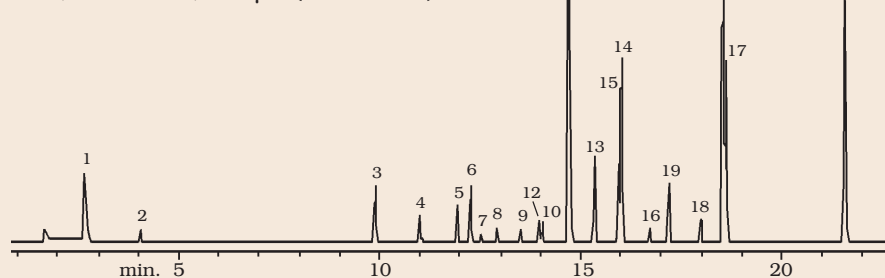
Figure 9

Primary herbicides are well-resolved (US EPA Method 8151).

Rtx®-CLPesticides
30m, 0.32mm ID, 0.50µm (cat.# 11139)



Rtx®-CLPesticides2
30m, 0.32mm ID, 0.25µm (cat.# 11324)



1.	dalapon methyl ester	1000 pg/µL
2.	1,4-dichlorobenzene	500
3.	3,5-dichlorobenzoic acid methyl ester	500
4.	4-nitroanisole	500
5.	DCAA methyl ester	500
6.	dicamba methyl ester	50
7.	MCPA methyl ester	50000
8.	MCPA methyl ester	50000
9.	dichloroprop methyl ester	200
10.	2,4-D methyl ester	200
11.	pentachloroanisole	200
12.	4,4'-dibromooctafluorobiphenyl	50
13.	2,4,5-TP (silvex) methyl ester	50
14.	chloramben methyl ester	500
15.	2,4,5-T methyl ester	50
16.	2,4-DB methyl ester	200
17.	picloram methyl ester	500
18.	bentazon methyl ester	500
19.	dinoseb methyl ester	150
20.	DCPA dimethyl ester	500
21.	acifluorfen methyl ester	500

On-column concentration: see peak ID list;
Oven temp.: 90°C (hold 1 min.) to 300°C (hold 10 min.) @ 9°C/min.; **Inj. port:** Direct, Uniliner® sleeve (cat.# 20335), at 200°C; **Detector:** ECD, 300°C with Anode Purge; **Dead time:** 1.86 min.; **Head pressure:** 12psi (constant); **Flow rate:** 1.2mL/min. @ 120°C, Helium.

Product Listings:

For customer service, call
800-356-1688, ext. 3
 (814-353-1300, ext. 3)

Rtx®-CLPesticides Column

ID	df (µm)	temp. limits	stable to	15-Meter	30-Meter
0.25mm	0.25	-60 to 310/330°C	340°C	11120	11123
0.32mm	0.50	-60 to 310/330°C	340°C	11136	11139
0.53mm	0.50	-60 to 310/330°C	340°C	11137	11140

Rtx®-CLPesticides2 Column

ID	df (µm)	temp. limits	stable to	15-Meter	30-Meter
0.25mm	0.20	-60 to 310/330°C	340°C	11320	11323
0.32mm	0.25	-60 to 310/330°C	340°C	11321	11324
0.53mm	0.42	-60 to 310/330°C	340°C	11337	11340

Rtx®-CLPesticides Kits

(Note: Columns are not preconnected in the following kits.)

0.53mm ID Rtx®-CLPesticides Kit

Includes:

30m, 0.53mm ID, 0.50µm Rtx®-CLPesticides column
 30m, 0.53mm ID, 0.42µm Rtx®-CLPesticides2 column
 Universal Angled "Y" Press-Tight® Connector
 5m, 0.53mm ID IP Deactivated Guard Column

cat.# 11197/kit

0.32mm ID Rtx®-CLPesticides Kit

Includes:

30m, 0.32mm ID, 0.50µm Rtx®-CLPesticides
 30m, 0.32mm ID, 0.25µm Rtx®-CLPesticides2
 Universal Angled "Y" Press-Tight® Connector
 5m, 0.32mm ID IP Deactivated Guard Column

cat.# 11198/kit

0.25mm ID Rtx®-CLPesticides Kit

Includes:

30m, 0.25mm ID, 0.25µm Rtx®-CLPesticides
 30m, 0.25mm ID, 0.20µm Rtx®-CLPesticides2
 Universal Angled "Y" Press-Tight® Connector
 5m, 0.32mm ID IP Deactivated Guard Column

cat.# 11199/kit

Analytical Reference Materials

Save \$ by ordering these reference materials with your Rtx®-CLPesticides Kits! Just add the appropriate suffix # to the Rtx®-CLPesticides Kit catalog number.

Pesticide Mix AB#1: cat.# 32291 Suffix #-530

Pesticide Mix AB#2: cat.# 32292 Suffix #-535

Method 8080 Organochlorine Pesticides

Organochlorine Pesticide Mix AB #1

aldrin	dieldrin
α-BHC	endosulfan I
β-BHC	endosulfan II
δ-BHC	endosulfan sulfate
γ-BHC (lindane)	endrin
α-chlordane	endrin aldehyde
γ-chlordane	endrin ketone
4,4'-DDD	heptachlor
4,4'-DDE	heptachlor epoxide (B)
4,4'-DDT	methoxychlor

200µg/mL ea. in hexane/toluene (1:1),
1mL/ampul

		w/data pack
Each	32291	32291-500
5-pk.	32291-510	32291-520
10-pk.		32391

Organochlorine Pesticide Mix AB #2

	µg/mL		µg/mL
aldrin	8	dieldrin	16
α-BHC	8	endosulfan I	8
β-BHC	8	endosulfan II	16
δ-BHC	8	endosulfan sulfate	16
γ-BHC (lindane)	8	endrin	16
α-chlordane	8	endrin aldehyde	16
γ-chlordane	8	endrin ketone	16
4,4'-DDD	16	heptachlor	8
4,4'-DDE	16	heptachlor epoxide (B)	8
4,4'-DDT	16	methoxychlor	80

In hexane/toluene (1:1), 1mL/ampul

		w/data pack
Each	32292	32292-500
5-pk.	32292-510	32292-520
10-pk.		32392

For tech support, call
800-356-1688, ext. 4
(814-353-1300, ext. 4)

Sample Preparation

Resprep™-C18 and -C8 SPE Disks

Meets requirements for EPA Methods 525.1, 506, 550.1, and 549.1.

Resprep™-C18-47: cat.# 24004, 20-pack

Resprep™-C8-47: cat.# 24048, 24-pack

Resprep™ Resin SPE Disk

Meets requirements for EPA Methods 515.2 and 553.

Resprep™ Resin SPE Disk: cat.# 26023, 20-pack

Resprep™ SPE Cartridges

(All cartridges are polypropylene and have polyethylene frits unless otherwise noted):

C18	6mL	500mg	30-pk.	cat.# 24052
	6mL	1000mg	30-pk.	cat.# 24051
Florisil®	3mL	500mg	50-pk.	cat.# 24031
	3mL	500mg	50-pk.	cat.# 24032*
	6mL	1000mg	30-pk.	cat.# 24034
	6mL	500mg	30-pk.	cat.# 26086**
	6mL	1000mg	30-pk.	cat.# 26085**
Silica	3mL	500mg	50-pk.	cat.# 24035
	3mL	500mg	50-pk.	cat.# 24036*
	6mL	1000mg	30-pk.	cat.# 24038
Carbon	3mL	250mg	50-pk.	cat.# 26088
	6mL	500mg	30-pk.	cat.# 26087

*Stainless steel frits

**Glass cartridges with Teflon® frits.

www.restekcorp.com

For customer service, call
800-356-1688, ext. 3
 (814-353-1300, ext. 3)

Method 508.1

508.1 Internal Standard Mix

pentachloronitrobenzene at 100µg/mL in ethyl acetate, 1mL/ampul

w/data pack		
Each	32091	32091-500
5-pk.	32091-510	32091-520
10-pk.	32191	

508.1 Surrogate Mix

4,4'-dibromobiphenyl at 500µg/mL in ethyl acetate, 1mL/ampul

w/data pack		
Each	32092	32092-500
5-pk.	32092-510	32092-520
10-pk.	32192	

508.1 GC Degradation Check Mix

4,4'-DDT endrin
 100µg/mL ea. in ethyl acetate,
 1mL/ampul

w/data pack		
Each	32093	32093-500
5-pk.	32093-510	32093-520
10-pk.	32193	

508 Performance Check Mix

δ-BHC (δ-HCH) 0.4µg/mL
 chlorothalonil 0.5
 chlorpyrifos 0.02
 DCPA (dacthal) 0.5

At concentrations listed in methyl-tert-butyl-ether, 1mL/ampul

w/data pack		
Each	32045	32045-500
5-pk.	32045-510	32045-520
10-pk.	32145	

508.1 Pesticide Kit

32045: 508 Performance Check Mix
 32091: 508.1 Internal Standard Mix
 32092: 508.1 Surrogate Mix
 32093: 508.1 GC Degradation Check Mix
 32094: 508.1 Calibration Mix #1
 32095: 508.1 Calibration Mix #2
 32096: 508.1 Calibration Mix #3

Contains 1mL each of these products.

Kit		Kit
		w/data pack
32097		32097-500

508.1 Calibration Mix #1

aldrin	endosulfan I
α-BHC	endosulfan II
β-BHC	endosulfan sulfate
δ-BHC	endrin
γ-BHC (lindane)	endrin aldehyde
4,4'-DDD	heptachlor
4,4'-DDE	heptachlor epoxide (B)
4,4'-DDT	methoxychlor
dieldrin	

500µg/mL ea. in ethyl acetate,
 1mL/ampul

w/data pack		
Each	32094	32094-500
5-pk.	32094-510	32094-520
10-pk.	32194	

508.1 Calibration Mix #2

chlorobenzilate	hexachlorobenzene
α-chlordane	cis-permethrin*
γ-chlordane	trans-permethrin*
chlorneb	propachlor
DCPA	trifluralin
etridiazole	

* 1000µg/mL total permethrin. Exact composition of each isomer is listed on certificate of analysis.

500µg/mL ea. in ethyl acetate,
 1mL/ampul

w/data pack		
Each	32095	32095-500
5-pk.	32095-510	32095-520
10-pk.	32195	

508.1 Calibration Mix #3

alachlor	hexachlorocyclopentadiene
atrazine	metolachlor
chlorthalonil	metribuzin
cyanazine	simazine

500µg/mL ea. in ethyl acetate,
 1mL/ampul

w/data pack		
Each	32096	32096-500
5-pk.	32096-510	32096-520
10-pk.	32196	

Method 608 Organochlorine Pesticides & PCBs

Method 608 Calibration Mix

aldrin	dieldrin
α -BHC	endosulfan I
β -BHC	endosulfan II
δ -BHC	endosulfan sulfate
γ -BHC (lindane)	endrin
4,4'-DDD	endrin aldehyde
4,4'-DDE	heptachlor
4,4'-DDT	heptachlor epoxide (B)

200 μ g/mL ea. in hexane: toluene (1:1)

1mL/ampul

		w/data pack
Each	32022	32022-500
5-pk.	32022-510	32022-520
10-pk.		32122

Method 608 Complete Kit

32022: Method 608 Calibration Mix
 32006: Aroclor® 1016
 32007: Aroclor® 1221
 32008: Aroclor® 1232
 32009: Aroclor® 1242
 32010: Aroclor® 1248
 32011: Aroclor® 1254
 32012: Aroclor® 1260
 32005: Toxaphene
 32021: Chlordane (technical)

Contains 1mL ea. of these products.

	Kit	w/data pack
	32060	32160

CLP GPC Calibration Mix

CLP GPC Calibration Mix

bis(2-ethylhexyl)phthalate	10mg/mL
corn oil	250mg/mL
methoxychlor	2.0mg/mL
perylene	0.2mg/mL
sulfur	0.8mg/mL

1mL/ampul:

	Each	10-Pack w/data pack
	32019	32119

5mL/ampul:

	Each	10-Pack w/data pack
	32023	32123

Revised GPC Calibration Mix

bis(2-ethylhexyl)phthalate	5mg/mL
corn oil	250mg/mL
methoxychlor	1.0mg/mL
perylene	0.2mg/mL
sulfur	0.8mg/mL

1mL/ampul:

	Each	10-Pack w/data pack
	32041	32141

5mL/ampul:

	Each	10-Pack w/data pack
	32042	32142

Pesticide Surrogate Solutions

Dibutylchlorendate Mix

200 μ g/mL in acetone

1mL/ampul:

		w/data pack
Each	32025	32025-500
5-pk.	32025-510	32025-520
10-pk.		32125

5mL/ampul:

		w/data pack
Each	32026	32026-500
5-pk.	32026-510	32026-520
10-pk.		32126

2,4,5,6-Tetrachloro-m-xylene Mix

200 μ g/mL in acetone

1mL/ampul:

		w/data pack
Each	32027	32027-500
5-pk.	32027-510	32027-520
10-pk.		32127

5mL/ampul:

		w/data pack
Each	32028	32028-500
5-pk.	32028-510	32028-520
10-pk.		32128

Decachlorobiphenyl Mix

200 μ g/mL in acetone

1mL/ampul:

		w/data pack
Each	32029	32029-500
5-pk.	32029-510	32029-520
10-pk.		32129

5mL/ampul:

		w/data pack
Each	32030	32030-500
5-pk.	32030-510	32030-520
10-pk.		32130

Pesticide Surrogate Mix

decachlorobiphenyl

2,4,5,6-tetrachloro-m-xylene

200 μ g/mL ea. in acetone, 1mL/ampul

		w/data pack
Each	32000	32000-500
5-pk.	32000-510	32000-520
10-pk.		32100

For tech support, call
800-356-1688, ext. 4
 (814-353-1300, ext. 4)

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Aroclor[®], Toxaphene, and Chlordane Solutions

1000µg/mL in hexane 1mL/ampul	Each	Each w/ data pack	5pk.	5pk. w/ data pack	10pk. w/ data pack
Aroclor [®] 1016	32006	32006-500	32006-510	32006-520	32106
Aroclor [®] 1221	32007	32007-500	32007-510	32007-520	32107
Aroclor [®] 1232	32008	32008-500	32008-510	32008-520	32108
Aroclor [®] 1242	32009	32009-500	32009-510	32009-520	32109
Aroclor [®] 1248	32010	32010-500	32010-510	32010-520	32110
Aroclor [®] 1254	32011	32011-500	32011-510	32011-520	32111
Aroclor [®] 1260	32012	32012-500	32012-510	32012-520	32112
Aroclor [®] 1016/1260	32039	32039-500	32039-510	32039-520	32139

200µg/mL in isooctane 1mL/ampul	Each	Each w/ data pack	5pk.	5pk. w/ data pack	10pk. w/ data pack
Aroclor [®] 1016	32064	32064-500	32064-510	32064-520	32164
Aroclor [®] 1221	32065	32065-500	32065-510	32065-520	32165
Aroclor [®] 1232	32066	32066-500	32066-510	32066-520	32166
Aroclor [®] 1242	32067	32067-500	32067-510	32067-520	32167
Aroclor [®] 1248	32068	32068-500	32068-510	32068-520	32168
Aroclor [®] 1254	32069	32069-500	32069-510	32069-520	32169
Aroclor [®] 1260	32070	32070-500	32070-510	32070-520	32170

1000µg/mL in hexane 1mL/ampul	Each	Each w/ data pack	5pk.	5pk. w/ data pack	10pk. w/ data pack
chlordane (technical)	32021	32021-500	32021-510	32021-520	32121
toxaphene	32005	32005-500	32005-510	32005-520	32105

5000µg/mL in isooctane 1mL/ampul	Each	Each w/ data pack	5pk.	5pk. w/ data pack	10pk. w/ data pack
chlordane (technical)	32072	32072-500	32072-510	32072-520	32172
toxaphene	32071	32071-500	32071-510	32071-520	32171

PCB Kit #1

1000µg/mL in hexane, 1mL/ampul
1 ea. of 32006, 32007, 32008, 32009, 32010, 32011, and 32012.

Kit	Kit w/data pack
32089	32089-500

PCB Kit #2

200µg/mL in isooctane, 1mL/ampul
1 ea. of 32064, 32065, 32066, 32067, 32068, 32069, and 32070.

Kit	Kit w/data pack
32090	32090-500

Restek Trademarks:

Rtx, Press-Tight, Thermolite, and the Restek logo.

Other Trademarks:

Aroclor (Monsanto Co.), Florisil (U.S. Silica Co.), RapidVap (Labconco), Teflon (E.I. du Pont de Nemours & Co., Inc.), TurboVap (Zymark), and Vespel (E.I. du Pont de Nemours & Co., Inc.).

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Website NEW : www.chromalytic.com.au E-mail : info@chromtech.net.au Tel: 03 9762 2034 . . . in AUSTRALIA

Universal "Y" Press-Tight® Connectors

- Split sample flow onto two columns.
- Split a single column flow into two different detectors.
- Perform confirmational analysis with a single injection.



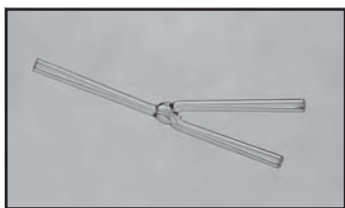
Universal "Y" Press-Tight® Connectors

cat.# 20405 (ea.)

cat.# 20406 (3-pk.)

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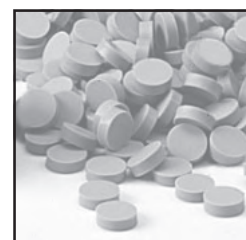
Septum Diameter	25-pk.	50-pk.	100-pk.
5mm ($\frac{3}{16}$ "	20351	20352	20353
6mm ($\frac{1}{4}$ "	20355	20356	20357
7mm	20381	20382	20383
8mm	20370	20371	—
9.5mm ($\frac{3}{8}$ "	20359	20360	20361
10mm	20378	20379	20380
11mm ($\frac{7}{16}$ "	20363	20364	20365
12.5mm ($\frac{1}{2}$ "	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Plug	20372	20373	20374

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A Guide to the Analysis of Chiral Compounds by GC.....



Inside:

*Definitions of
Chirality and Chiral
Chromatography*

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*Chiral Columns
Offer Unique
Selectivity*

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*Optimization
of Chiral
Separations*

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*Chiral Specific
Applications of
Essential Oils,
Flavors, and
Pharmaceuticals*

.....

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A team of researchers at the University of Neuchâtel developed β -cyclodextrins with superb enantiomeric selectivity. They joined forces with Restek, a manufacturer of top quality columns, to provide a unique line of commercially available β -cyclodextrin stationary phases with enhanced capabilities for chiral capillary gas chromatography.

The Rt- β DEXsm and Rt- β DEXse chiral capillary columns offer extensive enantiomeric separation of monoterpenes, monoterpene alcohols, and monoterpene ketones that cannot be matched by permethylated β -cyclodextrin columns. Rt- β DEXsp and Rt- β DEXsa are secondary columns that best resolve specific flavor and fragrance chiral components. The Rt- β DEXcst provides excellent resolution of some complex flavor compounds and has demonstrated great potential with pharmaceutical substances as well.

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Dr. Raphael Tabacchi

Born in Ticino, Switzerland, Dr. Tabacchi has been a professor for Analytical and Organic Structure at the University of Neuchâtel, Switzerland, since 1978. His research interests focused upon natural product chemistry, and development of HPLC and GC stationary phases. He has developed β -cyclodextrins with unique substitutions to create novel chiral phases for capillary GC.



Dr. Georges Claude Saturnin

Born in St. Joseph, Martinique, Dr. Saturnin became a Senior Assistant and Assistant Professor in 1990 at the University of Neuchâtel. He is involved in the development of HPLC phases. His focus is the synthesis of these new cyclodextrin materials that characterize the new chiral columns.



Claire-Lise Porret

Born in Neuchâtel, Switzerland, Ms. Porret was previously a technician at Nestlé and joined Dr. Tabacchi's team in 1991. She is also involved in the synthesis of organic compounds and with the development of GC and HPLC stationary phases.



Maurus Biedermann

Maurus has been with Dr. Konrad Grob's GC/LCGC group at the Kantonales Laboratorium Zurich (Official Food Control Authority in Switzerland) since 1990 and has participated in the development of several LCGC methods for food analysis. During his sabbatical at Restek, he demonstrated the ability of these new and unique chiral phases for many applications such as the authenticity of essential oils, "natural" flavor extracts, and the analysis of drugs for enantiomeric composition.



Sherry Sponsler

Sherry is an Applications Chemist and has been with Restek since 1990. She conducts method and product development for analysis of foods, flavors and fragrances, as well as some pharmaceutical samples. Frequent communication with customers has helped Sherry to identify many important chiral applications in these industries. She has demonstrated many of these key separations, especially for fragrances and amphetamines, using the new cyclodextrin capillary columns.



Lori Bitzer

After completing her Chemistry degree at West Virginia University in 1995, Lori joined Restek as a Fused Silica Manufacturing Chemist. She is involved with the design and production of new products including capillary chiral columns. Lori ensures product quality and consistency that are characteristic of all Restek products.

RESTEK
CORPORATION

WHAT ARE CHIRAL COMPOUNDS?

Any carbon atom that is bonded to four different functional groups is termed a chiral or an asymmetric carbon. Molecules containing one or more of these carbon centers are considered chiral molecules. Chiral centers can exist in two forms called enantiomers. These two forms are non-superimposable mirror images of each other, but both have similar properties. For example, both enantiomers will have the same boiling point, densities, and reaction rates as achiral molecules. They do, however, generally possess different aroma and flavor characteristics; more impor-

tantly, they possess differences in toxicity and biological activity.

Enantiomers are also known as optical isomers because they rotate plane polarized light in different directions. Optical isomers that rotate plane polarized light to the right, or clockwise, are termed dextrorotary (denoted as (d) or (+)). Optical isomers that rotate in the left direction are termed levorotary (denoted as (l) or (-)).

Enantiomers can be denoted by the specific configuration around the chiral center. Groups on the carbon center are assigned a "priority" based on atomic number of the first bonded atom (Cahn-Ingold-Prelog rules). The group with the highest atomic num-

ber is rated first. If priority cannot be established with the first atom, work outward until priority differences can be determined. Once priorities have been established for all four groups, specific configuration can be determined. An R configuration is designated when the priority around the asymmetric carbon is in a clockwise direction, whereas a counterclockwise direction is denoted as S. (Figure 1A)¹

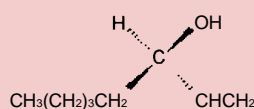
A chiral compound can possess multiple chiral centers and many combinations of configurations. Linalool oxides possess two chiral centers, resulting in four enantiomers. (Figure 1B) Note that configuration (R or S) is independent from optical activity (+ or -) or interaction with plane-polarized light.

Figure 1A

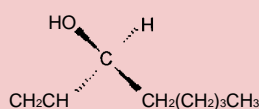
Linalool is a chiral compound because it contains an asymmetric carbon center. The mirror images are not superimposable and so they are enantiomers.



Enantiomers can be distinguished by configuration. Following groups from high to low priority in the clockwise direction is denoted R, and S for the counterclockwise direction.



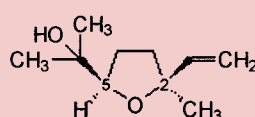
S configuration



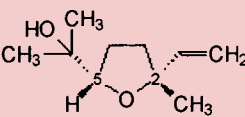
R configuration

Figure 1B

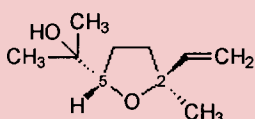
Linalool oxides have two chiral centers at carbon numbers 2 and 5 and exist as four enantiomers.



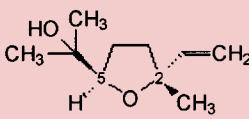
2S, 5R
(+)-cis-linalool oxide



2R, 5S
(-)-cis-linalool oxide



2S, 5S
(-)-trans-linalool oxide



2R, 5R
(+)-trans-linalool oxide

WHAT ARE CHIRAL COMPOUNDS?

Any carbon atom that is bonded to four different groups is termed a chiral or an asymmetric carbon. Molecules containing one or more of these carbon centers are considered chiral molecules. Chiral centers can exist in two forms called enantiomers. These two forms are non-superimposable mirror images of each other, but both have similar properties.

WHAT IS CHIRAL CHROMATOGRAPHY?

Chiral chromatography is the separation of enantiomeric compounds. Common liquid stationary phases used in gas chromatography resolve components from one another, but they do not possess adequate selectivity for enantiomeric separation. Addition of derivatized cyclodextrin macromolecules to common stationary phases creates capillary columns with the ability to separate enantiomers as well.

The permethylated derivative of beta-cyclodextrin in cyanopropyl-dimethylpolysiloxane liquid stationary phase is commonly used for such stereochemical separations, but it exhibits limited applications. Beta-cyclodextrins derivatized with alkyl substituents can enhance the enantiomeric resolution of various compound classes. Restek's five capillary columns incorporate various combinations of alkylated beta-cyclodextrins into a cyanopropyl-dimethylpolysiloxane liquid stationary phase to achieve significant separation.

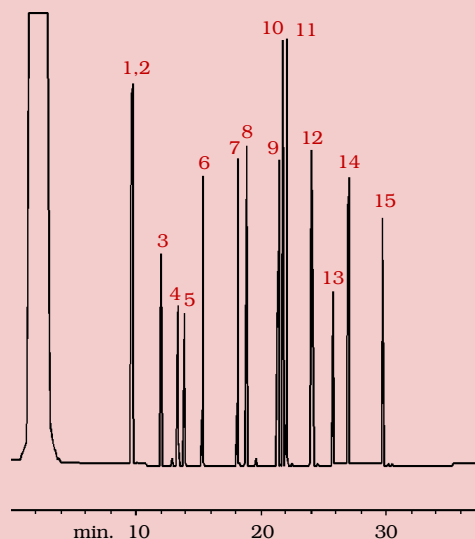
These columns also exhibit stability and extended lifetime. From the first injection to the 250th injection on a chiral column, enantiomeric separation is maintained with almost no loss in resolution (**Figures 2A and B**).

Chiral chromatography is the separation of enantiomeric compounds. Common liquid stationary phases do not possess adequate selectivity for enantiomeric separation. Addition of derivatized cyclodextrin macromolecules to common stationary phases creates capillary columns with the ability to separate many enantiomers.

Figure 2

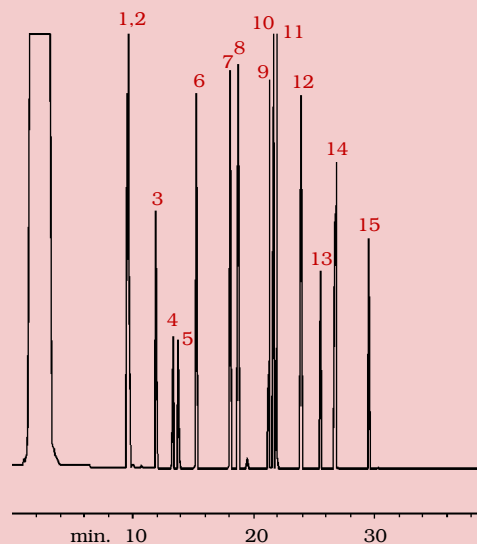
Restek's chiral columns demonstrate exceptional lifetime and stability for more than 250 injections without loss of resolution.

A: 1st injection



- | | | |
|-------------------------|-----------------------|------------------------|
| 1. (-) α -pinene | 6. undecane | 11. (-) phenylethanol |
| 2. (+) α -pinene | 7. nonanal | 12. methyl decanoate |
| 3. decane | 8. 1-octanol | 13. dicyclohexylamine |
| 4. (-) 2,3-butanediol | 9. 2,6-dimethylphenol | 14. methyl undecanoate |
| 5. (+) 2,3-butanediol | 10. (+) phenylethanol | 15. methyl dodecanoate |

B: 250th injection



30m, 0.32mm ID, 0.25 μ m Rt- β DEXsa (cat.# 13108)
Oven temp.: 40°C (hold 1 min.) to 230°C @ 2°C/min.;
Carrier gas: hydrogen; 80cm/sec. set @ 40°C; **Detector:** FID set @ 220°C

RESTEK'S CHIRAL COLUMNS OFFER UNIQUE SELECTIVITY

Figure 3

The Rt- β DEXsm column provides the best chiral separation of isoborneol and α -terpineol.

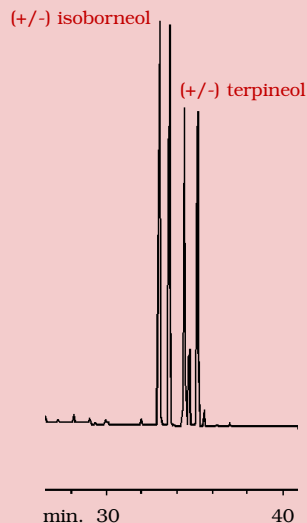
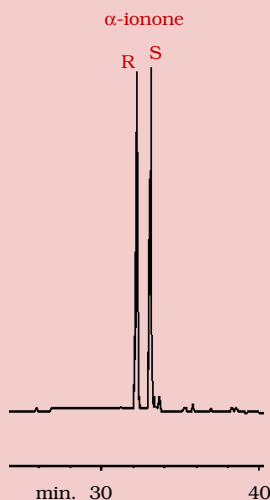


Figure 4

The Rt- β DEXsm column offers complete resolution of α -ionone.



30m, 0.32mm ID, 0.25 μ m Rt- β DEXsm (cat.# 13104)

Oven temp.: 40°C (hold 1 min.) to 230°C @ 2°C/min. (hold 3 min.);

Carrier gas: hydrogen; 80cm/sec. set @ 40°C; Detector: FID set @ 220°C

Each of the five chiral columns possesses a specific combination of alkyl substituents on the derivatized β -cyclodextrins. These unique combinations provide a wide range of utilization for each type of chiral column. Table I, on page 8, indicates that certain columns provide better resolution of specific compounds.

Rt- β DEXsm

Of the chiral columns evaluated, only the Rt- β DEXsm separates all of the 25 tested compounds, with 19 being baseline resolved. This column provides the best enantiomeric separation of α -pinene, isoborneol, α -ionone, linalool oxides, hexobarbital, and methobarbital (**Figures 3 and 4**).

Rt- β DEXse

The Rt- β DEXse is similar in performance to the Rt- β DEXsm, but it provides better resolution for limonene, linalool, linalyl acetate, ethyl-2-methylbutyrate, 2,3-butane-diol, and styrene oxides. Sometimes extensive separation results in overlap of enantiomeric pairs, as shown in **Figures 5 and 6**.

Figure 5

The Rt- β DEXse column resolves optical isomers of ethyl-2-methylbutyrate, styrene oxide, and camphor with some overlap.

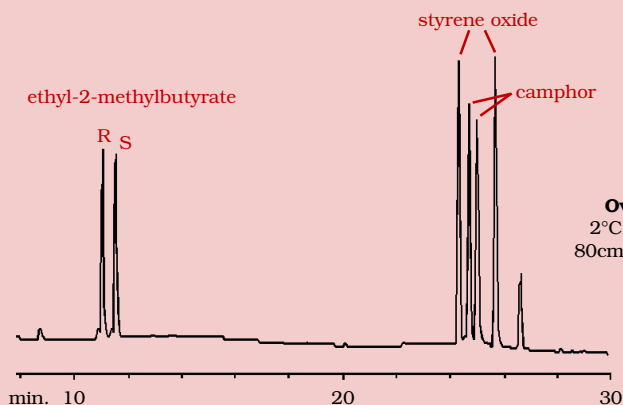
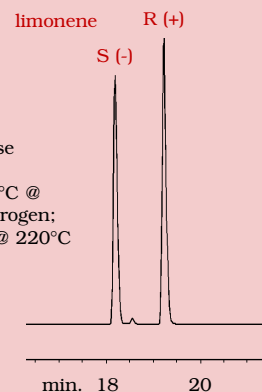


Figure 6

The Rt- β DEXse column resolves limonene enantiomers.



30m, 0.32mm ID, 0.25 μ m Rt- β DEXse (cat.# 13106)

Oven temp.: 40°C (hold 1 min.) to 230°C @ 2°C/min. (hold 3 min.); Carrier gas: hydrogen; 80cm/sec. set @ 40°C; Detector: FID set @ 220°C

Rt- β DEXsm and Rt- β DEXse

Cis and trans linalool oxides, linalool, and linalyl acetate are commonly found together in lavender oils, and resolution of all enantiomers is desirable. The Rt- β DEXsm separates the linalool oxides, but it does not resolve linalyl acetate. Conversely, Rt- β DEXse separates linalool and linalyl acetate, but does not resolve all of the linalool oxides. Combining both together in a dual column system will provide resolution for all of these enantiomers (Figure 7).

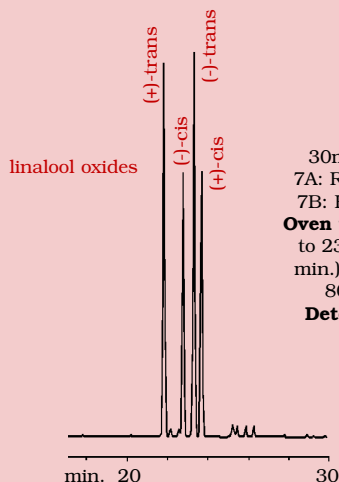
Rt- β DEXsp

Rt- β DEXsp is a specialized column that best resolves menthol. It would be useful in addition to the Rt- β DEXsm or Rt- β DEXse column for analyzing complete profiles of mint oils (Figure 8).

Rt- β DEXsa

The Rt- β DEXsa has a significantly different selectivity than the other chiral columns. It provides the best separation of 1-octen-3-ol, carvone, camphor, 1-phenylethanol, β -citronellol, and rose oxides (Figure 9).

Figure 7A
Cis and trans linalool oxide enantiomers separated on an Rt- β DEXsm column.



30m, 0.32mm ID, 0.25 μ m
7A: Rt- β DEXsm (cat.# 13104)
7B: Rt- β DEXse (cat.# 13106)
Oven temp.: 40°C (hold 1 min.)
to 230°C @ 2°C/min. (hold 3
min.); **Carrier gas:** hydrogen;
80cm/sec. set @ 40°C;
Detector: FID set @ 220°C

Figure 7B
Linalool and linalyl acetate enantiomers are resolved on the Rt- β DEXse column.

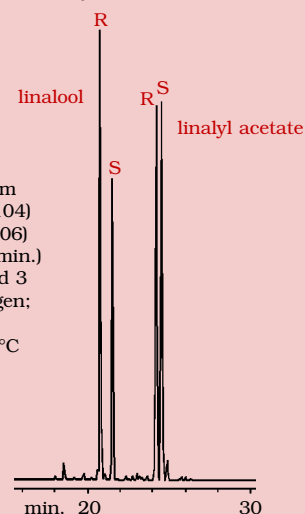


Figure 8
Menthol enantiomers are best resolved on the Rt- β DEXsp column.

30m, 0.32mm ID, 0.25 μ m Rt- β DEXsp (cat.# 13110)
Oven temp.: 60°C (hold 1 min.) to 200°C @
2°C/min.; **Carrier gas:** hydrogen; 80cm/sec. set @
40°C; **Detector:** FID set @ 220°C

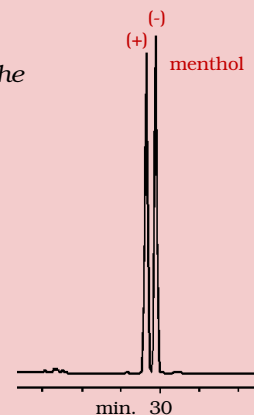


Figure 9A
1-octen-3-ol and carvone enantiomers are best resolved on an Rt- β DEXsa column.

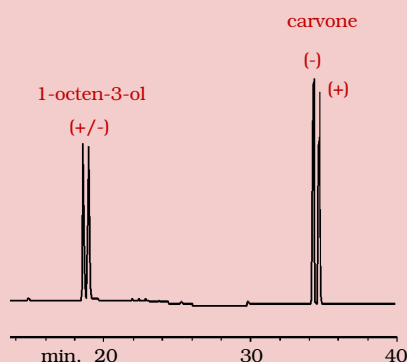
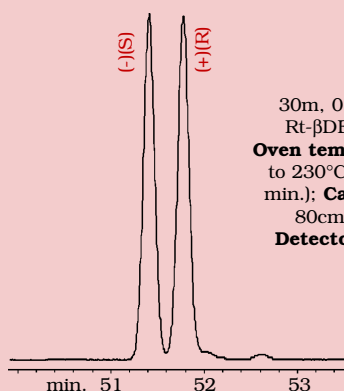


Figure 9B
An Rt- β DEXsa column provides baseline chiral resolution for β -citronellol.



30m, 0.32mm ID, 0.25 μ m
Rt- β DEXsa (cat.# 13108)
Oven temp.: 40°C (hold 1 min.)
to 230°C @ 2°C/min. (hold 3
min.); **Carrier gas:** hydrogen;
80cm/sec. set @ 40°C;
Detector: FID set @ 220°C

Figure 9C
An Rt- β DEXsa column separates racemic cis and trans rose oxides.

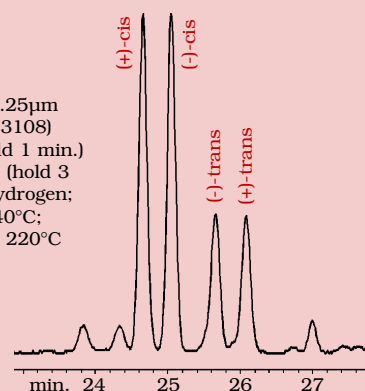


Figure 10

All of the irone isomers are resolved without overlapping of the enantiomeric pairs on the Rt-βDEXcst column.

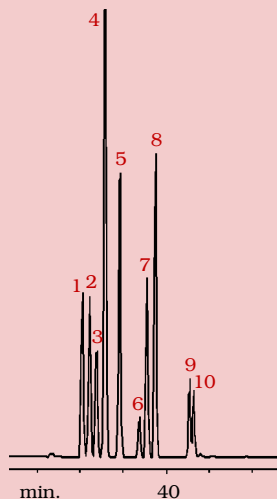
1. (-)-(2R,6R)-trans-α-irone
2. (+)-(2S,6S)-trans-α-irone
3. (+)-(2R,6R)-trans-γ-irone
4. (-)-(2S,6S)-trans-γ-irone
5. (+)-(2R,6S)-cis-α-irone
6. (+)-(2R,6S)-cis-γ-irone
7. (-)-(2S,6R)-cis-γ-irone
8. (-)-(2S,6R)-cis-α-irone
9. (+)-(2R)-β-irone
10. (-)-(2R)-β-irone

30m, 0.32mm ID, 0.25μm
Rt-βDEXcst (cat.# 13102)

Oven temp.: 40°C (hold 1 min.) to
230°C @ 2°C/min. (hold 3 min.);

Carrier gas: hydrogen; 80cm/sec. set @ 40°C;

Detector: FID set @ 220°C



Rt-βDEXcst

This column is optimum for semi-volatile chiral compounds because lower boiling components show peak broadening (discussed in the "Optimization of Chiral Separations" section). All of the irone isomers (found in iris flowers) are resolved without overlapping of the enantiomeric pairs (**Figure 10**). This is also the best column for resolution of the γ- and δ-lactones (**Figures 11A and B**).

The Rt-βDEXcst column is good for separating the enantiomers of some barbiturates and TFA-derivatives of amphetamines as well. This is discussed in further detail on pages 22 and 23.

Figure 11

The Rt-βDEXcst column provides maximum resolution of the γ-lactones and δ-lactones.

A. γ-lactones on the Rt-βDEXcst column

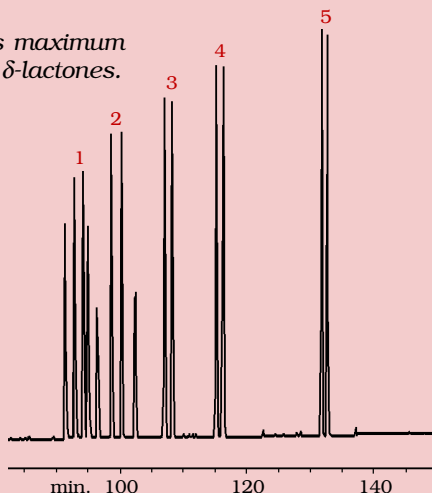
1. (+/-)γ-heptalactones
2. (+/-)γ-octalactones
3. (+/-)γ-nonolactones
4. (+/-)γ-decalactones
5. (+/-)γ-dodecalactones

30m, 0.32mm ID, 0.25μm
Rt-βDEXcst (cat.# 13102)

Oven temp.: 60°C (hold 1 min.) to 200°C
@ 1°C/min.; **Carrier gas:** hydrogen;

40cm/sec. set @ 60°C;

Detector: FID set @ 220°C



B. δ-lactones on the Rt-βDEXcst column

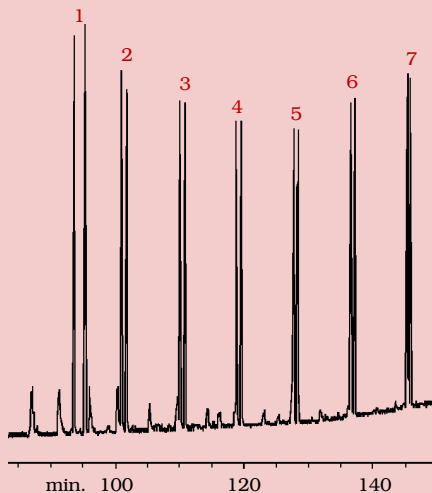
1. (+/-)δ-pentalactones
2. (+/-)δ-hexalactones
3. (+/-)δ-heptalactones
4. (+/-)δ-octalactones
5. (+/-)δ-nonolactones
6. (+/-)δ-decalactones
7. (+/-)δ-dodecalactones

30m, 0.32mm ID, 0.25μm
Rt-βDEXcst (cat.# 13102)

Oven temp.: 60°C (hold 1 min.) to 200°C
@ 1°C/min.; **Carrier gas:** hydrogen;

40cm/sec. set @ 60°C;

Detector: FID set @ 220°C



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To demonstrate the abilities of the five different types of chiral columns, we analyzed twenty-five chiral compounds commonly found in flavors, fragrances, and pharmaceutical analyses. The extent to which two enantiomers are resolved (or any two peaks) can be determined by the resolution equation and are known as resolution factors, sometimes de-

noted R. An R value of 1.5 indicates baseline resolution. Resolution factors for all chiral compounds on all the β -cyclodextrin columns were compared to those obtained on the existing Rt- β DEXm (permethylated cyclodextrin) column. **Table I** shows the degree of enantiomeric separation by resolution factor for all twenty-five components on each column. The col-

umn that has the largest resolution factor provides the best separation of a particular compound. These values can easily be compared to help determine which column is optimum for specific chiral components. **Charts 1–6** illustrate the degree of enantiomeric separation of each compound on each chiral column.

Table I
Resolution of common chiral compounds on Restek's cyclodextrin columns.

Compounds		Formula	m.w.	Column Resolution Factors						
				Rt-βDEXsm	Rt-βDEXse	Rt-βDEXsp	Rt-βDEXsa	Rt-βDEXcst	Rt-βDEXm	
Terpenes	1. α-pinene	C ₁₀ H ₁₆	136	3.50	0.80	ns	ns	0.90b	3.30	
	2. limonene	C ₁₀ H ₁₆	136	5.10	7.30	3.20	ns	2.70b	1.40	
Alcohols	3. 1-octen-3-ol	C ₈ H ₁₆ O	128	1.10	ns	0.50	2.30	0.60b	0.50t	
	4. linalool	C ₁₀ H ₁₈ O	154	3.30	6.00	4.30b	3.70	2.60t	1.00	
	5. α-terpineol	C ₁₀ H ₁₈ O	154	5.30	5.50	2.20	4.00	4.30t	1.70	
	6. terpinen-4-ol	C ₁₀ H ₁₈ O	154	2.40	2.20	ns	ns	1.20t	1.80t	
	7. isoborneol	C ₁₀ H ₁₈ O	154	4.00	3.30t	ns	ns	1.90t	2.00	
	8. β-citronellol	C ₁₀ H ₂₀ O	156	0.90	0.80	ns	1.00t	ns	ns	
	9. menthol	C ₁₀ H ₂₀ O	156	1.10	1.10	2.20	ns	1.10	1.60t	
	10. 2,3-butanediol	C ₄ H ₁₀ O ₂	90	7.50	8.10	2.20	4.60	4.00	2.60	
	11. 1-phenylethanol	C ₈ H ₁₀ O	122	7.30	6.40	1.10	7.80	6.30	6.60	
	Ketones	12. carvone	C ₁₀ H ₁₄ O	150	1.30	ns	ns	2.50	1.10	ns
		13. camphor	C ₁₀ H ₁₆ O	152	1.80	2.10t	1.30b	4.30	2.50t	ns
14. α-ionone		C ₁₃ H ₂₀ O	192	6.40	3.20	1.30	4.40	ns	3.20	
Lactones	15. γ-nonalactone	C ₉ H ₁₆ O ₂	156	4.70	5.00	3.40	4.40	5.90	1.00	
	16. γ-undecalactone	C ₁₁ H ₂₀ O ₂	184	2.90	2.90	1.70	3.70	4.50	ns	
	17. δ-decalactone	C ₁₀ H ₁₈ O ₂	170	0.90	ns	ns	2.10	2.70	ns	
Esters	18. ethyl-2-methylbutyrate	C ₇ H ₁₄ O ₂	130	1.30	3.50	1.10b	ns	ns	ns	
	19. linalylacetate	C ₁₂ H ₂₀ O ₂	196	0.60	2.20	1.20	0.10	ns	ns	
Epoxides	20. styreneoxide	C ₈ H ₈ O	120	4.80	10.20	6.50	1.00	1.40t	2.50t	
	21. trans-linalooloxides	C ₁₀ H ₁₈ O ₂	170	10.10	2.40	ns	1.50	4.30b	6.80*	
	21. cis-linalooloxides			6.20	3.60	ns	1.00	3.50b	4.20*	
Drugs	22. hexobarbital	C ₁₂ H ₁₆ O ₃ N ₂	236	11.30	4.70	0.90	ns	8.90	6.30	
	23. mephobarbital	C ₁₃ H ₁₄ O ₃ N ₂	246	8.40	3.80	0.60	ns	6.20	6.10	
	24. fenfluramine	C ₁₂ H ₁₆ F ₃ N	231	1.80	2.90t	ad	ad	ns	ad	
	25. fenfluramine TFAA der.	C ₁₄ H ₁₅ F ₆ NO	327	1.20	ns	ns	2.40	3.40	ns	

ns = no separation of enantiomers

t = peak tailing

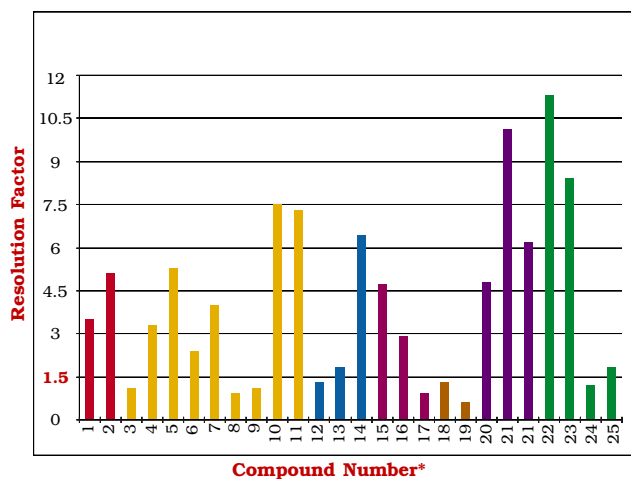
b = peak broadening

ad = adsorption of nonderivatized drug compound

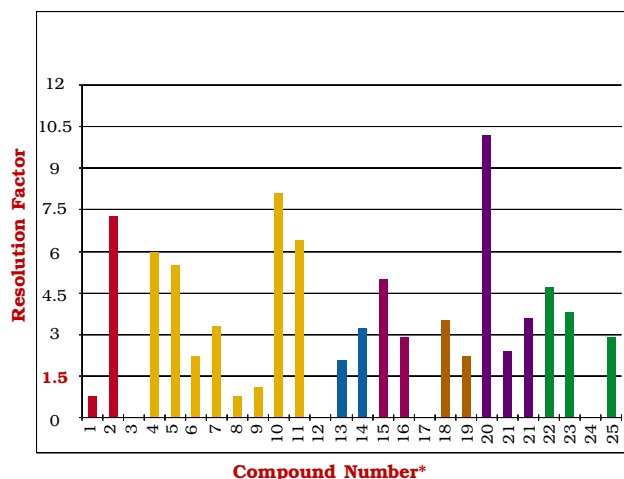
* = cis and trans linalool oxides analyzed @ 9.0 psi head pressure

Charts 1-6 illustrate the unique separation capabilities of each cyclodextrin column.

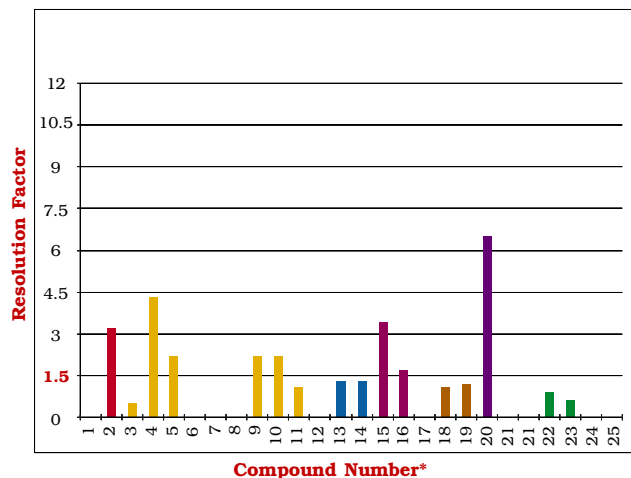
Rt- β DEXsm



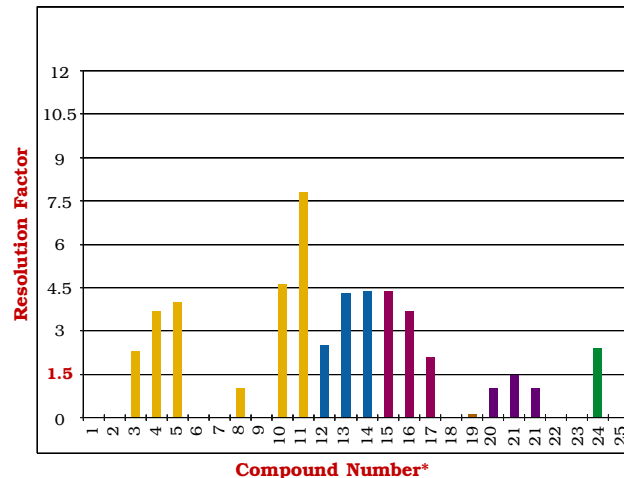
Rt- β DEXse



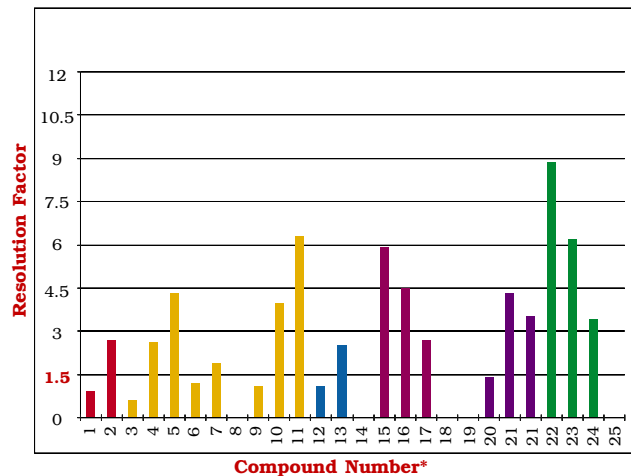
Rt- β DEXsp



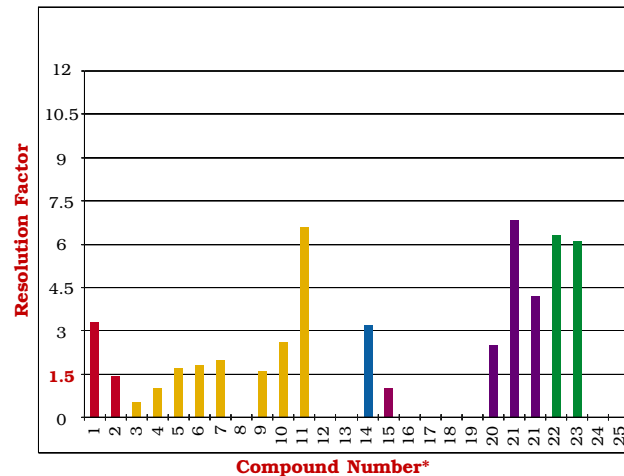
Rt- β DEXsa



Rt- β DEXcst



Rt- β DEXm



*Refer to Table I for compound identification.

OPTIMIZATION OF CHIRAL SEPARATIONS

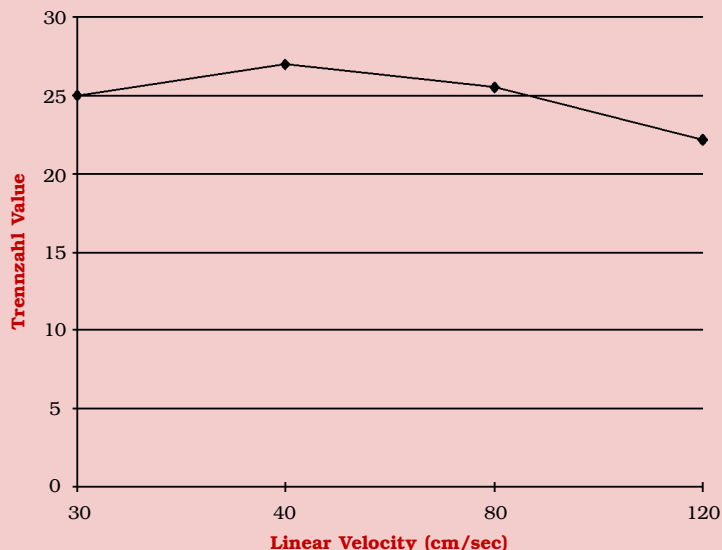
Although the new β -cyclodextrin columns can resolve a variety of chiral compounds, certain parameters must be optimized to obtain maximum separation and column performance. Variation in linear velocity and temperature ramp rate can greatly affect the resolution of enantiomers. Depending on the type of chiral column, initial GC oven temperature can affect peak width. Column sample capacity varies with different compounds, and overloading results in broad tailing peaks and reduced enantiomeric separation.

Linear Velocity (Column Flow)

The resolution between the enantiomeric pairs can be improved by increasing the linear velocity. This is especially important if the resolution factor is below two for optical isomers (see Table I). Trennzahl values are measurements of column separation efficiency, which are often optimum at a linear velocity of 40 cm/sec with hydrogen carrier gas. This is illustrated in **Figure 12A**. Although optimal linear velocity can be different for each chiral compound and column, the typical optimum linear velocity for maximum enantiomeric separation is around 80 cm/sec with hydrogen carrier gas, as illustrated with six chiral compounds on the Rt- β DEXsa column in **Figure 12B**. This is twice the linear velocity required to achieve maximum efficiency as indicated by the Trennzahl values of 1-octen-3-ol enantiomers in **Figure 12A**.

Figure 12A

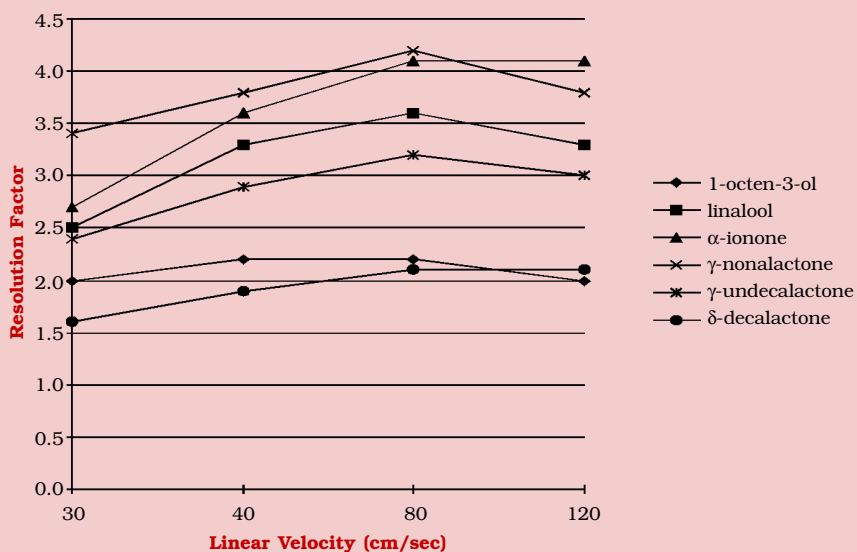
Higher Trennzahl values are obtained at 40 cm/sec with hydrogen carrier gas.



Rt- β DEXsa: 40°C to 200°C @ 2°C/min. (hold 1 min.).
Hydrogen carrier gas.

Figure 12B

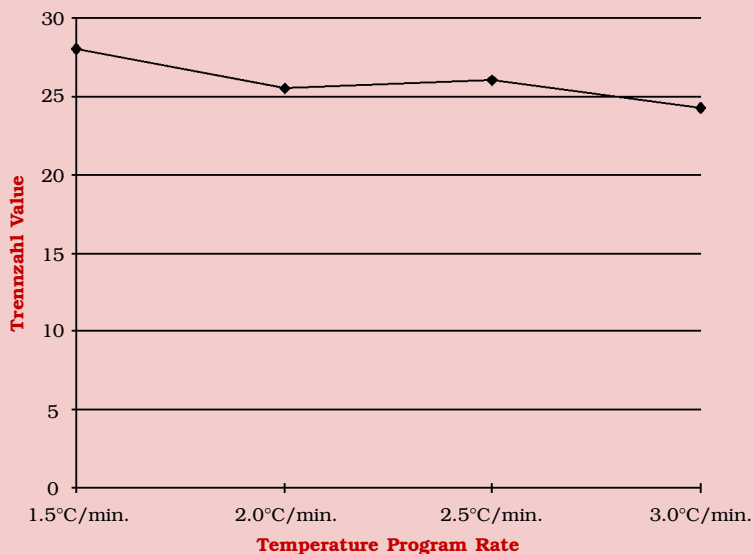
Faster linear velocities provide maximum resolution of chiral pairs.



Rt- β DEXsa: 40°C (80°C for lactones) to 200°C @ 2°C/min. (hold 1 min.). Hydrogen carrier gas.

Figure 13A

Trennzahl values increase with enantiomeric resolution factors as temperature ramp rates decrease.



Rt-βDEXsa: 40°C to 200°C @ 2°C/min. (hold 1 min.).
Hydrogen carrier gas.

Temperature Program

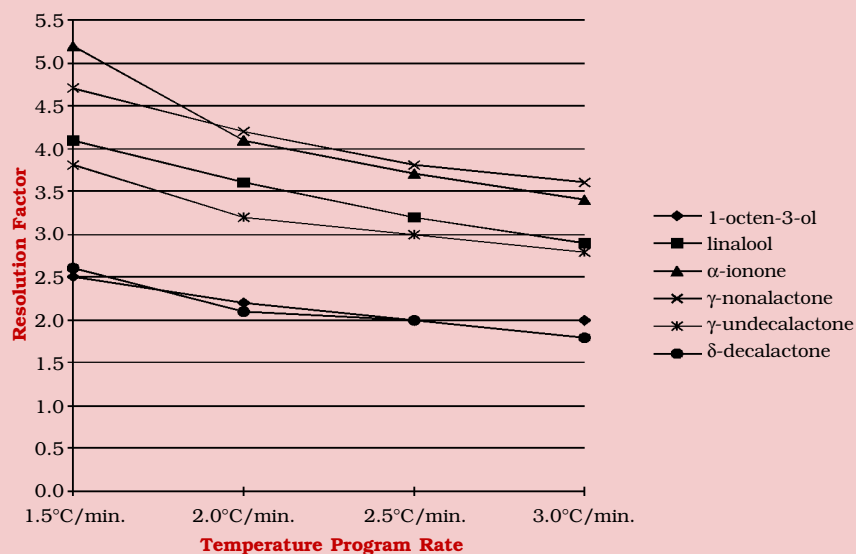
The resolution between the enantiomeric pairs can be improved by using slow temperature ramp rates. The best temperature ramp rates are 1-2°C/min. Trennzahl values improve along with enantiomeric resolution as the temperature ramp rate is decreased (**Figures 13A and B**).

Remember, to optimize chiral separation use:

- 1) **Faster linear velocities (80 cm/sec.) with hydrogen carrier gas.**
- 2) **Slower temperature ramp rates (1-2°C/min.).**
- 3) **Appropriate minimum operating temperature (40 or 60°C).**
- 4) **On-column concentrations of 50ng or less.**

Figure 13B

Lower temperature ramp rates provide maximum resolution of chiral pairs.



Rt-βDEXsa: 40°C (80°C for lactones) to 200°C @ 2°C/min. (hold 1 min.). Hydrogen carrier gas.

Call 800-356-1688, ext. 4, for knowledgeable technical support Monday–Thursday, 8 a.m.–7 p.m., Friday, 8 a.m.–6 p.m., or Fax anytime at 814-353-1310.

RESTEK CORPORATION

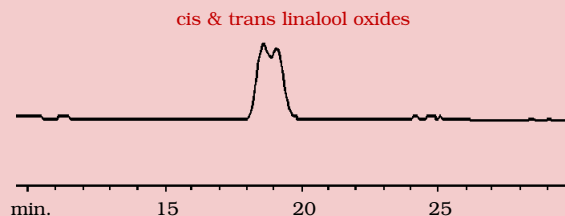
Minimum Temperature

For maximum resolution of chiral compounds with low boiling points (below 100°C), initial temperatures of 35–40°C are recommended for the Rt-βDEXsm, Rt-βDEXse, and Rt-βDEXsa columns. In contrast, the same volatile compounds exhibit a very broad peak shape on the Rt-βDEXsp and Rt-βDEXcst columns at these initial oven temperatures. Linalool oxides are volatile compounds that exhibit peak broadening and almost no resolution on the Rt-βDEXcst column with an initial oven temperature of 40°C (**Figure 14A**). The peak shapes and overall resolution of the linalool oxides improve when initial temperature is increased to 70°C, even though the individual enantiomers of both the cis and trans isomers are not separated (**Figure 14B**). Higher initial temperatures do not always completely eliminate peak broadening for some components. However, the improvement in solvent peak shape indicates a physical transition of the cyclodextrin macromolecules from a crystalline structure to a liquid at this higher temperature. Thus the recommended minimum operating temperature of Rt-βDEXsp and Rt-βDEXcst columns is 60°C.

Higher initial temperatures do not always completely eliminate peak broadening for some components. However, the improvement in solvent peak shape indicates a physical transition of the cyclodextrin macromolecules from a crystalline structure to a liquid at this higher temperature.

Figure 14A

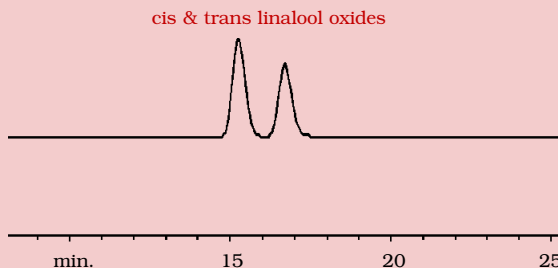
Linalool oxides exhibit extreme peak broadening and poor resolution on the Rt-βDEXcst column with an initial oven temperature of 40°C.



30m, 0.32mm ID, 0.25µm Rt-βDEXcst (cat.# 13102)
Oven temp.: 40°C (hold 1 min.) to 200°C @
 2°C/min.; **Carrier gas:** hydrogen; 80cm/sec. set @ 40°C;
Detector: FID set @ 220°C

Figure 14B

Linalool oxides exhibit improved peak shape and resolution on the Rt-βDEXcst column when initial oven temperature is increased to 70°C.

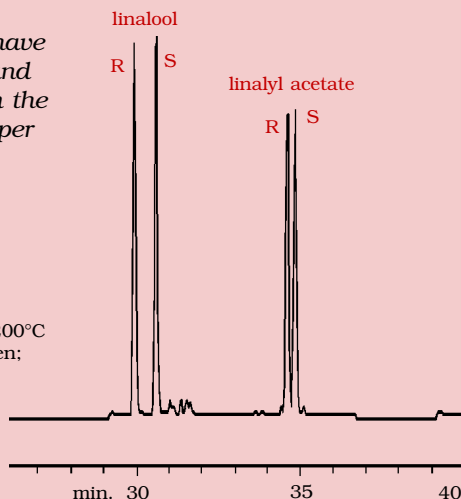


30m, 0.32mm ID, 0.25µm Rt-βDEXcst (cat.# 13102)
Oven temp.: 70°C (hold 1 min.) to 200°C @
 2°C/min.; **Carrier gas:** hydrogen; 80cm/sec. set @ 40°C;
Detector: FID set @ 220°C

Figure 15A

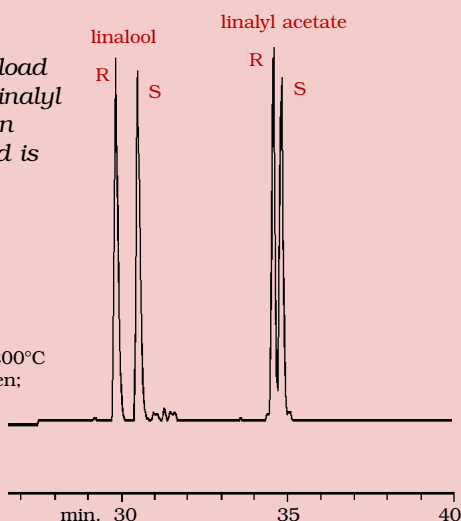
Linalool and linalyl acetate have symmetrical peak shapes and excellent chiral separation on the Rt- β DEXse column at 25ng per component on-column.

30m, 0.32mm ID, 0.25 μ m
Rt- β DEXse (cat.# 13106)
Oven temp.: 40°C (hold 1 min.) to 200°C
@ 2°C/min.; **Carrier gas:** hydrogen;
80cm/sec. set @ 40°C;
Detector: FID set @ 220°C

**Figure 15B**

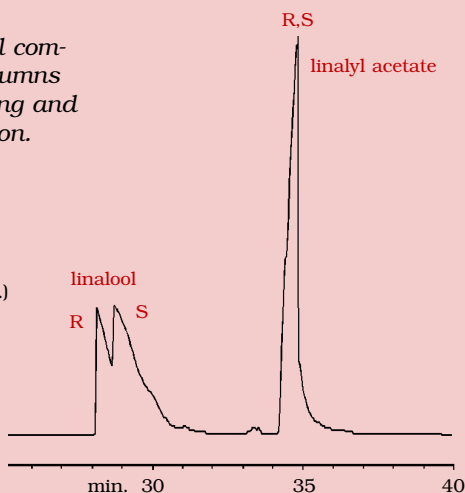
Linalool shows signs of overload with slight peak tailing, and linalyl acetate has a small loss in resolution when sample load is increased to 160ng.

30m, 0.32mm ID, 0.25 μ m
Rt- β DEXse (cat.# 13106)
Oven temp.: 40°C (hold 1 min.) to 200°C
@ 2°C/min.; **Carrier gas:** hydrogen;
80cm/sec. set @ 40°C;
Detector: FID set @ 220°C

**Figure 15C**

Excessive overload of chiral compounds on cyclodextrin columns results in extreme peak tailing and complete loss in resolution.

30m, 0.32mm ID, 0.25 μ m
Rt- β DEXse (cat.# 13106)
Oven temp.: 40°C (hold 1 min.)
to 200°C @ 2°C/min.;
Carrier gas: hydrogen;
80cm/sec. set @ 40°C;
Detector: FID set @ 220°C



Overloading and Tailing

Some chiral compounds show overloading at lower concentrations than achiral compounds. One reason is the different amounts of cyclodextrin (5-50%) dissolved in the stationary phase. Unlike the classical fronting peaks of normal stationary phases, the characteristic of an overloaded peak on cyclodextrin stationary phases is indicated by a tailing peak. Overloading chiral compounds results in loss of resolution, even when column capacity has not been exceeded. **Figure 15A** shows the enantiomeric separations of linalool and linalyl acetate on the Rt- β DEXse. The amount for each component in the column is about 25 ng. **Figure 15B** shows the same components at a higher concentration of 160ng on-column. Note that the linalool enantiomers are beginning to tail, and there is a small loss in chiral resolution for linalyl acetate. Even though the maximum sample capacity for 0.32mm ID capillary columns is normally 400-500ng per component, the peak shapes of chiral compounds indicate overload at one-third of the sample amount. Again, there is much less cyclodextrin for which a chiral compound can interact. **Figure 15C** shows pronounced overloading of these compounds at 5 μ g on-column. Extreme tailing and complete loss in resolution are the result.

CHIRAL SPECIFIC APPLICATIONS OF ESSENTIAL OILS, FLAVORS, AND PHARMACEUTICALS

ESSENTIAL OILS

Chiral capillary GC has proven to be a convenient method for characterizing essential oils and differentiating natural flavors from those of synthetic origin. Chiral compounds from natural origins usually exist as one predominant optical isomer. Also, the inspection of enantiomeric ratios can characterize regional differences between oils. Although sometimes a result of processing, the presence of racemic pairs (one-to-one ratios of each enantiomer) most often indicates adulteration or unnatural origin.

Since most chiral compounds naturally exist as one predominant isomer, resolution is more challenging, especially for components in higher concentrations. For primary constituents in essential oils, select a chiral column that provides a resolution factor value greater than two to overcome possible loss of resolution.

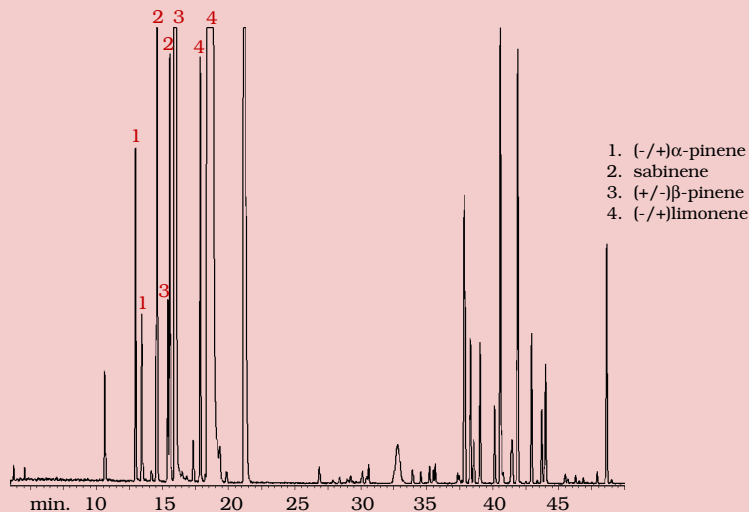
Since essential oils are mixtures of many compounds, coelution of peaks and overlapping of certain optical pairs are sometimes hard to avoid. Not all of the chiral compounds found in an essential oil or flavor extract may separate on the same column. Connecting two different columns together is possible, but the elution order of some enantiomers may reverse with this combination, resulting in loss of separation. Dual column analysis is a logical alternative to obtain a more complete enantiomeric profile and to provide confirmational identification of individual constituents. To reduce analysis time, both columns can be installed into the same injection port for simultaneous confirmation. (Consult Restek's Chromatography Products Guide for more information about dual column analysis.)



*For primary constituents
in essential oils, select
a chiral column that
provides a resolution
factor value greater than
two to overcome possible
loss of resolution.*

Figure 16

The Rt- β DEXsm column provides chiral resolution of the primary terpenes in Artificial Lemon oil.



1. (-/+) α -pinene
2. sabinene
3. (+/-) β -pinene
4. (-/+limonene

30m, 0.32mm ID, 0.25 μ m Rt- β DEXsm (cat.# 13104);
Oven temp.: 40°C (hold 1 min.) to 200°C @ 2°C/min. (hold 3 min.);
Carrier gas: hydrogen; 80cm/sec.; **Detector:** FID set @ 220°C

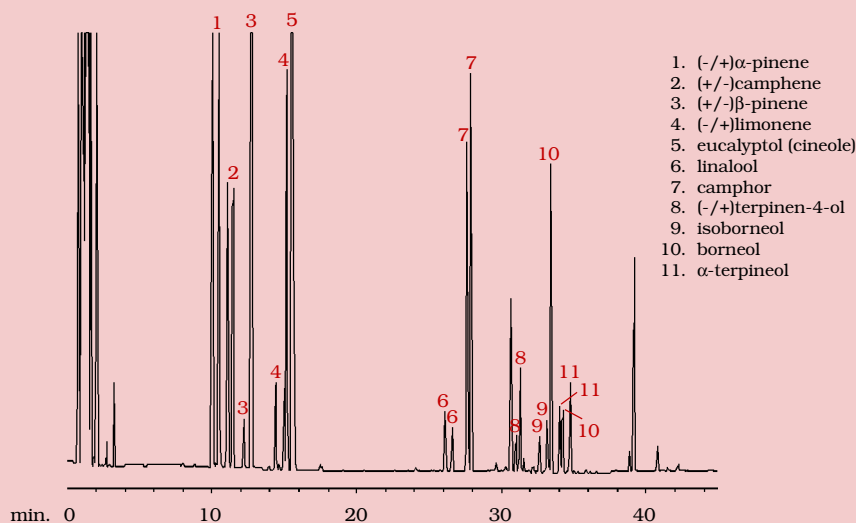
Lemon Oil

The Rt- β DEXsm is the optimum column for obtaining chiral profiles of Lemon and other citrus oils since it provides enantiomeric separation for the main terpene constituents like α - and β -pinenes, sabinene (these enantiomers overlap with those of β -pinene) and limonene (**Figure 16**).

The Rt- β DEXsm is the optimum column for obtaining chiral profiles of Lemon and other citrus oils.

**Figure 17**

The Rt- β DEXsm, the most versatile β -cyclodextrin column for essential oil analysis, resolves enantiomers of α -pinene, β -pinene, camphene, limonene, linalool, camphor, terpinen-4-ol, α -terpineol, borneol, and isoborneol in rosemary oil.



1. (-/+) α -pinene
2. (+/-)camphene
3. (+/-) β -pinene
4. (-/+limonene
5. eucalyptol (cineole)
6. linalool
7. camphor
8. (-/+terpinen-4-ol
9. isoborneol
10. borneol
11. α -terpineol

30m, 0.32mm ID, 0.25 μ m Rt- β DEXsm (cat.# 13104);
Oven temp.: 40°C (hold 1 min.) to 200°C @ 2°C/min. (hold 3 min.);
Carrier gas: hydrogen; 80cm/sec.; **Detector:** FID set @ 220°C

Rosemary Oil

Chiral constituents in this oil include α -pinene, β -pinene, camphene, limonene, linalool, camphor, terpinen-4-ol, α -terpineol, borneol, and isoborneol. Baseline enantiomeric separation is easily achieved for all of these compounds on the new Rt- β DEXsm column. The common permethylated β -cyclodextrin column cannot completely resolve the optical isomers of limonene, linalool, and camphor (**Figure 17**).

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Peppermint Oil

The Rt- β DEXsm column is optimum for the separation of (+/-) α - and β -pinene, limonene and menthone. Since menthone and menthol enantiomers are major constituents of peppermint oil, reducing the sample size to prevent over-loading of these components and provide better enantiomeric resolution may be necessary.

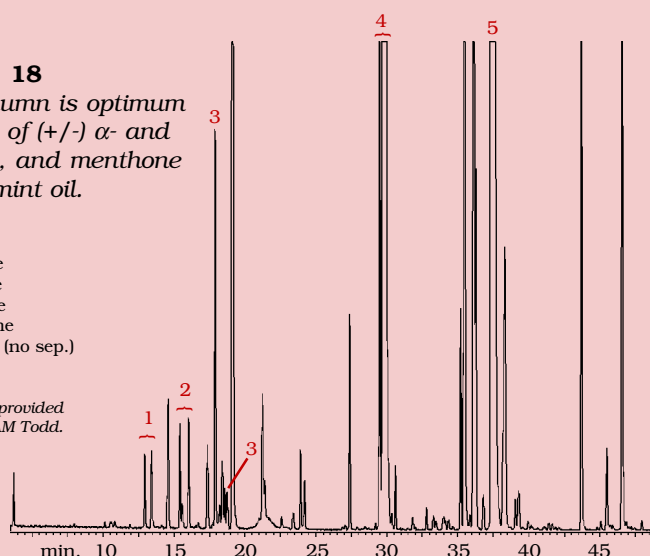
An alternative solution is to use an Rt- β DEXsp as a secondary column since it provides better resolution of menthol. However, do not use this column in a dual column system with an Rt- β DEXsm. The minimum temperatures of these phases differ by 20°C, which would minimize volatile terpene separation on the Rt- β DEXsm (**Figure 18**).

Figure 18

The Rt- β DEXsm column is optimum for the separation of (+/-) α - and β -pinene, limonene, and menthone in peppermint oil.

1. (-/+) α -pinene
2. (+/-) β -pinene
3. (-/+) limonene
4. (+/-) menthone
5. (+/-) menthol (no sep.)

Peppermint Oil provided
by AM Todd.



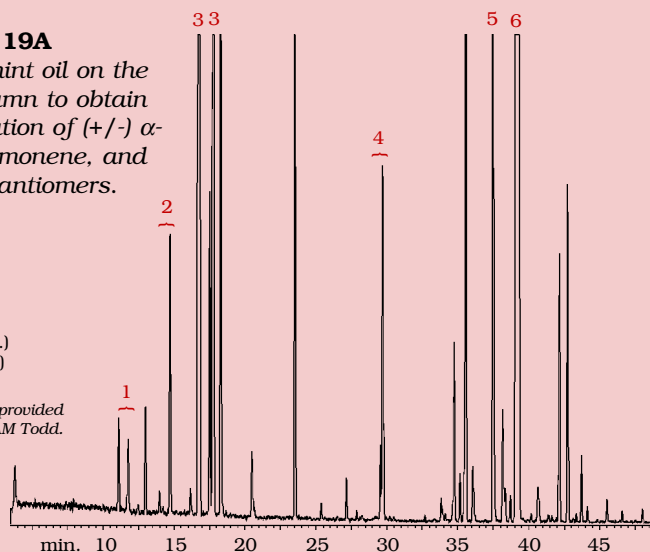
30m, 0.32mm ID, 0.25 μ m Rt- β DEXsm (cat.# 13104); **Oven temp.:** 40°C (hold 1 min.) to 200°C @ 2°C/min. (hold 3 min.); **Carrier gas:** helium; 60cm/sec.; **Detector:** MSD set @ 220°C

Figure 19A

Analyze spearmint oil on the Rt- β DEXsm column to obtain maximum separation of (+/-) α - and β -pinene, limonene, and menthone enantiomers.

1. α -pinene
2. β -pinene
3. limonene
4. menthone
5. menthol (no sep.)
6. carvone (no sep.)

Spearmint Oil provided
by AM Todd.



30m, 0.32mm ID, 0.25 μ m Rt- β DEXsm (cat.# 13104); **Oven temp.:** 40°C (hold 1 min.) to 200°C @ 2°C/min. (hold 3 min.); **Carrier gas:** helium; 60cm/sec.; **Detector:** MSD set @ 220°C

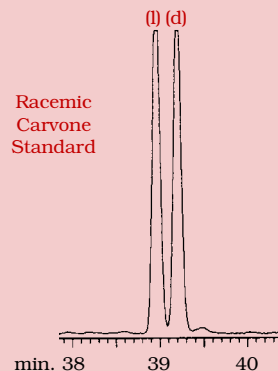
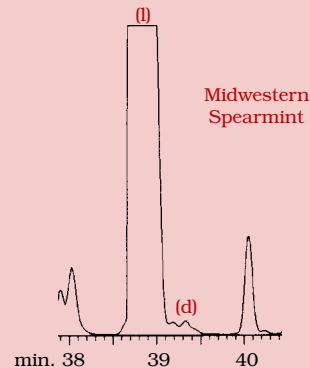
Spearmint Oil

The Rt- β DEXsm column yields maximum separation of (+/-) α - and β -pinene, limonene and menthone enantiomers in spearmint oil. The enantiomeric ratios of the primary chiral constituents in an artificial spearmint oil can be seen in the chromatogram shown in **Figure 19A**.

Although the optical isomers of carvone best separate on the Rt- β DEXsa column, α -pinene and limonene do not. For the separation of carvone, use a dual column system comprised of 30-meter Rt- β DEXsm and Rt- β DEXsa columns, since both have a minimum operating temperature of 40°C. **Figure 19B** compares carvone in natural sources of spearmint oil to a racemic standard on the Rt- β DEXsa column.

Figure 19B

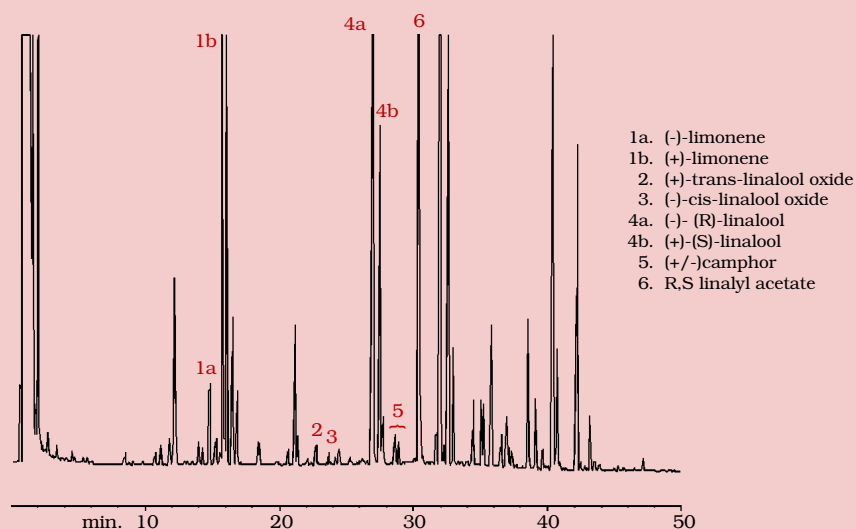
The optical isomers of carvone best separate on the Rt- β DEXsa column.



30m, 0.32mm ID, 0.25 μ m Rt- β DEXsa (cat.# 13108); **Oven temp.:** 40°C (hold 1 min.) to 200°C @ 2°C/min.; **Carrier gas:** helium; 60cm/sec. set @ 40°C; **Detector:** MSD set @ 220°C

Figure 20A

Linalool and linalool oxides in lavender oils are stereochemically separated on the Rt-βDEXsm column.



30m, 0.32mm ID, 0.25μm Rt-βDEXsm (cat.# 13104);
Oven temp.: 40°C (hold 1 min.) to 200°C @ 2°C/min. (hold 3 min.);
Carrier gas: hydrogen; 80cm/sec.; **Detector:** FID set @ 220°C
Lavender oils provided by Belmay.

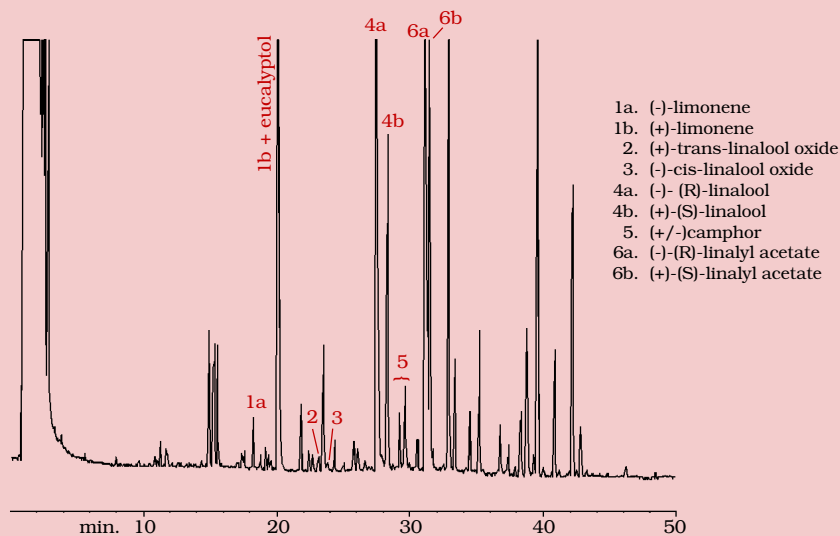
Lavender Oil

The Rt-βDEXsm column separates the enantiomers of the primary chiral compounds found in lavender oil, including linalool. Both the cis and trans enantiomeric pairs of the furanoid linalool oxides, which contribute characteristic odors to lavender oils and Clary sage oil, are separated on this column. (R)-Linalool is present in at least 85% enantiomeric excess. (2R)-Configured linalool oxides are present in about 77% enantiomeric excess in authentic Lavender oils.² Both the cis and trans (R)-linalool oxides are essentially enantiomerically pure in this oil, as shown in **Figure 20A** (peaks 2 and 3).

Linalyl acetate is another primary constituent in lavender oils. The (R)-(-) enantiomer is predominant in authentic lavender oils.³ A dual column system comprised of both the Rt-βDEXsm and Rt-βDEXse columns can be used to resolve the enantiomers of linalyl acetate as well (peak 6 in **Figure 20B**). Note that the (-)-(R)-enantiomer constitutes >92% of linalyl acetate in this lavender oil.

Figure 20B

The Rt-βDEXse column resolves enantiomers of linalyl acetate in lavender oils.



30m, 0.32mm ID, 0.25μm Rt-βDEXse (cat.# 13106);
Oven temp.: 40°C (hold 1 min.) to 200°C @ 2°C/min. (hold 3 min.);
Carrier gas: hydrogen; 80cm/sec.; **Detector:** FID set @ 220°C
Lavender oils provided by Belmay.

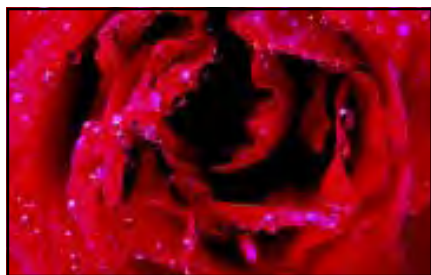
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ext. 4, for technical
assistance.

Geranium Oil

Chiral constituents in geranium oils include cis and trans rose oxides, linalool, and β -citronellol. The Rt- β DEXsa column provides chiral resolution for all of these compounds. In authentic samples of geranium oil, (-)-(4R)-configured diastereomers of cis- and trans-rose oxides predominate over their (+)-enantiomers.³ The (-)-(S) form of β -citronellol is 74-80% of the enantiomeric ratio.⁴ Note that cis- and trans-rose oxides and β -citronellol are racemic in this particular commercial geranium oil, as shown in **Figure 21A**. These racemic compounds indicate that this oil is not authentic.

Rose Oil

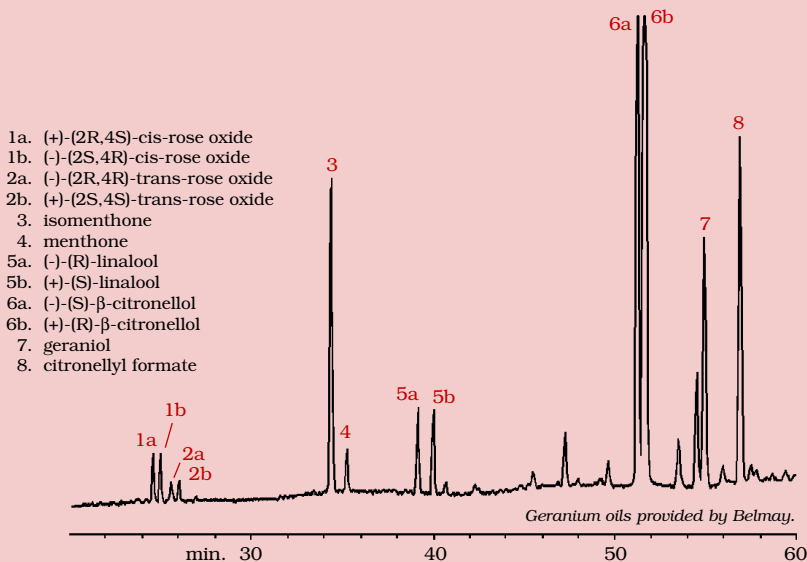
As with geranium oils, (-)-(2S,4R)-cis and (-)-(2R,4R)-trans rose oxides and (-)-(S)- β -citronellol are specific indicators of genuine rose oils.⁵ Note the enantiomeric purity of these compounds in Rose Oil Maroc, as shown in **Figure 21B**.



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 assistance.

Figure 21A

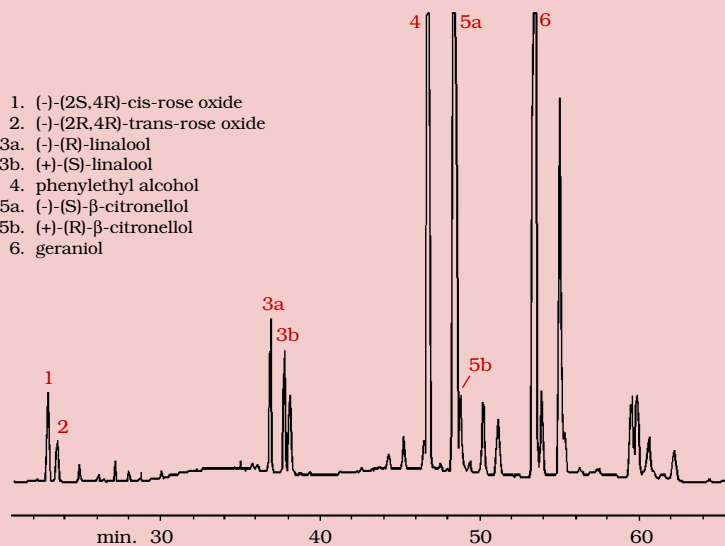
The Rt- β DEXsa column provides chiral resolution for cis and trans rose oxides, linalool, and β -citronellol in Chinese Geranium oil.



30m, 0.25mm ID, 0.25 μ m Rt- β DEXsa (cat.# 13109);
Oven temp.: 60°C to 110°C @ 1°C/min. (hold 30 min.);
Carrier gas: hydrogen; 40cm/sec. set @ 60°C; **Detector:** FID set @ 220°C

Figure 21B

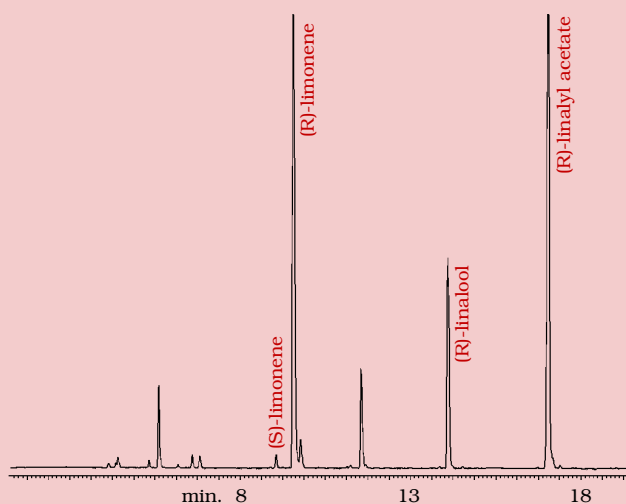
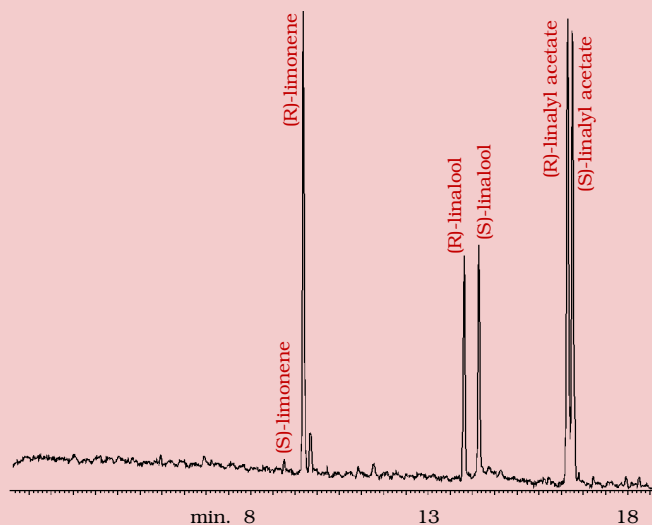
The Rt- β DEXsa column reveals authenticity of Rose oil Maroc.



30m, 0.25mm ID, 0.25 μ m Rt- β DEXsa (cat.# 13109);
Oven temp.: 60°C to 110°C @ 1°C/min. (hold 30 min.);
Carrier gas: hydrogen; 40cm/sec. set @ 60°C; **Detector:** FID set @ 220°C

Figure 22

The Rt- β DEXse column can differentiate Bergamot extract from Bergamot flavor.

A: Bergamot Extract**B: Bergamot Flavor**

30m, 0.32mm ID, 0.25 μ m Rt- β DEXse (cat.# 13106);

Oven temp.: 40°C (hold 1 min.) to 200°C @ 4°C/min.;

Carrier gas: helium; 60cm/sec. set @ 40°C; Detector: MSD set @ 220°C

FLAVORS

Bergamot oil and a few of the popular fruit flavorings such as raspberry, strawberry, and peach were examined. The composition of extracts from natural sources were compared to those from commercially available flavored teas and drinks. Some target chiral compounds examined were linalool and linalyl acetate in bergamot oil, α -ionone and δ -decalactone in raspberry, and γ -lactones in peach extracts.

Bergamot Flavor

A genuine cold-pressed bergamot oil should contain only the (R)-isomers of linalool and linalyl acetate.⁶ The enantiomeric purity of (R) limonene should also be considered.⁷ Chromatogram A in **Figure 22** is a natural source of bergamot oil. Only the (R)-enantiomers of limonene, linalool and linalyl acetate were present. Chromatogram B illustrates an extract from an artificially flavored tea. Both samples were analyzed on an Rt- β DEXse column. The presence of racemic linalool and linalyl acetate indicates bergamot flavor of unnatural origin.

Peach Flavor

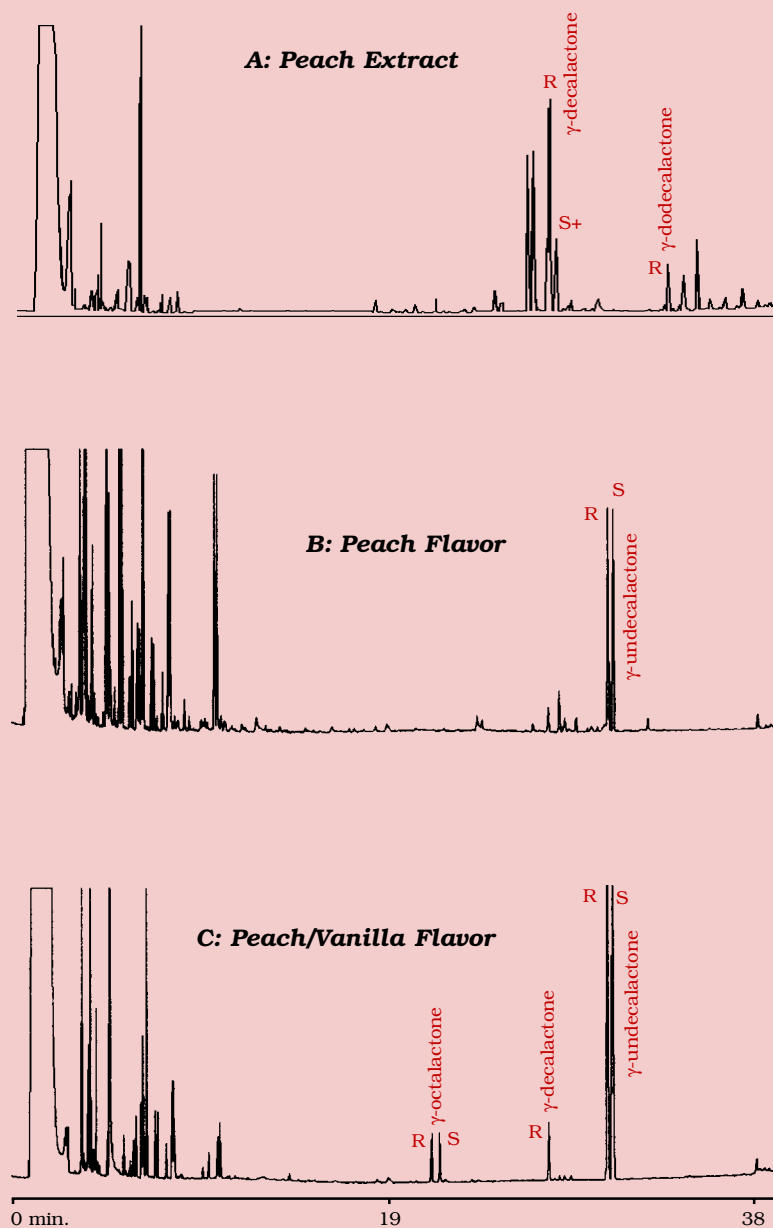
Both δ - and γ -lactones are present in peaches, but only the γ -lactones are analyzed for the adulteration of peach flavor. Gamma-decalactone occurs in the 89% (R) : 11%(S)- enantiomers in natural Peach flavor.⁸ In **Figure 23**, Chromatogram A is a peach extract. A significant amount of the (R)- γ -decalactone was present, along with the (S)-enantiomer, which coeluted with an unknown. A small amount of (R)- γ -dodecalactone was also detected. Chromatogram B is an extract from a beverage with “all natural” peach flavor. Gamma-decalactone was not present, but racemic γ -undecalactone was found in a 1:1 ratio. This was the same result with another peach-flavored beverage. Chromatogram C is from an “all natural-flavored beverage,” with peach and vanilla flavors. Although only the (R)-enantiomer of γ -decalactone was present, the amount is very small. Both γ -octalactone and γ -undecalactone were found to be racemic, indicating adulteration.



The γ -lactones are inspected for the adulteration of peach flavor.

Figure 23

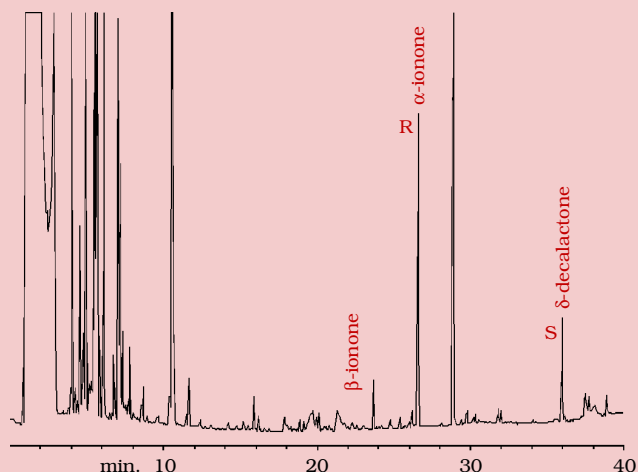
The γ -lactones are analyzed for the adulteration of peach flavor on the Rt- β DEXsa column.



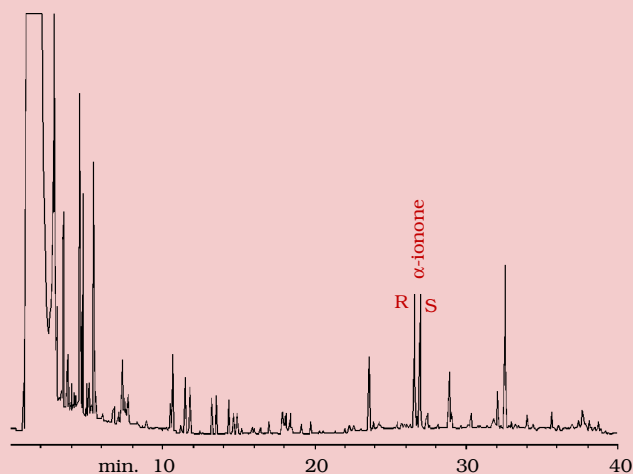
30m, 0.32mm ID, 0.25 μ m Rt- β DEXsa (cat.# 13108); **Oven temp.:** 60°C (hold 2 min.) to 100°C @ 15°C/min., then to 220°C @ 3°C/min.;
Carrier gas: helium; 60cm/sec. set @ 60°C; **Detector:** MSD set @ 220°C.

Figure 24

The Rt- β DEXsa column resolves isomers of α -ionone to determine raspberry authenticity.

A: Raspberry Extract

30m, 0.32mm ID, 0.25 μ m Rt- β DEXsa (cat.# 13108);
Oven temp.: 60°C (hold 2 min.) to 200°C @ 3°C/min.;
Carrier gas: helium; 60cm/sec. set @ 60°C; **Detector:** MSD set @ 220°C.

B: Raspberry Flavor

30m, 0.32mm ID, 0.25 μ m Rt- β DEXsa (cat.# 13108);
Oven temp.: 60°C (hold 2 min.) to 200°C @ 3°C/min.;
Carrier gas: helium; 60cm/sec. set @ 60°C; **Detector:** MSD set @ 220°C.

Raspberry Flavor

Alpha-ionone from raspberries occurs as an enantiopure (R)(+)-enantiomer, illustrated on an Rt- β DEXsa column in **Figure 24A**. Chromatogram B represents a “naturally flavored” raspberry iced tea. A racemic mixture of α -ionone was present, indicating that it is not a completely natural raspberry flavor. Thus, α -ionone serves as a good marker compound for determining raspberry authenticity.



*Alpha-ionone serves as a
 good marker compound
 for determining
 raspberry authenticity.*

DRUGS

Stereochemical properties of chiral drugs have been found in many instances to be the controlling factor concerning activity. One enantiomer may provide a biological function. The other may be inactive or exhibit another functionality, which could result in side effects. In some cases, one optical isomer may be harmful. The FDA requires drug manufacturers to test the individual enantiomers of new drugs for toxicity.

Fenfluramine

Fenfluramine is an appetite suppressant to promote weight loss with obese patients.⁹ Although it is structurally similar to amphetamines, it differs somewhat pharmacologically. Norfenfluramine is a metabolite that is found in urine and serum of patients. The purpose of the chiral isolations, like many analyses in the pharmaceutical industry, is of proprietary nature. The TFA derivatives of both fenfluramine and norfenfluramine are separated into their enantiomers on the Rt- β DEXcst column (Figure 25).

Barbiturates

Mephobarbital and Hexobarbital are barbiturates with sedative, hypnotic and anticonvulsant properties.¹⁰ Because psychological and physical dependence may occur with continuing use, they are controlled substances in the U.S. Code of Federal Regulations. The optical isomers of these barbiturates can be simultaneously resolved on an Rt- β DEXcst column (Figure 26).

Amphetamines

Dextroamphetamine (*d*-amphetamine), *d,l*-amphetamine, and *d*-methamphetamine are sympathomimetic amines with central nervous system stimulant activity.¹¹ They are significant drugs of abuse in the United States and are included among the drugs to be tested under

Figure 25

The optical isomers of TFA-fenfluramine and its metabolite are well resolved on the Rt- β DEXcst column.

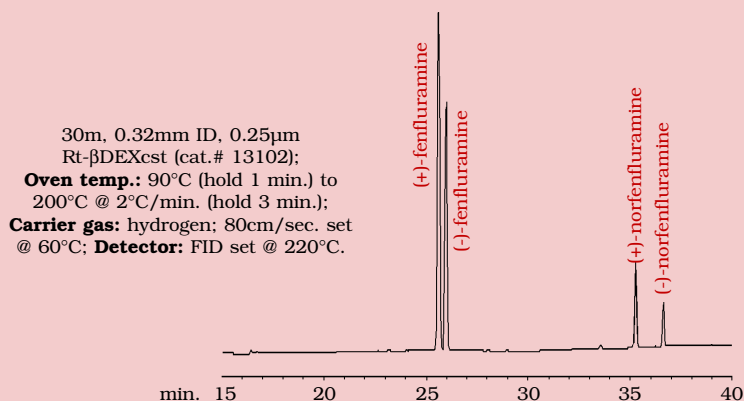


Figure 26

The Rt- β DEXcst column can resolve the enantiomers of common barbiturates.

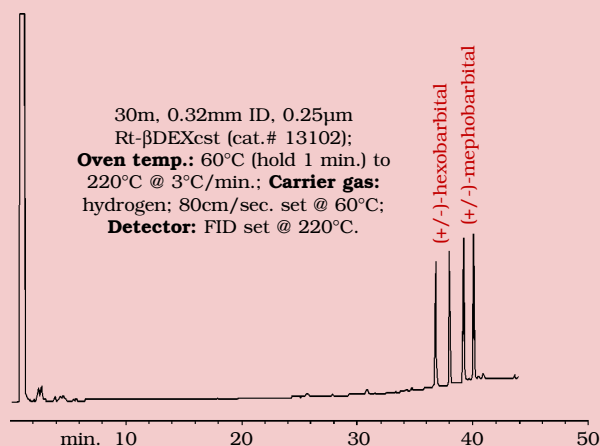
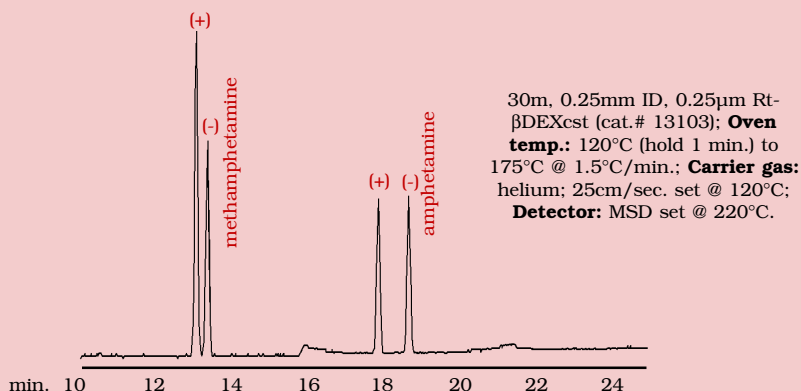


Figure 27

The Rt- β DEXcst column simultaneously resolves the enantiomers of TFA-amphetamine and TFA-methamphetamine.



the federal guidelines for workplace drug testing.¹² However, *l*-methamphetamine (deoxyephedrine) is found in over-the-counter decongestants and is not a controlled substance.¹³ Enantiomeric separation of these

compounds, which is necessary for accurate interpretation of drug tests, is easily achieved on the Rt- β DEXcst chiral capillary GC column (**Figure 27**).



Stereochemical properties of chiral drugs have been found in many instances to be the controlling factor concerning activity. One enantiomer may provide a biological function and the other may be inactive or may exhibit another functionality, which could result in side effects.

PRODUCT LIST

Rt- β DEXsm (30m, 0.25 μ m)				Rt- β DEXse (30m, 0.25 μ m)			
mm ID	cat.#	min. temp.	max. temp.	mm ID	cat.#	min. temp.	max. temp.
0.25	13105	40°C	230°C	0.25	13107	40°C	230°C
0.32	13104	40°C	230°C	0.32	13106	40°C	230°C
Rt- β DEXsa (30m, 0.25 μ m)				Rt- β DEXsp (30m, 0.25 μ m)			
0.25	13109	40°C	230°C	0.25	13111	60°C	230°C
0.32	13108	40°C	230°C	0.32	13110	60°C	230°C
Rt- β DEXcst (30m, 0.25 μ m)				Rt- β DEXm (30m, 0.25 μ m)			
0.25	13103	60°C	230°C	0.25	13100	40°C	230°C
0.32	13102	60°C	230°C	0.32	13101	40°C	230°C

Restek Trademarks: Rtx, Rt- β DEX, and the Restek logo.

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Operating Hints for Using Split/Splitless Injectors



Inside:

Overviews of split and splitless injection techniques

Backpressure-regulated injection systems

Headpressure-regulated injection systems

Operating in the split injection mode

Inlet liners for split injections

Operating in the splitless injection Mode

Inlet liners for splitless injections

Septum purge optimization

Problems associated with split and splitless injections

Direct injection as an alternative to splitless injection

Hints for analyzing dirty samples

Hints for performing routine injection port maintenance

Product listing

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Overview of Split/Splitless Injection Techniques

In capillary and micropacked gas chromatography (GC) there are four primary techniques for vaporizing a sample and transferring it onto the inlet of the analytical column: split, splitless, direct, and on-column injections. Of these, split and splitless injections are the most commonly used techniques. This technical guide focuses on split and splitless injections—their optimization, troubleshooting, and system maintenance.

Split and splitless injections are techniques that introduce the sample into a heated injection port as a liquid, and then rapidly and completely vaporize the sample solvent as well as all of the analytes in the sample. The vaporized sample is transferred to the head of the column.

In the split injection mode, only a fraction of the vaporized sample is transferred onto the head of the column. The remainder of the vaporized sample is removed from the injection port via the split vent line. Split injections should be used only when sample concentrations are high enough to allow a portion of the sample to be discarded during the injection process, while still maintaining a sufficient concentration of analytes at the detector to produce a signal.

When target analyte concentrations are so low that splitting the sample in the injection port will not allow an adequate signal from the detector, the injector should be operated in the splitless injection mode. In the splitless injection mode, most of the vaporized sample is transferred to the head of the column.

The process of performing either a split or splitless injection is controlled by changing the flow path and flow rate of carrier gas through the injection port. The position of a switching valve in the injection port determines the flow path. In split injections, a high carrier gas flow rate rapidly moves the vaporized sample through the injection port liner, past the column (with only a minimal amount directed to the head of the column), and out the split vent. In splitless injections, a relatively slow carrier gas flow rate directs most of the vaporized sample into the head of the column.

Split/splitless injection ports can be either backpressure-regulated or headpressure-regulated systems. Most modern GCs are backpressure regulated. However, some GC manufacturers still find headpressure regulation advantageous and use this design in their split/splitless injectors. It is important for analysts to be familiar with their injection port hardware and the operating principles of their instruments, so that they factor in the variables affecting the accuracy and reproducibility of their results.

Backpressure-Regulated Injection Systems

Figure 1 illustrates the components of a typical backpressure-regulated split/splitless injection system (e.g., Agilent 5890, 6850, 6890 GCs; Varian 3300, 3400, 3500, 3600, 3800 GCs; Shimadzu 17A GCs). A flow controller, positioned upstream from the injection port, controls the total amount of carrier gas that enters the injection port. A backpressure regulator, located downstream from the injection port body, regulates the pressure inside the injection port. Carrier gas flow rate in the column is determined by the pressure that is maintained in the injection port. The outlet of the backpressure regulator is the outlet of the split vent line. The split vent line outlet is at the ambient pressure of the laboratory. The flow controller and the backpressure regulator work together to determine the column flow rate, septum purge flow rate, and split vent flow rate.

Split and splitless injections in backpressure-regulated systems are controlled by the position of the 3-way solenoid valve. In the split injection mode, the flow path is always open from the injection port body through the 3-way solenoid valve to the split vent line. In the splitless injection mode, the flow path is temporarily closed from the injection port body to the split vent line. The carrier gas flow rate through the injection port liner is simply the column flow rate. Any excess flow is directed through the septum purge line, into the 3-way solenoid valve, and out the split vent line.

In backpressure-regulated systems, the split vent flow rate is changed by adjusting the flow controller. An increase in the total flow being delivered to the injection port will result in a higher split vent flow rate and a higher split ratio. Column flow rate is not affected by changes in the total flow being delivered to the injection port, but by the backpressure regulator. To maintain the same pressure at all times, use the backpressure regulator to compensate for a change in the total flow delivered to the injection port.

A flow-controlled, backpressure-regulated system is beneficial as it gives some measure of protection against a catastrophic loss of carrier gas. If there is a leak at an injection port fitting or a column fitting, the maximum rate of carrier gas loss would be the total flow rate into the injection port as determined by the flow controller. Unlimited flow of carrier gas into the injection port is prevented by having the flow controller at the inlet of the injection port. Leaks are indicated by a failure to maintain split vent flow rate. A common mistake analysts make when they observe a reduced split vent flow rate is to increase the total system flow, rather than check for leaks at the injector and column fittings. By understanding the characteristics of backpressure regulated pneumatics, analysts can detect and correct a leak, to avoid poor chromatography.

Figure 1.

Split injection flowpaths in a typical flow-controlled/backpressure-regulated system.

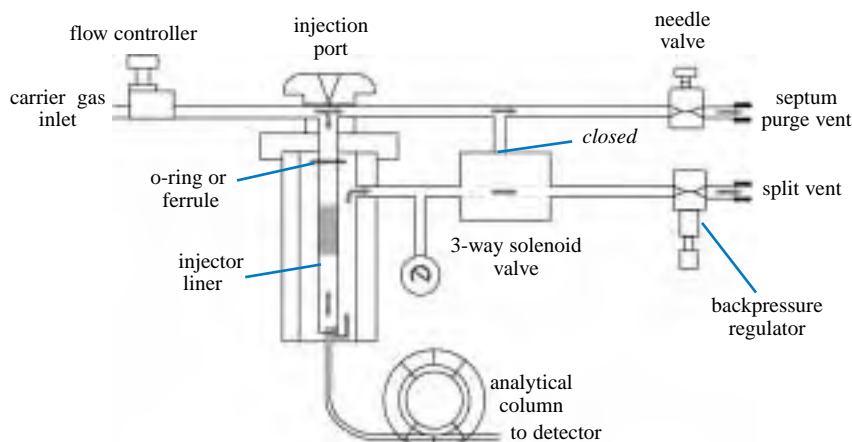


Figure 1.

- All carrier gas except septum purge flow directed through injector.
- Column flow (established by backpressure regulator) enters column.
- Solenoid valve open from injector to split vent. Bulk of gas flows out of injector liner, through solenoid valve, out split vent.
- Sample vapor is directed onto column or vented through split vent and is split in the same proportions as for carrier gas.
- Split ratio = portion of sample vented from split vent/portion of sample that enters column.

Headpressure-Regulated Injection Systems

Figure 2 illustrates the components of a typical headpressure-regulated split/splitless injection system (e.g., PE Autosystem; Shimadzu 9A & 14A; Thermo Finnigan Trace 2000 GCs). A pressure regulator upstream from the injection port regulates or maintains the pressure inside the injection port. The pressure regulator supplies an unlimited flow of carrier gas until the desired pressure is reached. The pressure inside the injection port establishes the carrier gas flow in the column and determines the column flow rate. Flows through the split vent line and the septum purge line are controlled by needle valves or restrictors downstream from the injection port. The outlet pressure of the septum purge and split vent lines is ambient pressure. As long as constant pressure is maintained in the injection port, needle valves and restrictors will give constant flows.

Figure 2.

Split injection flowpaths in a typical headpressure-regulated system.

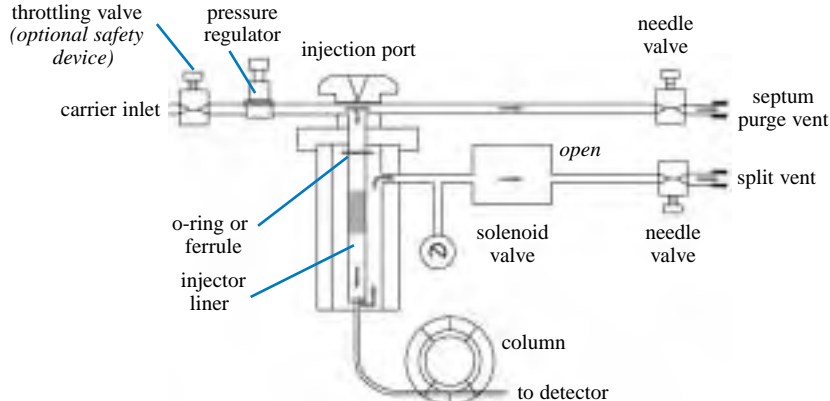


Figure 2.

- Solenoid valve open: column flow passes into column, split flow exits through split vent.
- Throttling valve guards against loss of carrier gas caused by leaks in injection system.

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Restek Flowmeter 6000



- Calculates linear velocity based on column ID.
- Useful for measuring flows for N₂, air, He, H₂, CO₂, O₂, Ar, 7.5% CH₄/Ar.
- Reads flow accurately from 0 to 500mL/min. (0–300mL/min. for CO₂).
- Accuracy is 0.2mL/min. or +/- 2.5%.
- Usable with inlet pressures up to 25psi.
- Measures split flow and calculates split ratio.
- Automatic shut-off.



Description	qty.	cat.#
Restek Flowmeter 6000 (9-volt battery-operated)	ea.	21622
Recalibration Service for Restek Flowmeter 6000	ea.	24618

Soap Film Bubble Flowmeters

- 1mL flowmeter measures flows between 0.1 and 10cc/min.
- 50mL flowmeter designed for flows between 10 and 300cc/min.
- Both flowmeters come with a reservoir bulb, twenty-four inches of 1/4-inch ID tubing, adaptor tubes for 1/8-inch tubing and 0.53mm ID capillary columns, and Velcro® fasteners.



Description	qty.	cat.#
1mL Bubble Flowmeter	ea.	20135
50mL Bubble Flowmeter	ea.	20136

An on/off solenoid valve is used in headpressure-regulated systems instead of the 3-way solenoid valve used in backpressure-regulated systems. The position of the solenoid valve determines whether the injection port is operated in the split or splitless injection mode. In the split injection mode, the solenoid valve is always in the open position and the carrier gas is allowed to flow through the injection port liner and out the split vent line. In the splitless injection mode, the solenoid valve is closed and the only flow through the injection port liner is the column flow. The pressure regulator compensates for excess carrier gas flow available when the solenoid valve closes.

The throttling valve upstream from the pressure regulator (Figure 2) is an optional component not typically included by the chromatograph manufacturer. We recommend installing a throttling valve (flow controller or needle valve) to guard against catastrophic loss of carrier gas if a leak occurs at an injection port fitting or a column fitting. To adjust the throttling valve, gradually close the valve, reducing the gas flow until it matches the requirements of the injection system. When the column headpressure begins to decrease, the throttling valve is closed too far.

Operating in the Split Injection Mode

When operating in the split injection mode (Figures 1 and 2), the solenoid valve is always open along the flowpath from the injection port body to the split vent. With the exception of the septum purge flow, all of the carrier gas entering the injection port flows through the injection port liner and toward the head of the column. At the head of the column, the carrier gas flow is split between two flow paths: a portion of the flow enters the column as the column flow rate, and the remaining carrier gas flow is allowed to escape from the injection port, out the split vent line via the solenoid valve. The amount of flow entering the column is determined by the pressure of the carrier gas inside the injection port and the dimensions of the analytical column. The relative proportions of the split vent flow and the column flow determine the split vent ratio.

Samples completely vaporized in the injection port liner behave in the same fashion as the carrier gas; sample vapors are split in the same proportions as the carrier gas, thereby allowing only a fraction of the sample to be introduced into the head of the column. A 50-to-1 split ratio can be used as a starting point when developing split injection methods. Table I shows the appropriate split vent flow rates for helium and hydrogen carrier gases when using common capillary column IDs.

Table I.

Typical split vent flow rates for 50-to-1 split ratio at optimum linear velocity when using a 30-meter column at 40°C.

Carrier Gas	Column ID (mm)/Split Vent Flow Rate			
	0.18	0.25	0.32	0.53
helium*	25cc/min.	37.5cc/min.	55cc/min.	135cc/min.
hydrogen**	50cc/min.	75cc/min.	110cc/min.	270cc/min.

*optimum carrier gas linear velocity=20cm/sec.

**optimum carrier gas linear velocity=40cm/sec.

Equation 1 shows how the split ratio is calculated. Split vent flow rates easily can be measured using a standard electronic flowmeter (cat.# 21622). However, measuring low flow rates (from 0.3 to 5cc/min.) exiting a capillary column can be difficult unless a special low-volume bubblemeter (cat.# 20135) or a sensitive electronic flowmeter is used. If a low flow-measuring device is not available, Equation 2 can be used to determine the approximate column flow.

Calculating the on-column concentration of analytes is necessary to ensure that the column is not overloaded and is operating within its capacity limits. Although quantitative analysis does not require that the on-column concentration be known, exceeding column capacity decreases resolution and reduces quantitative accuracy. Equation 3 illustrates how to calculate the approximate on-column concentration in the split mode.

Setting the injection port temperature properly is critical for obtaining good peak shape and response. Injection port temperature must be hot enough to provide rapid vaporization of all

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Equation 1.*Calculating the split ratio.*

$$\text{Split ratio} = \frac{\text{column flow} + \text{split vent flow}}{\text{column flow}}$$

Equation 2.*Calculating the approximate column flow rate.*

$$\text{Flow} = \frac{(\pi) (\text{column radius in cm})^2 (\text{column length in cm})}{\text{dead volume time (min.)}}$$

where $\pi = 3.14159$

For example, a 30m x 0.53mm ID column operated at 20cm/sec. linear velocity (helium) retains methane for 2.50 min., and therefore has a flow rate of 2.65cm³/min.:

$$\text{Flow} = \frac{(3.14159) (0.0265\text{cm})^2 (3000\text{cm})}{2.50 \text{ min.}} = 2.65\text{cm}^3/\text{min.}$$

Equation 3.*Calculating the approximate on-column concentration for split injections.*

$$\text{Concentration} = \frac{\text{concentration in sample } (\mu\text{g}/\mu\text{L}) \times \text{sample vol. injected } (\mu\text{L})}{\text{split ratio}}$$

sample components. In the split injection mode, the residence time of the sample in the injection port is very short because of the high carrier gas flow rate through the injection port liner and out the split vent. As a result, vaporization must be completed as rapidly as possible. However, injection port temperatures must not be so high that they cause sample degradation.

When set up properly, split injections are very reproducible. Samples introduced under constant temperature, pressure, and flow conditions will vaporize and split consistently.

Split injections can be used for both qualitative and quantitative work. Internal or external reference compounds are split under identical conditions compared to analytes in samples. Any variations experienced by the sample also are experienced by the reference compounds when the sample matrix and standard matrix match exactly. In general, split inlet liners are designed to have added surface area to help with sample vaporization. Improved vaporization can be achieved with changes in liner geometry that increase the surface area. Examples include incorporating fused silica or glass wool, CarboFrit™ packing, or using a laminar cup.

Caution!

When analyzing hazardous compounds in the split mode, make sure they do not enter the lab atmosphere through the split vent. A small, charcoal-filled split vent trap connected to the split vent protects you from breathing contaminated air (cat. # 20698).

**High-Capacity Split Vent Trap**

- Reduces the release of hazardous materials from the capillary split vent into the lab.
- Lasts one month or 1,500 injections.
- Includes connecting lines and mounting kit.



Description	qty.	cat. #
High-Capacity Split Vent Trap	ea.	20698
High-Capacity Split Vent Trap	5-pk.	20699

For customer service, call
800-356-1688, ext. 3
 (814-353-1300, ext. 3)
 or call your local
 Restek representative.

Methane Cylinder

Setting the column flow rate by injecting methane and optimizing linear velocity is a preferred method for establishing reproducible retention times (ASTM Method E1510-93). Measuring the linear velocity of your carrier gas is made easy by using the Scotty® 14 cylinder containing 1% methane in helium. The complete kit includes the Scotty® 14 cylinder, a MINICYL regulator, a syringe adaptor, and a package of twenty septa for the adaptor.

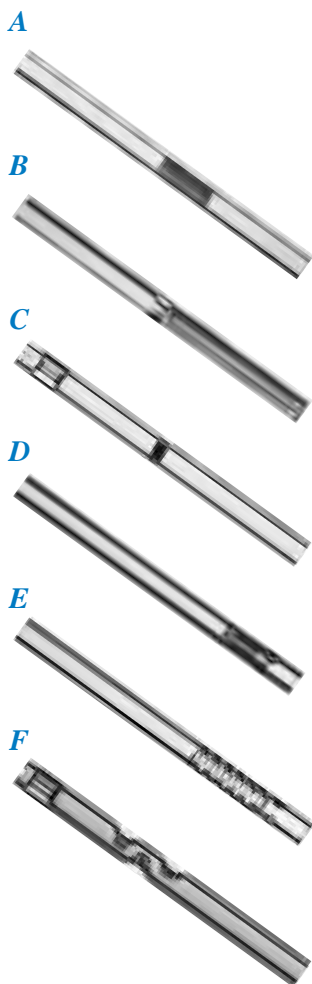
Description	qty.	cat. #
Complete Kit	kit	20197
Replacement Septa	20-pk.	20198
Replacement Cylinder	ea.	20199

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Inlet Liners for Split Injections

Split liners are designed with mixing chambers and tortuous flow paths, to fully vaporize the sample into a homogeneous vapor cloud before it reaches the split point. All Restek split liners are fully deactivated using a high-temperature silanizing reagent. This caps surface silanol groups so active compounds in the sample do not degrade or adsorb onto the hot glass surface.

To trap non-volatile residue and prevent column contamination when analyzing dirty samples, pack split liners with wool, CarboFrit™ packing, or fused silica beads. Some of the more commonly used inlet liners are described below.



A) Split Liner with Wool

The wool provides a large surface area to allow rapid vaporization of the sample and deliver a uniform vapor cloud to the split point. The low mass of the wool fiber promotes complete vaporization.

Benefits:

- Low cost.
- Reproducible performance.

Drawbacks:

- Wool can be adsorptive, especially if fibers are broken.
- High maintenance requirements.

B) Laminar Cup Splitter

The sample flows through a small opening and encounters the head of the elongated glass cup. It then travels around the outside of the elongated cup before the flow is inverted twice. Larger volume injections are possible because the liquid is trapped at the inner base and cannot escape until vaporized.

Benefits:

- Recommended by chromatography expert Dr. Konrad Grob¹.
- Best splitter liner for high molecular weight compounds.
- Laminar flow profile provides highest resolution.

Drawbacks:

- Costly.

C) Frit Splitter

The sample must pass through the porous ceramic frit. The high surface area and tortuous flow path ensure complete vaporization.

Benefits:

- Traps septum particles and residue.

Drawbacks:

- Ceramic frit can be active.
- Difficult to clean.

D) Cup Splitter

The sample flows through a mini funnel and encounters a glass cup. The flow path then inverts twice before reaching the split point.

Benefits:

- Tortuous flow path aids in sample vaporization.
- Minimizes molecular weight discrimination.
- Can be packed with wool to trap particles.

Drawbacks:

- Difficult to clean.

E) Cyclosplitter® (Patent #: 5,119,669)

This patented design incorporates a cylindrical glass spiral in the sample pathway, providing a large area for sample vaporization.

Benefits:

- Ideal for dirty samples.
- Allows many injections of dirty samples before cleaning is required.
- Easy to clean.

Drawbacks:

- Not recommended for large volume injections.

F) Baffle Splitter

The baffle induces turbulent flow that directs the sample against the wall of the glass liner.

Benefits:

- Reproducible performance.

Drawbacks:

- Prone to molecular weight discrimination.
- Septum particles and residue can enter column.
- Subject to incomplete vaporization.

**all liners are
100%
deactivated**

See page 17.

All Restek liners are deactivated to prevent adsorption of active compounds. Call for information on custom deactivations.

¹"Injectors Providing Complete Sample Evaporation Above the Column Entrance in Vaporizing GC Injections," K. Grob and C. Wagner, *HRC & CC*, Vol. 16, p. 429.

Operating in the Splitless Injection Mode

When operating in the splitless injection mode (Figures 3 and 4), the solenoid valve is switched, changing the flow path of the carrier gas. At the beginning of a splitless injection, the solenoid valve is set to prevent the flow of carrier gas from the injection port body through the solenoid valve. When the solenoid valve is in this position, only the column flow moves through the injection port liner. Column flow rate is determined by the pressure of the carrier gas in the injection port as set by the pressure regulator and the analytical column dimensions.

Figure 3.

Splitless injection flowpaths (injector purge off) in a typical flow-controlled/backpressure-regulated system.

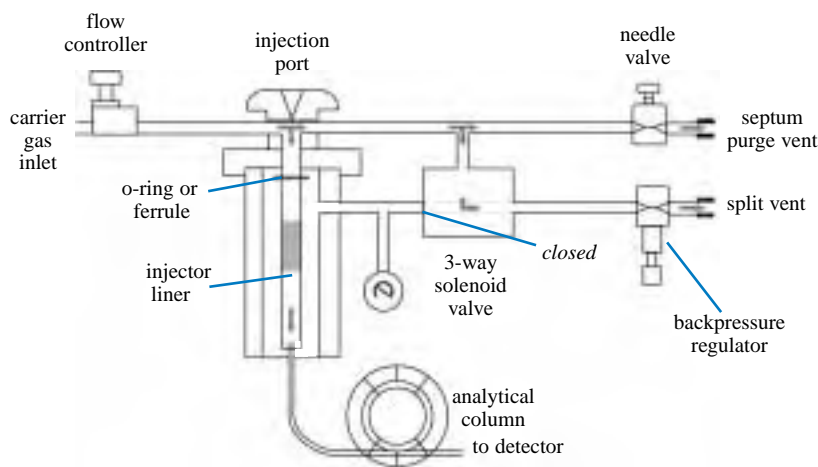


Figure 3.

- Solenoid valve closed between injector and split vent: only column flow enters injector; column flow passes into column.
- Needle valve at septum purge vent allows only septum purge flow to exit septum purge vent: most of carrier gas diverted through solenoid valve, out through split vent.
- Sample vapor in injector liner can exit only to column, mixed with column flow of carrier gas.
- Solenoid valve switched to establish flowpaths as in split injection: sample vapor remaining in injection port swept out of split vent.
- Splitless hold time determined by sample composition.

Figure 4.

Splitless injection flowpaths in a typical headpressure-regulated system.

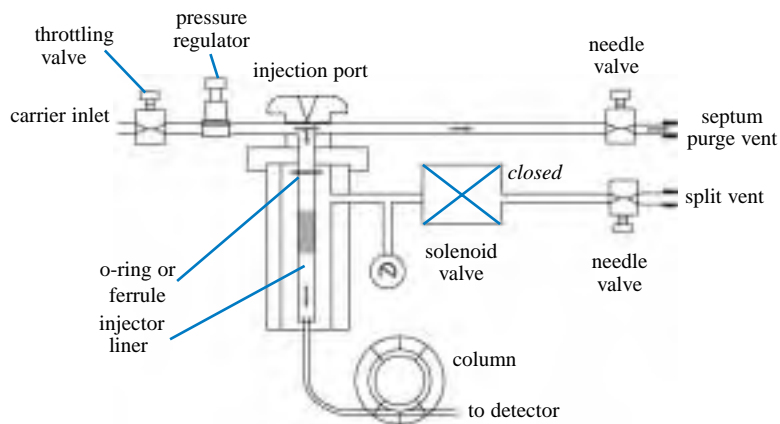


Figure 4.

- Solenoid valve closed: entire carrier gas flow and entire sample directed onto analytical column.
- Carrier gas flow rate into system reduced to enable entire flow to pass through analytical column.

After a carefully determined time (the splitless hold time) the solenoid valve is switched to re-establish the flow paths as used in the split injection mode. This allows any vaporized sample remaining in the injection port to be quickly swept out of the injection port liner through the split vent. A typical splitless hold time is between 60 and 90 seconds. The ideal splitless hold time is long enough to allow most of the vaporized sample in the injection port liner to be transferred to the analytical column. Excessively long splitless hold times can produce tailing peaks and broad peaks. The splitless hold time must be determined through experimentation, and will vary according to sample composition, column length and

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Table II. Typical splitless hold times.

Column ID	Hold Time	Column Flow Rate		Sample Transfer Time*	
		He	H ₂	He	H ₂
0.18mm	2 min.	0.3cc/min.	0.6cc/min.	2.7 min.	1.4 min.
0.25mm	1 min.	0.7cc/min.	1.4cc/min.	1.2 min.	0.6 min.
0.32mm	0.75 min.	1.2cc/min.	2.4cc/min.	0.7 min.	0.4 min.
0.53mm	0.5 min.	2.6cc/min.	5.2cc/min.	0.3 min.	0.2 min.

*2 μ L of liquid methylene chloride expanded to 0.8mL vapor at 250°C (10psig headpressure).

ID, carrier gas flow rate, and injection port liner configuration. Table II lists approximate splitless hold times for various column IDs when operated with helium or hydrogen. The splitless hold time will decrease as either the column ID or column flow rate increases.

Setting an optimal splitless hold time also is dependent on the choice of sample solvent and the sample size. Use Table III to estimate the volume of vapor produced when using different solvents at different pressures. The volume of vapor cloud formed should be divided by the column flow rate to determine the approximate time needed to keep the solenoid valve closed for complete sample transfer. The calculated splitless hold time also should be evaluated to provide the optimum response for the sample analytes. If the solenoid valve is

Table III.
Solvent expansion volumes.

Solvent	Density (g/mL)	MW	Expansion Volume in μ L at various column headpressures		
			5psig	10psig	15psig
Heptane	0.68	100	219	174	145
Hexane	0.66	86	245	196	163
Pentane	0.63	72	280	224	186
Toluene	0.87	92	303	242	201
Ethyl acetate	0.90	88	328	261	217
Chloroform	1.49	119	400	319	266
Methylene chloride	1.33	85	500	399	332
Methanol	0.79	32	792	629	525
H ₂ O	1.00	18	1776	1418	1179

The expansion volumes were determined using a 1.0 μ L injection volume, a 250° C injection port temperature, and a headpressure of 5, 10, or 15psig (common operating pressures for 30m columns having IDs of 0.53, 0.32, or 0.25mm, respectively). For 2 μ L injections, double the expansion volumes.

Use these formulas to calculate values not listed in Table III:

$$\text{Expansion volume} = nRT / P$$

n = number of moles of solvent and sample.

$$= [\text{volume (mL)} \times \text{density (g/mL)}] / \text{mol. wt. (g/mole)}$$

R = gas law constant

$$= 82.06 \text{cc atm/mole } ^\circ\text{K}$$

T = absolute temperature of injector (°K)

$$(^\circ\text{K} = ^\circ\text{C} + 273)$$

P = absolute column headpressure = gauge pressure (atm) + 1 atm

$$\text{atm} = \text{psig} \times 0.06804 \text{ atm / psig}$$

$$\text{injector liner volume}^* = \pi r^2 L$$

$$\pi = 3.14$$

r = liner internal radius (cm)

L = liner length (cm)

*Also use this formula to determine capillary column internal volume.

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opened too quickly, responses will be low. However, if the solenoid valve remains closed too long, the solvent peak will tail and peak resolution will suffer. To help determine the optimal splitless hold time, a series of injections should be made using increasingly longer splitless hold times. When the response for the analytes of interest plateaus, the sample transfer process has been optimized (Figure 5).

Setting the injection port temperature for splitless injections is critical, just as it is for split injections. The injection port temperature must be high enough to completely vaporize the sample, yet not so high that it causes sample degradation. This is especially important because the residence time for a sample in the injection port during splitless injections is longer, compared to split injections.

Solvent Focusing and Analyte Focusing

The long residence time for samples in the injection port also affects peak shape. In splitless injections, samples are transferred to the head of the column over a longer period of time than in split injections. As a result, initial peak bandwidths can be very broad unless vaporized samples are refocused at the head of the column. Two techniques can be used to refocus vaporized samples at the head of the column: solvent focusing and analyte focusing. The difference between the two methods is the initial temperature of the column oven. For solvent focusing, the initial oven temperature is low enough to allow the solvent to recondense at the head of the column. This forms a zone of liquid solvent that traps all of the vaporized sample analytes in a narrow band at the head of the column. Analyte focusing requires an initial oven temperature that allows the solvent to move through the column as a vapor immediately after injection. Analytes that have a significantly higher boiling point than the solvent are recondensed at the head of the column because of the lower oven temperature.

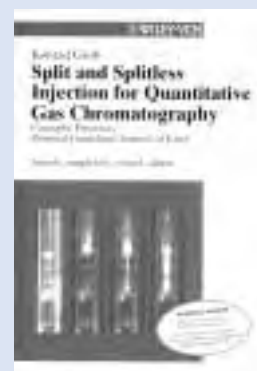
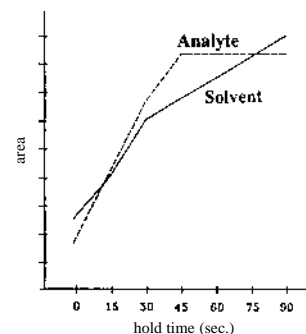
A typical sequence of events for performing a splitless injection using solvent focusing is as follows:

1. Set the initial oven temperature approximately 20°C below the boiling point of the sample solvent.
2. Close the solenoid valve to divert the entire sample onto the head of the column.
3. Inject the sample and hold the oven temperature at the initial temperature to recondense the solvent and focus the sample at the head of the column. The initial oven temperature is typically held for the same amount of time that the solenoid valve is closed.
4. Switch the solenoid valve to open the flow path to the split vent line and rapidly program the oven temperature (10 to 30°C/min.) until the first analyte of interest elutes.
5. Slow the oven program rate to enhance resolution of the remaining analytes of interest.

Figure 5.

Optimization of splitless hold time.

The splitless hold time is optimized when further increases do not increase analyte response but result in solvent tailing.



Split and Splitless Injection in Capillary GC, 4th Ed.

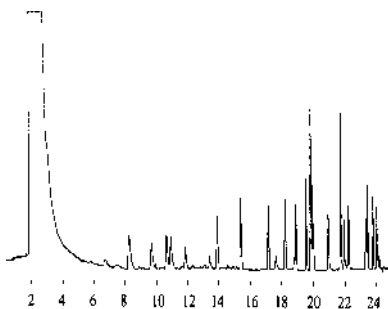
This comprehensive handbook of split and splitless injection techniques has been totally revised and updated, containing information on sample evaporation in the injector, matrix effects, and a new chapter on injector design. It also includes a CD-ROM containing visualization of the evaporation process during split and splitless injection.

K. Grob, Wiley-VCH, 2001, 460pp., ISBN 3-527-29879-7 cat.# 20451 (ea.)

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Figure 6.

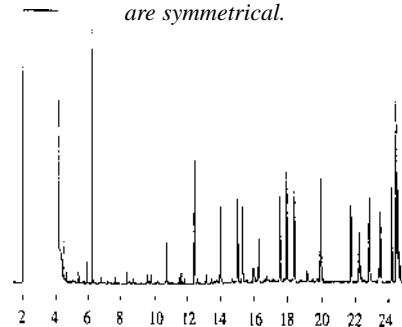
Initial oven temperature too high for improper solvent focusing: solvent peak and early eluting compounds are tailing.



30m, 0.25mm ID, 0.25µm Rtx®-5 (cat.# 10223)
1.0µL splitless injection of a pesticide mix in
hexane (5ng/µL);
Oven temp.: 150°C to 275°C @ 4°C/min.

Figure 7.

Initial oven temperature at least 20°C below boiling point of earliest eluting analyte: early eluting compounds are symmetrical.



30m, 0.25mm ID, 0.25µm Rtx®-5 (cat.# 10223)
1.0µL splitless injection of a pesticide mix in
hexane (5ng/µL); **Oven temp.:** 40°C to 150°C
@ 25°C/min. then to 275°C @ 4°C/min.

The sequence of events for analyte focusing is the same, except for the initial oven temperature; instead of starting 20°C below the boiling point of the solvent, the oven temperature is started 60–80°C below the boiling point of the earliest eluting compound.

Figure 6 shows an example of improper solvent focusing. The sample solvent is hexane, which has a boiling point of 69°C. The initial oven temperature is 150°C, or 80°C above the boiling point of hexane. The solvent peak is tailing, and the early-eluting compounds have broad peak shapes and are poorly resolved from one another. Figure 7 illustrates proper solvent focusing. The initial oven temperature, 40°C, is well below the boiling point of hexane. The square solvent peak is a good indicator of proper solvent focusing. Also notice the sharp peak shapes for both early- and late-eluting compounds. When the solvent is not detected or elicits a low response, such as hexane with electron capture detectors (ECDs), the only indication of proper solvent focusing is narrow peaks for early-eluting compounds.

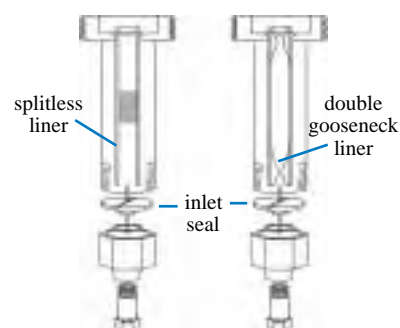
For optimal solvent focusing, choose a solvent that has a boiling point at least 20°C below the boiling point of the earliest eluting target analyte. In some cases, it is not possible to select the perfect solvent to achieve focusing. For example, methylene chloride (boiling point 40°C) is frequently used for splitless work because of sample preparation techniques. Analyses performed with an initial oven temperature of 40°C will not allow the solvent to recondense at the head of the column and will not refocus the sample analytes. Ideally, analysts would start the oven temperature at 20°C when using methylene chloride as the sample solvent, but because this is not practical, they must rely more on analyte focusing to refocus sample analytes at the head of the column.

An important part of solvent focusing is the ability of the solvent to “wet” the stationary phase in the column. Non-polar solvents should be used for splitless injections on non-polar stationary phases (e.g., use hexane or isooctane for injections on Rtx®-1 and Rtx®-5 columns). Non-polar solvents are more soluble in non-polar stationary phases and will form a more efficient zone of recondensed solvent in the column. Polar solvents are not as soluble in non-polar stationary phases and will bead up on the stationary phase rather than forming an even layer of recondensed solvent at the head of the column. Mismatches between the polarity of the solvent and the polarity of the stationary phase can cause band broadening, peak splitting, and poor resolution.

Once again, the same basic procedures are followed for analyte focusing, except the initial oven temperature is 60–80°C below the boiling point of the earliest eluting compound, instead of 20°C below the boiling point of the solvent, as with solvent focusing.

A unique situation with Agilent 5890 and 6890/6850 split/splitless inlets makes a double gooseneck liner highly desirable for samples that contain compounds prone to catalytic degradation through contact with hot metal surfaces. Agilent splitless inlets contain a metal seal at the base of the inlet (just under the liner outlet). Because the column is installed only a few millimeters above the seal surface, the sample contacts the seal while it is being slowly drawn into the column. A double gooseneck inlet liner minimizes contact between the sample and the metal seal. A dirty seal increases the breakdown of endrin (a pesticide prone to decomposition) from 6% to 12.8% in an Agilent 5890 inlet when a 4mm straight inlet liner is installed. However, when a double gooseneck inlet liner is used, the breakdown remains at 2% regardless of whether the seal is clean or dirty. (For more information, see page 24 of this guide for a description of our Vespel® Ring Inlet Seal.)

Double gooseneck inlet liner minimizes the catalytic effects of sample contact with the metal disk in an Agilent inlet.



Liner Type	Endrin Breakdown	
	Clean Seal	Dirty Seal
Splitless with Wool	6.0%	12.8%
Double Gooseneck	2.0%	2.4%

Inlet Liners for Splitless Injections

The residence time of the sample in a splitless liner is between 0.5 and 2 minutes, so splitless inlet liners do not require large surface areas for efficient vaporization (unless you are using a rapid-injecting autosampler). Splitless liners usually are designed as straight tubes. Alternative splitless liner designs, such as gooseneck restrictions, help contain the sample cloud in the injector and minimize the breakdown of compounds sensitive to catalytic decomposition on metal inlet parts. Splitless liners should be packed with wool or fused silica beads to help with vaporization, trap non-volatile residue, and prevent column contamination when analyzing dirty samples. Some of the more commonly used splitless liners are described below.

A) Straight Tube

Use for samples containing a narrow molecular weight distribution and for those not prone to thermal decomposition. Packing with wool is recommended. Wool aids in vaporization of high molecular weight compounds and minimizes discrimination.

Benefits:

- Low cost.

Drawbacks:

- Potential decomposition of active compounds such as endrin and phenols when packed with wool.
- Prone to high molecular weight discrimination.
- Sample exposed to metal surface below liner.

B) Gooseneck

C) Recessed Gooseneck

Benefits:

- Increases splitless efficiency.
- Decreases breakdown of active compounds such as endrin and DDT.
- Chamber contains sample vaporization cloud.
- Can be packed with wool.

Drawbacks:

- No known drawbacks.

D) Double Gooseneck

E) Recessed Double Gooseneck

Best liner for catalytically labile or high molecular weight compounds. Isolates sample from metal injection port parts. Use the cyclo-version for dirty samples.

Benefits:

- Highest splitless efficiency.
- Breakdown of active compounds decreased.
- Chamber contains vaporization cloud.

Drawbacks:

- Higher cost than straight splitless liners.
- Only recessed double goosenecks can be packed with wool.

Note: Recessed gooseneck liners offer the same benefits as single or double gooseneck liners, but the base of the recessed gooseneck can be packed with wool and the liner can be used for dual-column analysis with a two-hole ferrule.

F) Drilled Uniliner®

This direct injection liner features a hole drilled into the inlet end that reduces sample discrimination, compared to typical splitless injections.

Benefits:

- Excellent transfer of analytes to column.
- Decreases injection port discrimination.
- Removes excess solvent vapor.
- Eliminates the need for wool.
- No sample contact with metal parts below liner, less adsorption.

Drawbacks:

- Higher amounts of non-volatile materials transferred to column.

G) 4mm Splitless with Fused Silica Wool

The wool provides a large surface area to allow rapid vaporization of the sample and deliver a uniform vapor cloud to the split point. The low mass of the wool fiber promotes complete vaporization.

Benefits:

- Low cost.
- Reproducible performance.

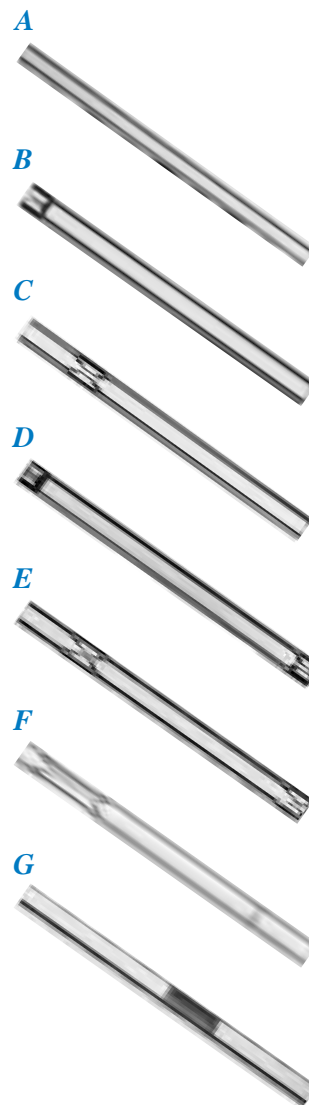
Drawbacks:

- Wool can be adsorptive, especially if fibers are broken.
- High maintenance requirements.

all liners are
100%
deactivated

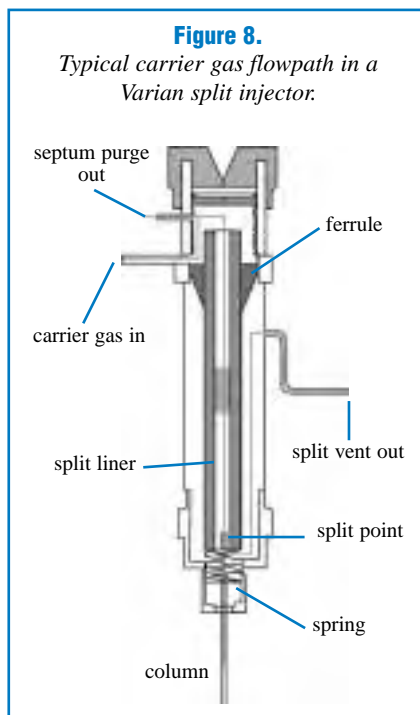
See page 17.

All Restek liners are deactivated to prevent adsorption of active compounds. Call for information on custom deactivations.



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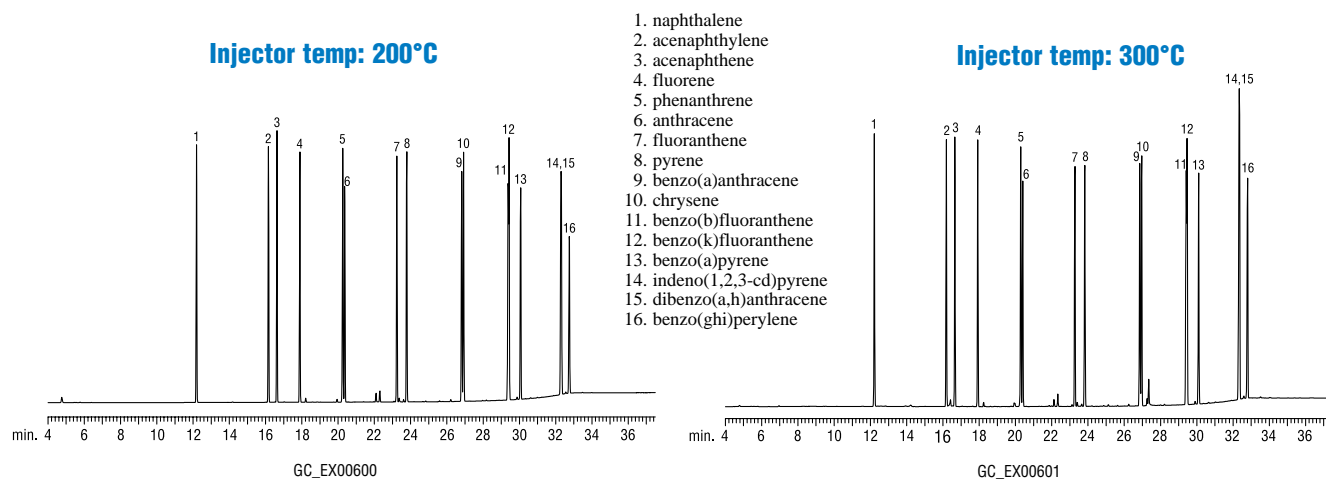
Septum Purge Optimization



The septum purge (Figure 8) serves two functions: to sweep septum bleed volatiles out of the system and to reduce the potential for sample backflash contaminating the carrier gas inlet line. Optimization of the septum purge flow rate is important, especially when the inlet is operated in the splitless mode. Most GC manufacturers recommend that the septum purge flow rate be set between 3 and 5cc/min. Flow rates exceeding 5cc/min. should not be used because highly volatile sample components could be preferentially purged from the inlet liner buffer volume after vaporization. Flow rates lower than 3cc/min. can allow septum bleed to enter the inlet liner and cause ghost peaks to appear on the chromatogram.

The septum purge flow rate must be readjusted each time the injection pressure is changed by more than 5psig. Most GCs have a low-flow needle valve that makes septum purge adjustments easy.

Figure 9.
Injector temperature affects the recovery of higher molecular weight compounds.



Rtx®-5 15m, 0.32mm ID, 1.50µm (cat.# 10266)
Sample: 50µg/mL PAH standard (cat.#31011) in hexane
Inj.: 1.0µL splitless (hold 2 min.),
4mm single gooseneck inlet liner w/FSwool (cat.# 22405)
Inj. temp.: 200°C
Carrier gas: helium, constant pressure
Linear velocity: 76cm/sec. @ 40°C
Oven temp.: 40°C(hold 4min.) to 325°C @10°C/min. (hold 5 min.)
Det.: FID @350°C

Figure 10.

The Donike Test illustrates the importance of injector temperature when a sample contains thermally labile compounds.

1. TMS tetracosanoate (thermolabile)
2. *n*-triacontane (stable)
3. TMS hexacosanoate (thermolabile)

15m x 0.32mm ID fused silica coated with 0.25µm bonded methyl silicone
 Sample: 1µL each of TMS *n*-tetracosanoate, TMS *n*-hexacosanoate, and *n*-triacontane in *n*-nonane at 2ng/µL each component.

GC: 3000 Series Varian gas chromatograph with 1077 split/splitless injector, FID and autosampler.

Split/splitless injector:

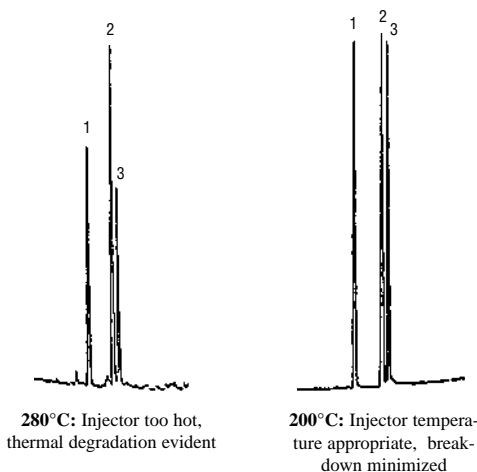
Run 1: SPI held at 280°C

Run 2: SPI held at 200°C

Carrier gas: helium at 47cm/sec.

Oven: 130°C to 280°C 20°C/min. (hold 2 min.)

FID: 300°C, 32 x 10⁻¹²



Chromatograms courtesy of Varian Instrument Co.

Problems Associated with Split and Splitless Injections

When performed properly, split and splitless injections are easy to automate, produce narrow peaks, and yield consistent run-to-run peak areas. However, split and splitless injections have inherent limitations associated with vaporizing the sample in a hot injection port.

Thermal Decomposition: The injection port temperature is a critical factor in optimizing hot vaporization injection techniques. If the injection port temperature is too low, high molecular weight analytes will not vaporize completely and will not be transferred to the head of the column efficiently (as shown by peaks 14, 15 and 16 in Figure 9). If the injection port temperature is too high, thermally labile compounds can break down inside the injection port before reaching the column. Figure 10 shows the effect of temperature on thermally labile TMS derivatives of fatty acids. When the injection port temperature is set at 280°C, the response for the TMS derivatives is reduced. When the injection port temperature is lowered to 200°C, the response for the TMS derivatives is comparable to triacontane at equivalent sample concentrations. Careful optimization of injection port temperatures will maximize sample vaporization while minimizing sample decomposition.

Active Compounds: Active compounds can be problematic in split or splitless injections. The high surface area and heat needed to uniformly vaporize the sample can cause these compounds to break down or be adsorbed onto the surface of the injection port liner. Deactivated inlet liners, and Silcosteel®-treated or gold-plated inlet seals can help minimize active sites in the injection port. If tailing peaks and poor response for active compounds cannot be corrected by using properly deactivated inlet liners and treated inlet seals, other injection techniques such as cold on-column or temperature-programmed injections should be considered.

Molecular Weight Discrimination: In hot vaporization injections, one injection port temperature is used to vaporize all of the analytes in one sample injection. Compounds spanning a range of molecular weights and boiling points will exhibit differences in response for equal concentrations of analyte. High molecular weight, high boiling point analytes will have a noticeably reduced response when compared to lower molecular weight, lower boiling point analytes. This effect is more pronounced when analyzing samples that have a broad range of molecular weights and boiling points. Samples containing analytes that are more closely grouped by molecular weight and boiling point show less molecular weight discrimination.

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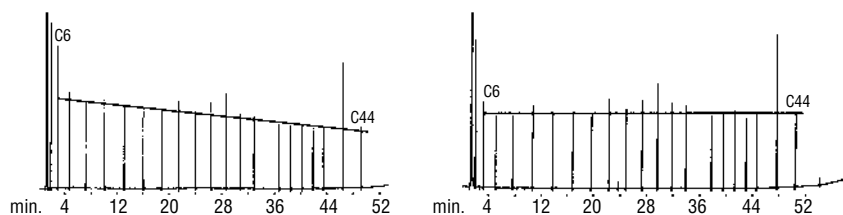
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Figure 11.*Splitter discrimination typical of split and splitless injections.*

Splitter discrimination is evident from relatively enhanced peak heights for the early-eluting compounds and diminished peak heights for the later-eluting higher molecular weight compounds. The same sample analyzed by cold on-column injection shows no discrimination; the peak heights for low and high molecular weight compounds are truly representative of this sample.



Discrimination typical of a split or splitless injector. Injector temperature: 340°C

30m, 0.32mm ID, 0.25µm Rt[®]-1 (cat.# 10124)
 Inj. volume: 0.2µL
 On-column conc.: 15ng.
 Oven temp.: 40°C to 340°C @ 5°C/min.

Cold on-column injection provides accurate information. Injector temperature: 40°C.

Det. (FID) temp.: 340°C
 Linear velocity: 50cm/sec., hydrogen
 Attenuation: 8x10⁻¹¹ AFS

Figure 11 demonstrates the molecular weight discrimination experienced when analyzing a series of hydrocarbons with a broad range of molecular weights (C6 through C44).

Alternative injection techniques, such as cold on-column injection, can be used to minimize molecular weight discrimination.

Molecular weight discrimination is usually very repeatable. In split and splitless injections, if the same injection port temperature, carrier gas pressure, sample size and sample solvent are used for every injection, sample vaporization should be a reproducible process. Any molecular weight discrimination experienced should be the same from one injection to the next. Because of this consistency, many analysts choose to ignore molecular weight discrimination unless it compromises overall sensitivity. To help compensate for differences in response due to molecular weight discrimination, multiple internal standards can be used to mimic the range of molecular weights and boiling points for the analytes in the sample.

Molecular weight discrimination can be minimized by choosing an injection port liner that ensures the sample is completely and uniformly vaporized. Inadequate vaporization causes the sample to approach the head of the column in both the aerosol and vapor states. Aerosol droplets, consisting predominantly of high molecular weight compounds, can be driven past the head of the column by the momentum of the carrier gas and will be preferentially swept out of the injection port and through the split vent. Injection port liners that are packed with glass wool or that incorporate a flow diverting device within their bore assist in vaporizing the sample and transferring a homogeneous representation to the head of the column.

Needle Discrimination: During sample injections, the syringe needle undergoes some degree of heating in the injection port. The temperature reached by the needle can influence the relative response for low and high molecular weight analytes. During the process of expelling the sample from the syringe, the contents in the needle are not completely transferred to the injection port. As the needle begins to heat, low molecular weight analytes begin to vaporize from the needle while higher molecular weight analytes remain inside the needle. Therefore, the lower molecular weight analytes will show enhanced response compared to higher weight analytes (Figure 12). Three techniques can be used to minimize needle discrimination in split and splitless injections.

The first technique is to inject the sample as rapidly as possible. Rapid injections minimize the amount of time the needle spends in the injection port and reduces the amount of heating the needle experiences. When making rapid injections in straight injection port liners for split or splitless analysis, the sample can be propelled beyond the inlet of the column and onto the injector base fitting. Always pack injection port liners with deactivated glass wool or CarboFrit[™] packing, or use a flow diverting device like a laminar cup to assist in sample

Figure 12.

Factors in discrimination: high molecular weight material clinging to the syringe needle and non-homogeneous vaporization of the sample in the inlet liner.

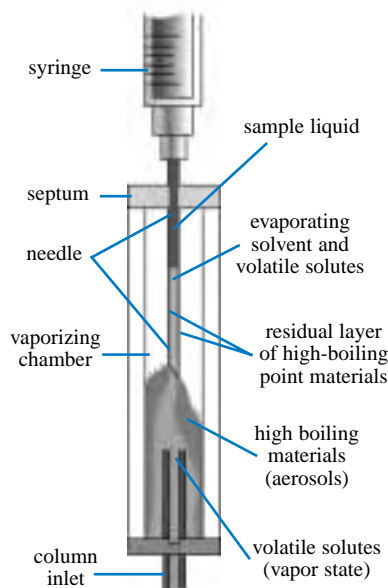
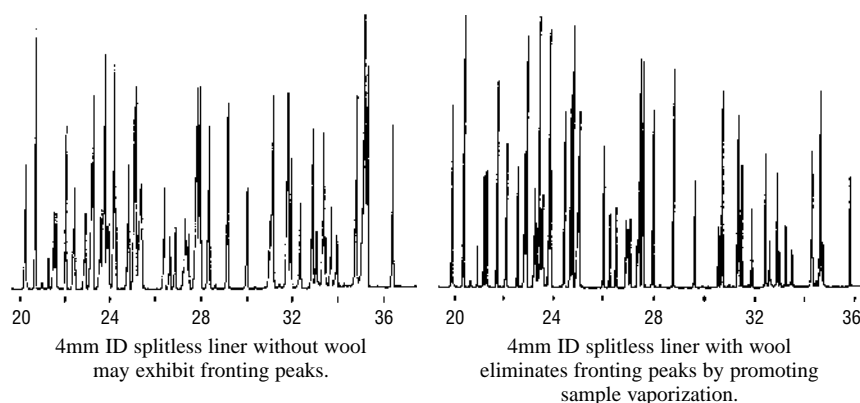


Figure 13.

Always pack splitless inlet liners with wool when using rapid injection autosamplers.



vaporization. Figure 13 shows the improvement in peak shape when an HP autosampler is used with an injection port liner packed with wool, versus a liner without wool.

The second technique is to use hot needle injection. Hot needle injections are performed by drawing the sample all the way into the syringe barrel, leaving the needle empty. When the needle is introduced into the injection port the injection is delayed for a short period of time (3–5 seconds, for example) to allow the needle to heat completely. Then the syringe plunger is depressed and the sample is expelled into the injection port liner.

The third technique is to use a solvent flush with each injection. This technique involves drawing a small amount of solvent into the syringe, followed by a small amount of air, followed by the desired amount of sample. All of the solvent, air, and sample are then drawn into the barrel of the syringe, just as in a hot needle injection. The needle is preheated, as in the hot needle injection, and the contents of the syringe are expelled into the injection port liner. The solvent that was first drawn into the syringe acts to flush the syringe barrel and needle, and completely transfers all of the sample during the injection process.

Backflash: Backflash occurs when the volume of the vaporized sample exceeds the volume inside the injection port liner. Most of the excess vaporized sample escapes out the top of the injection port liner. Some of it is swept down the septum purge line. Another portion of it can back up into the carrier gas supply line, and some of it can be re-introduced into the injection port. Backflash can cause poor peak area reproducibility, tailing peaks, split peaks, and poor resolution.

Table III (page 8) shows the estimated expansion volumes for 1 μ L injections of a variety of solvents. When using an injection port temperature of 250°C and a carrier gas pressure of 10psig, most solvents will vaporize and expand to a volume that exceeds the capacity of a 2mm ID injection port liner (approximately 240 μ L, see Table IV). In order to minimize backflash, injection port parameters must be carefully optimized. Injection port temperature, carrier gas pressure, sample size, and rate of injection all should be adjusted to ensure the vaporized sample remains inside the liner prior to being transferred to the head of the column.

Sample Size and Injection Port Temperature: As the equation in Table III shows, the volume of vaporized sample produced is directly related to the size of the liquid sample (n) and the temperature of the injection port (T). A decrease in either of these values will translate into a smaller vaporized sample volume. If the injection port temperature cannot be decreased because of vaporization problems and the sample size cannot be decreased because of sensitivity issues, backflash must be minimized by optimizing the rate of injection or by adjusting the carrier gas pressure.

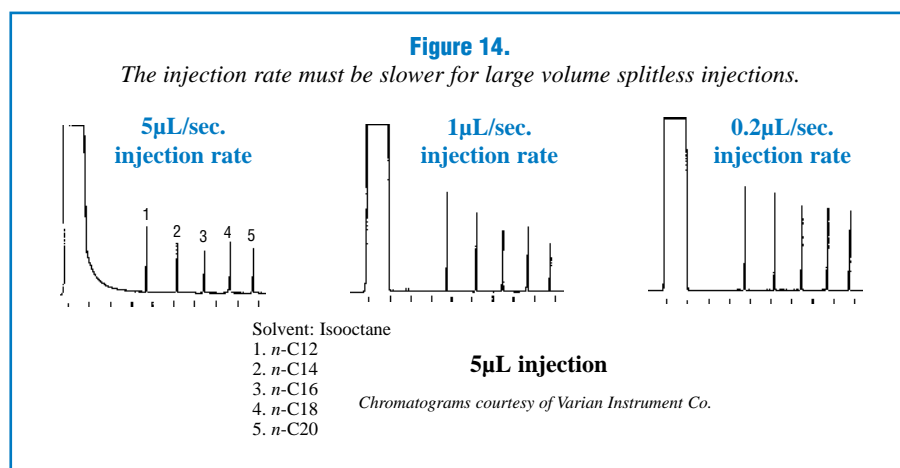
Table IV.
Liner Volumes.

	Theoretical*	Effective
1.0mm ID =	59 μ L	30 μ L
2.0mm ID =	236 μ L	118 μ L
3.0mm ID =	530 μ L	265 μ L
4.0mm ID =	942 μ L	471 μ L

**Liner volume actually available for vaporization with carrier gas present is $\leq 1/2$ theoretical, due to the presence of carrier gas in the liner.*

From Split and Splitless Injection in Capillary GC, 3rd Ed., K. Grob, Wiley-VCH, 2001.

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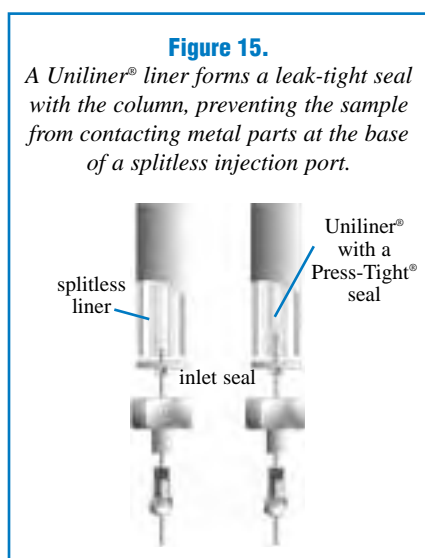
Optimizing the Rate of Injection: Figure 14 shows the effect of varying rates of injection for a 5µL sample. When a rapid injection (5µL/sec.) is made, the solvent peak tails and the responses for equal concentrations of each analyte are not reproducible. A 1µL/sec. injection rate improves the solvent peak shape, but the response for each analyte still is not proportional to the concentration of each analyte. Only when the injection rate is slowed to 0.2µL/sec. does the response for each analyte become consistent with the amount injected.

Some autosamplers are capable of slowing the injection rate to minimize backflash, but most autosamplers use a rapid injection sequence. If large-volume injections must be made rapidly, adjustments to the carrier gas pressure must be used to control sample expansion.

Pressure Programming: Pressure (P) is in the denominator of the equation in Table III (page 8). Any increase in carrier gas pressure will help to reduce sample expansion volume. Most of the latest models of GCs incorporate electronic pressure control (EPC) of the carrier gas pressure. Pressure can be time-programmed so that the carrier gas pressure initially is very high, then is reduced after the injection to optimize carrier gas flow rate for best resolution. Setting the initial carrier gas pressure to a high value will reduce the amount of sample expansion that occurs at the point of injection and will speed up the transfer of the vaporized sample from the liner to the head of the column.

Direct Injection as an Alternative to Splitless Injection

Direct injections are an alternative approach for injecting samples with low concentrations of analytes. Direct injections vaporize the entire sample in a heated injection port, just like split and splitless injections. However, in direct injections, there is only one flow path through the injection port. All of the carrier gas is directed into the column and, hence, the entire vaporized sample is directed into the column as well. This can be accomplished by using a specially designed injection port liner. Unliner® injection port liners have an internal taper in one end that allows a direct connection between the liner and the capillary column. With this connection, the flow path from the injection port body through the split vent is blocked and all of the carrier gas flow is directed into the capillary column. Figure 15 illustrates how a Unliner® injection port liner with a Press-Tight® seal forms a leak-free connection between the liner and the column.



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Because all of the carrier gas flow and the entire vaporized sample is directed into the capillary column, direct injections give comparable performance to splitless injections. Faster carrier gas flow rates usually are used to speed up the sample transfer process, and improve peak shapes and resolution. Direct injections can be used as another option to minimize molecular weight discrimination and loss of active compounds.

A Uniliner® inlet liner can be used as a direct replacement for a splitless liner. It can be installed in the same manner as a splitless liner, except that the system must be operated continuously with the solenoid valve closed. Uniliner® inlet liners are designed to accommodate 0.32 or 0.53mm ID columns. Request Restek's *Guide to Direct On-Column Vaporization Injection* (lit. cat.# 59882) for more information on how to perform and optimize direct injections.

Standard Gooseneck Uniliner® Inlet Liner



The buffer volume chamber contains the sample vaporization cloud and prevents analyte contact with metal injector parts. Peak tailing is reduced and larger injections can be made.

Cyclo-Uniliner® Inlet Liner



The glass cyclo spiral provides an excellent vaporization surface for high and low molecular weight samples. Particles are trapped on the first turn of the spiral, reducing subsequent residue/sample interaction. In comparison to liners packed with wool, Cyclo-Uniliner® liners accept up to five times as many injections of dirty samples before calibration curves degrade. Because they are deactivated, they are ideal for active samples.

Open-top Uniliner® Inlet Liner



Open-top Uniliner® liners are ideal for extremely dirty samples because they can be packed with fused silica wool to trap dirt and sample residue. Contaminated wool is easily replaced and the liner can be cleaned with a nylon brush or pipe cleaner.

Drilled Uniliner® Inlet Liner



A specially modified injection port liner, developed by Restek chemists, reduces sample contact with active metal parts in split/splitless injection ports. The Drilled Uniliner® liner gives the benefits of both direct injection and splitless injection. The column is connected to the liner by a press-fit connection, thus preventing the sample from contacting the metal at the bottom of the injection port. The hole on the side of the liner allows the purge flow to escape from the liner when the injection mode is switched from splitless to split.

Deactivation

Siltek™ Deactivation

- Revolutionary deactivation lowers endrin breakdown to less than 1%.
- Inertness retained over a wide range of sample pH.
- Minimal bleed.
- Recommended for difficult matrix and reactive compound analysis.
- Ideal for chlorinated pesticide analysis.
- Recommended for use with Rtx®-CLPesticides, Stx-CLPesticides, Stx-IHT, and Rtx®-TNT columns.

Base-Deactivation

- Provides excellent inertness for basic compounds.
- Recommended for use with Rtx®-5 Amine, Rtx®-35 Amine, and Stabilwax®-DB columns.

Intermediate Polarity (IP) Deactivation

Our standard deactivation for liners. Phenylmethyl-deactivated surface provides optimum compatibility for both polar and non-polar compounds.

In most cases, the standard IP deactivation should be chosen. The IP surface contains methyl groups, as well as phenyl groups, making this surface compatible with most common solvents.

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Guard Columns

Guard columns protect analytical columns in several ways:

Guard columns trap non-volatile residues, preventing them from collecting at the analytical column inlet. These residues may be very high molecular weight organic compounds, inorganic salts, or particles. If these contaminants enter the analytical column, they can cause adsorption of active compounds, loss of resolution, and poor peak symmetry. When this contamination begins to affect sample analysis, a small section of the analytical column must be removed to restore proper performance. Each time a column section is removed, retention times change, and some resolution is lost. By using a guard column and removing contaminated loops from it instead of from the analytical column, analytical column length and inertness remain intact.

Guard columns also allow more injections to be made before contamination interferes with analytical results. Because there is no stationary phase coated on a guard column, the amount of time the sample spends in the guard column is minimal. This reduces the interaction between sample components and contamination from non-volatile residue in the guard column.

For more information on selecting a guard column for your analysis, request our *Fast Facts* GC Capillary Column Guard Columns (lit. cat.# 59319).

Mini Wool Puller/Inserter

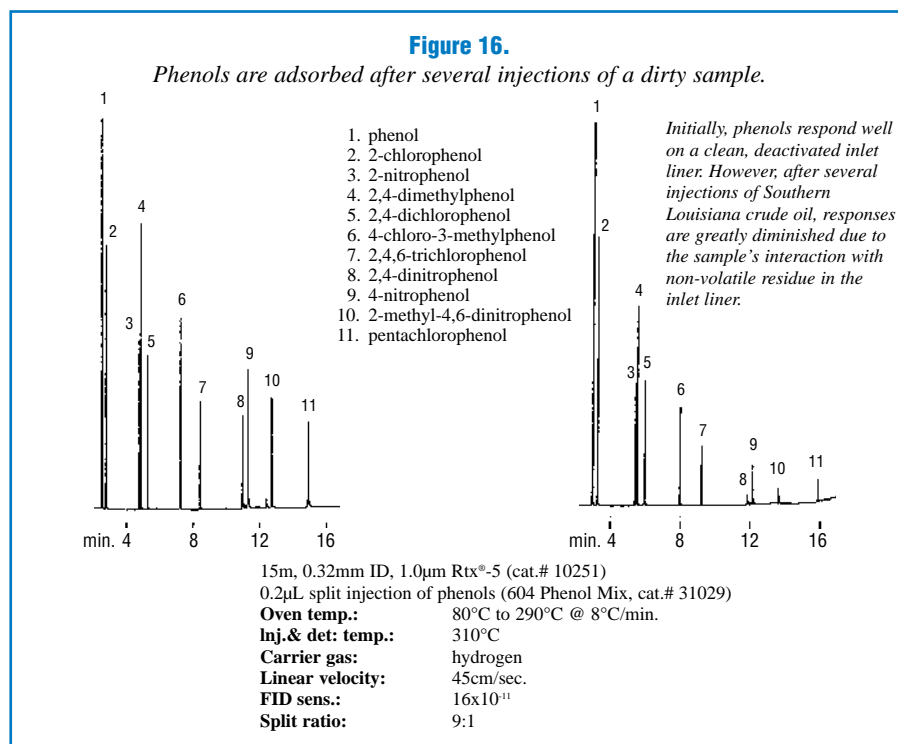
Makes inserting and removing wool easy. Not recommended for double gooseneck liners.



Description	qty.	cat.#
Mini Wool Puller/Inserter	2-pk.	20114

Hints for Analyzing Dirty Samples

When injecting dirty samples, non-volatile contaminants such as high molecular weight compounds, septum particles, derivatization reagents, salts, and pyrolyzed samples adhere to the interior wall of the injection port liner after the sample solvent and sample analytes have been vaporized. As this layer of residue thickens, it can cause loss of response for active compounds. Figure 16 illustrates this effect when highly active phenols are analyzed on a clean and a dirty inlet liner. In this example, responses are reduced because of adsorptive effects in the liner.



Non-volatile contamination can be trapped in the injection port liner by using a small plug of deactivated fused silica or glass wool. Usually a 1cm plug of wool, positioned in the center of the injection port liner, is sufficient to provide a surface for non-volatile contamination to collect. Some instrument manufacturers provide specific instructions on packing injection port liners to maximize quantitative accuracy and minimize discrimination. If fused silica wool or glass wool is used in an injection port liner, it should be replaced as part of the routine maintenance schedule for the injection port. Regular replacement of the wool in the injection port liner will extend the lifetime of the injection port liner as well as prevent chromatographic problems from extensive non-volatile contaminant build up. When replacing the wool during routine maintenance, minimize handling of the wool by using a wool puller tool (cat.# 20114).

If fused silica or glass wool is not an effective mode of trapping non-volatile contamination under your conditions, injection port liners with a "cyclo" or glass frit design can be used to trap non-volatile contamination. While these types of injection port liners may provide effective trapping of non-volatile contamination, they are harder to clean than straight injection port liners packed with wool.

In the past, some instruments were supplied with injection port liners that were packed with a small amount of packed column packing material. We do not recommend using this type of injection port liner. Diatomites used in packed column GC packings often are active and contain impurities that increase adsorptive effects for active compounds. Also, the stationary phases that are used in these packings can produce significant bleed when used in injection ports at elevated temperatures.

In addition to using a clean and deactivated injection port liner, we recommend using a five-meter deactivated guard column when analyzing dirty samples. Routine maintenance of the liner and the guard column will prevent dirty samples from contaminating the analytical column, and will help ensure reproducible and accurate analytical results.

Hints for Performing Routine Injection Port Maintenance

Injection port maintenance should be performed prior to installing any capillary column. Maintenance of the injection port after a column is installed should be performed periodically, based on the number of injections made and the cleanliness of the samples. Maintenance includes cleaning, deactivating, or replacing injection port liners, and replacing critical inlet seals and the septum. Review the instrument manual inlet diagram prior to disassembling the inlet.

Cleaning and Deactivating Injector Liners

For optimum column performance, the injection port liner must be free of septum particles, sample residue, and ferrule fragments. Use a deactivated injection port liner when analyzing samples with compounds that are active or prone to decomposition or adsorption on untreated glass surfaces. Table V illustrates the importance of a deactivated injection port liner when analyzing active compounds. The response factors (RF) for all three of these active compounds were much lower with non-deactivated inlet liners.

Table V.

Deactivated inlet liners show higher response factors for active components.

Compound	RF Deactivated Liner	RF Undeactivated Liner	RF relative to naphthalene; N=3
2,4-dinitrophenol	0.248	0.185	
pentachlorophenol	0.240	0.188	
benzidine	0.327	0.234	

If the injection port liner is deactivated and is not excessively dirty, cleaning with organic solvents usually is enough to restore original performance. Most organic solvents will not affect the integrity of the surface deactivation. First, remove septum particles that adhere to the inside wall of the injection port liner by rinsing with methanol or isopropanol. Next, use pentane, methylene chloride or toluene to remove sample residue. Do not use laboratory detergents, acids, or bases to clean injection port liners. Harsh cleaning agents will remove or damage the deactivation layer and the liner will require re-deactivation. Nylon brushes and pipe cleaners (cat.# 20108) can be used for mild abrasive cleaning of injection port liners.

Replacing Critical Seals

Replace critical seals prior to installing an injection port liner (see the instrument manual for seal locations). In most capillary injection ports, an o-ring or ferrule made of rubber or graphite is used to seal the injection port liner into the injection port body. It is critical that the seal fits tightly around the liner, to prevent the carrier gas from leaking around the outside of the liner. Check for leaks with a thermal conductivity-type leak detector (e.g., Leak Detective™ II, cat.# 20413).

Changing Septa

Always use a high-quality, low-bleed septum. We recommend replacing the septum frequently, to prevent leaks and fragmentation. Multiple injections and continuous exposure to hot injection port surfaces will decompose the septum and cause particles to fall into the injection port liner. Septum particles are a potential source of ghost peaks, loss of inertness, and carrier gas flow occlusion. It is best to install a new septum at the end of an analytical sequence so that it can condition in the injector and reduce the incidence of ghost peaks. To avoid contamination, always use forceps when handling septa. Restek's high quality, low-bleed Thermolite® septa are available for most common models of capillary GCs. For more information, request a copy of Restek's *Guide to Minimizing Septa Problems* (lit. cat.# 59886).

For additional hints for analyzing dirty samples, request a copy of Restek's *A Guide When Injecting Dirty Samples* (lit. cat.# 59881).

Nylon Tube Brushes and Pipe Cleaner

Use to remove small septum fragments and residue from dirty glass inlet liners. Brushes are 1/8-, 3/16-, and 1/4-inch in diameter; pipe cleaner is one foot long.



Description	qty.	cat.#
Nylon Tube Brushes and Pipe Cleaner	set	20108

Leak Detective™ II Leak Detector

- Affordable thermal conductivity leak detector—every analyst can have one.*
- Compact, ergonomic design is easy to hold and operate with one hand.
- Helium, hydrogen, and nitrogen can be detected at 1×10^{-4} cc/sec. or at an absolute concentration as low as 100ppm.**
- Fast results—responds in less than 2 seconds to trace leaks of gases with thermal conductivities different than air.
- Micro-chip design improves sensitivity and response time over previous models.
- Auto zeroing with the touch of a button.
- Battery-operated for increased portability (one 9-volt).



Description	qty.	cat.#
Leak Detective™ II Leak Detector (9 volt, Battery-Operated)	ea.	20413

*Never use liquid leak detectors on a capillary system because liquids can be drawn into the column.

**Caution: NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possibly hazardous conditions.

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Siltek™ Deactivation—The Next Generation

- Maximizes the inertness of the sample pathway.
- Minimizes breakdown.
- Low bleed.
- Thermally stable.
- “Clean and green”—manufactured without the use of harmful organic solvents.

Restek offers the next generation of deactivation. The Siltek™ deactivation process (patent pending) produces a highly-inert glass surface, which features high temperature stability, extreme durability, and low bleed. Try Siltek™ liners, guard columns, wool, and connectors for better recovery of sample analytes.

For Siltek™ inlet liners, add the corresponding suffix number to your liner catalog number.

qty.	Siltek™	Siltek™ with Siltek™ wool	Siltek™ with CarboFrit™
each	-214.1 addl. cost	-213.1 addl. cost	-216.1 addl. cost
5-pk.	-214.5 addl. cost	-213.5 addl. cost	-216.5 addl. cost
25-pk.	-214.25 addl. cost	-213.25 addl. cost	-216.25 addl. cost

Deactivation—Which Should You Choose?

Siltek™ Deactivation

- Revolutionary deactivation lowers endrin breakdown to less than 1%.
- Inertness retained over a wide range of sample pH.
- Minimal bleed.
- Recommended for difficult matrix and reactive compound analysis.
- Ideal for chlorinated pesticide analysis.
- Recommended for use with Rtx®-CLPesticides, Stx-CLPesticides, Stx-IHT, and Rtx®-TNT columns.

Base-Deactivation

- Provides excellent inertness for basic compounds.
- Recommended for use with Rtx®-5 Amine, Rtx®-35 Amine, and Stabilwax®-DB columns.

Base-Deactivated Inlet Liners for Agilent GCs

If you do not see the deactivated liner you need, you can order it on a custom basis by adding the appropriate suffix number to the liner catalog number. For base deactivation: each (-210.1), 5-pack (-210.5), 25-pack (-210.25). For base-deactivated liners packed with base-deactivated wool: each (-211.1), 5-pack (-211.5), 25-pack (-211.25).

ea.	5-pk.	25-pk.
4mm Split Straight w/ Wool		
20781-211.1	20782-211.5	20783-211.25
Cycloplitter®		
20706-210.1	20707-210.5	—
4mm Splitless Straight		
20772-210.1	20773-210.5	20774-210.25
2mm Gooseneck		
20795-210.1	20796-210.5	20797-210.25
4mm Gooseneck		
20798-210.1	20799-210.5	20800-210.25

Prepacked Liners

Let Restek do the work! Just add the appropriate suffix to the liner catalog number.

Prepacked Inlet Liners Suffix Numbers				
qty.	FS Wool	FS Beads	Glass Wool	CarboFrit™†
ea.	-200.1	-201.1	-202.1	-209.1
5-pk.	-200.5	-201.5	-202.5	-209.5
25-pk.	-200.25	-201.25	-202.25	-209.25

†CarboFrit™ inserts require a neck greater than 2mm.

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(814-353-1300, ext. 3)






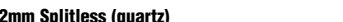





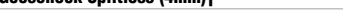







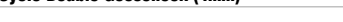

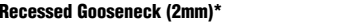

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all liners are
100%
deactivated

Liners for Agilent/Finnigan GCs

C O L U M N I N S T A L L S T H I S E N D

Splitless Liners for Agilent/Finnigan GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to Agilent part #	ea.	cat.# 5-pk.	25-pk.
	2mm Splitless	trace samples <2µL	2.0 ID 6.5 OD x 78.5	18740-80220 5181-8818	20712	20713	20714
	4mm Splitless	trace samples >2µL	4.0 ID 6.5 OD x 78.5	19251-60540	20772	20773	20774
	Siltek™ 4mm Splitless	trace samples >2µL	4.0 ID 6.5 OD x 78.5	19251-60540	20772-214.1	20773-214.5	20774-214.25
	4mm Splitless w/ FS Wool	trace samples >2µL	4.0 ID 6.5 OD x 78.5	19251-60540	22400	22401	22402
	2mm Splitless (quartz)	trace samples <2µL	2.0 ID 6.5 OD x 78.5	18740-80220 5181-8818	20914	20915	-
	4mm Splitless (quartz)	trace samples >2µL	4.0 ID 6.5 OD x 78.5	18740-80220 5181-8818	20912	20913	-
	4mm Splitless (quartz) w/ FS Wool	trace samples >2µL	4.0 ID 6.5 OD x 78.5	18740-80220 5181-8818	22403	22404	-
	Gooseneck Splitless (2mm)	trace samples <2µL	2.0 ID 6.5 OD x 78.5	5181-3316***	20795	20796	20797
	Siltek™ Gooseneck Splitless (2mm)	trace samples <2µL	2.0 ID 6.5 OD x 78.5	5181-3316***	20795-214.1	20796-214.5	20797-214.25
	Gooseneck Splitless (4mm)†	trace samples >2µL	4.0 ID 6.5 OD x 78.5	5181-3316	20798	20799	20800
	Siltek™ Gooseneck Splitless (4mm)†	trace samples >2µL	4.0 ID 6.5 OD x 78.5	5181-3316	20798-214.1	20799-214.5	20800-214.25
	Gooseneck Splitless (4mm) w/ FS Wool†	trace samples >2µL	4.0 ID 6.5 OD x 78.5	5062-3587	22405	22406	22407
	Siltek™ Gooseneck Splitless (4mm) w/ Siltek™ Glass Wool†	trace samples >2µL	4.0 ID 6.5 OD x 78.5	5062-3587	22405-213.1	22406-213.5	22407-213.25
	Double Gooseneck Splitless (4mm)	trace, active samples >2µL	4.0 ID 6.5 OD x 78.5	5181-3315	20784	20785	20786
	Siltek™ Double Gooseneck Splitless (4mm)	trace, active samples >2µL	4.0 ID 6.5 OD x 78.5	5181-3315	20784-214.1	20785-214.5	20786-214.25
	Cyclo Double Gooseneck (2mm)	trace, active, dirty samples <2µL	2.0 ID 6.5 OD x 78.5	-	20907	20908	-
	Cyclo Double Gooseneck (4mm)	trace, active, dirty samples >2µL	4.0 ID 6.5 OD x 78.5	-	20895	20896	20997
	Siltek™ Cyclo Double Gooseneck (4mm)	trace, active, dirty samples >2µL	4.0 ID 6.5 OD x 78.5	-	20895-214.1	20896-214.5	20997-214.25
	Recessed Gooseneck (2mm)*	base easily packs with wool for dirty samples <2µL	2.0 ID 6.5 OD x 78.5	-	20980	20981	20982
	Recessed Gooseneck (4mm)*	base easily packs with wool for dirty samples >2µL	4.0 ID 6.5 OD x 78.5	-	20983	20984	20985
	Siltek™ Recessed Gooseneck (4mm)*	base easily packs with wool for dirty samples >2µL	4.0 ID 6.5 OD x 78.5	-	20983-214.1	20984-214.5	20985-214.25
	Recessed Gooseneck (4mm)* w/ FS Wool	base easily packs with wool for dirty samples > 2µL	4.0 ID 6.5 OD x 78.5	-	22408	22409	22410
	Recessed Double Gooseneck (4mm)*	base easily packs with wool for dirty, active samples > 2µL	4.0 ID 6.5 OD x 78.5	-	20986	20987	20988

*Use with two-hole ferrule for dual-column analysis.

**Nominal ID at syringe needle expulsion point.

***Restek design changes improve performance over the original Agilent liner.

†Use this liner for increased sensitivity.

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Liners for Agilent/Finnigan GCs

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Split Liners for Agilent/Finnigan GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to Agilent part#	ea.	cat.# 5-pk.	25-pk.
	1mm Split†	for purge & trap inlet splitting or sample <1µL	1.0 ID 6.3 OD x 78.5	-	20972	20973	-
	4mm Split with Wool*	universal, use with Agilent 7673 autosampler	4.0 ID 6.3 OD x 78.5	19251-60540	20781	20782	20783
	Siltek™ 4mm Split w/ Siltek™ Glass Wool	featuring Siltek™ deactivation universal, use with Agilent 7673 autosampler	4.0 ID 6.3 OD x 78.5	19251-60540	20781-213.1	20782-213.5	20783-213.25
	Laminar Cup Splitter	high MW compounds	4.0 ID 6.3 OD x 78.5	18740-80190	20801	20802	-
	mini-Lam™ Split	high MW compounds	4.0 ID 6.3 OD x 78.5	-	20990	20991	-
	Cup Splitter	high & low MW compounds	4.0 ID 6.3 OD x 78.5	18740-80190	20709	20710	-
	Siltek™ Cup Splitter	featuring Siltek™ deactivation high & low MW compounds	4.0 ID 6.3 OD x 78.5	18740-80190	20709-214.1	20710-214.5	-
	Cyclosplitter®	dirty samples, many injections before cleaning required	4.0 ID 6.3 OD x 78.5	-	20706	20707	20708
	4mm Split Precision Liner	dirty samples, trace samples	4.0 ID 6.3 OD x 78.5	-	21022	21023	20979
	Siltek™ 4mm Split Precision Liner w/ Siltek™ Glass Wool	featuring Siltek™ deactivation dirty samples, trace samples	4.0 ID 6.3 OD x 78.5	-	21022-213.1	21023-213.5	20979-213.25
Split/Splitless Liners for Agilent 6890 GCs		Benefits/Uses	ID**/OD & Length (mm)	Similar to Agilent part #	ea.	cat.# 5-pk.	cat.# 5-pk.
	Low Pressure Drop Liner w/ Wool	universal, use with Agilent 6890 GCs	4.0 ID 6.3 OD x 78.5	5183-4647	21032	21033	21033
DI Liners for Agilent/Finnigan GCs (For 0.32/0.53mm ID Columns)		Benefits/Uses:	ID**/OD & Length (mm)	cat.# ea.	cat.# 5-pk.		
	Siltek™ 1mm Uniliner***	featuring Siltek™ deactivation trace, active samples, samples <1µL	1.0 ID 6.3 OD x 78.5	21052-214.1	21053-214.5		
	Uniliner***	trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	20335	20336		
	Siltek™ Uniliner***	featuring Siltek™ deactivation trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	20335-214.1	20336-214.5		
	Cyclo-Uniliner***	trace, dirty, high MW active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	20337	20338		
	Siltek™ Cyclo-Uniliner***	featuring Siltek™ deactivation trace, dirty, high MW active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	20337-214.1	20338-214.5		
	Open-top Uniliner® with Wool***	trace, dirty, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	20843	20844		
DI Liners for Agilent 5890 & 6890 GCs (For 0.25/0.32/0.53mm ID Columns)		Benefits/Uses:	ID**/OD & Length (mm)	cat.# ea.	cat.# 5-pk.		
	Drilled Uniliner®	allows direct injection when using an EPC-equipped GC	4.0 ID 6.3 OD x 78.5	21054	21055		
	Siltek™ Drilled Uniliner®	featuring Siltek™ deactivation allows direct injection when using an EPC-equipped GC	4.0 ID 6.3 OD x 78.5	21054-214.1	21055-214.5		
	Siltek™ 1mm Drilled Uniliner®	featuring Siltek™ deactivation allows direct injection when using an EPC-equipped GC	1.0 ID 6.3 OD x 78.5	21390-214.1	21391-214.5		

*Use with two-hole ferrule for dual-column analysis.

**Nominal ID at syringe needle expulsion point.

***Restek design changes improve performance over
the original Agilent liner.

†Use this liner for increased sensitivity.

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ECHnology Pty Ltd

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O-Rings

Viton® O-Rings

- For Agilent and PE AutoSys GCs.
- Viton® O-rings fit split (6.3mm OD) or splitless (6.5mm OD) liners.
- Graphite O-rings have excellent thermal stability.

Description	Max. temp.	Similar to Agilent part #	qty.	Restek cat.#
Viton® (fluorocarbon) O-rings	350°C	5180-4182	25-pk.	20377



Graphite O-Rings

- For Agilent and Varian 1177 GCs.
- Excellent thermal stability at injection port temperature up to 450°C!

Description	Max. temp.	Similar to Agilent part #	Restek cat.#	
			10-pk.	50-pk.
6.35mm ID Graphite O-rings for split liners	450°C	5180-4168	20296	20297
6.5mm ID Graphite O-rings for splitless liners	450°C	5180-4173	20298	20299



High-Temperature O-Rings

- Stable to 400°C.
- Will not crack or melt.
- Softer and easier to use than graphite.

Description	Max. temp.	qty.	cat.#
High-temperature O-rings	400°C	5-pk.	20437



Inlet and FID Maintenance Kits for Agilent GCs

- Kits include the most common consumable supplies.
- All parts meet or exceed instrument manufacturer's specifications.
- Includes parts list that makes reordering easy.

Inlet kits include:

- 0.4, 0.5, and 0.8mm ID graphite ferrules.
- Viton® o-rings.
- Capillary nuts.
- Inlet seals.
- Reducing nut.
- Scoring wafer.
- 11mm Thermolite® septa.
- 4.0mm single gooseneck liner.
- 4.0mm split liner with wool.
- Capillary column caps.
- 1/4- to 5/16-inch wrench.
- Septum puller.
- Installation gauge.
- Wire cleaning brush.
- Jet reamers/ferrule removers.
- Inlet liner removal tool.

FID kits include:

- 1/4-Inch, 0.4, 0.5, and 0.8mm ID graphite
- FID/NPD capillary adaptor.
- Capillary nuts.
- Jet reamers/ferrule removers.
- 1/4-Inch nut.
- Scoring wafer.
- Capillary column caps.
- Ignitor for either Agilent 5890 or 6890/6
- FID flow measuring adaptor.
- 1/4- to 5/16-inch wrench.
- Installation gauge.
- Wire cleaning brush.
- High-performance Silcosteel®-treated FID jet for either Agilent 5890 or 6890/6850 GCs.
- 1/4-Inch nut driver for jet removal.



Description	qty.	cat.#
Inlet Maintenance Kit for Agilent 5890/6890/6850 GCs	kit	21069
FID Maintenance Kit for Agilent 5890 GCs	kit	21070
FID Maintenance Kit for Agilent 6890/6850 GCs	kit	21071

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Vespel® Ring Inlet Seals for Agilent 5890/6890 and 6850 GCs

- Easy-to-use, patent-pending design makes a better seal, easily.
- Prevents oxygen from damaging your columns.
- Reduces wear on the injection port body.

In Agilent split/splitless injection ports, it can be difficult to make and maintain a good seal with a conventional metal inlet disk. The metal-to-metal seal dictates that the analyst apply considerable torque to the reducing nut, and, based on our testing, this does not ensure a leak-tight seal. Over the course of oven temperature cycling, metal seals are prone to leaks, which ultimately can degrade the capillary column, and cause other analytical difficulties.

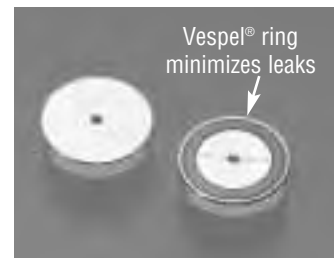
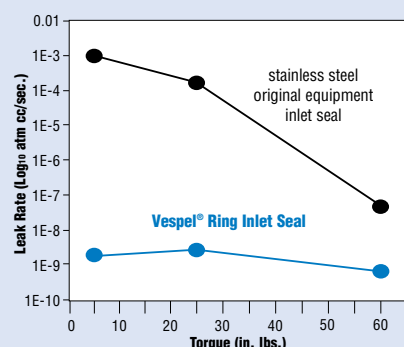


Figure 1

The Vespel® Ring Inlet Seal achieves leak-tight seals even at low torque, reducing the chance of leaks.



Our Vespel® Ring Inlet Seal greatly improves injection port performance—it seals even after repeated temperature cycles and without retightening the reducing nut! This seal features a Vespel® ring embedded into its face. This soft Vespel® ring will not harm the critical seal on the injector body, and is outside the sample flow path. Tests using a high sensitivity helium leak detector indicate the Vespel® Ring Inlet Seal seals equally effectively at torques of 5lb. or 60lb. (Figure 1).

Why trust a metal-to-metal seal when you can make leak-tight seals quickly and easily—and more reliably—with the Restek Vespel® Ring Inlet Seal? Use the stainless steel seal for analysis of unreactive compounds. To reduce breakdown and adsorption of active compounds, use the gold-plated or Silcosteel®-treated seals. The gold surface offers better inertness than standard stainless steel; Silcosteel® treatment provides inertness similar to that of fused silica capillary columns.

Vespel® Ring Inlet Seals for Agilent 5890/6890/6850 GCs

0.8mm ID Vespel® Ring Inlet Seal (washers included)	2-pk.	10-pk.
Gold-Plated	21562	21563
Silcosteel®	21564	21565
Stainless Steel	21560	21561
1.2mm ID Vespel® Ring Inlet Seal (washers included)	2-pk.	10-pk.
Gold-Plated	21568	21569
Silcosteel®	21570	21571
Stainless Steel	21566	21567



Achieve a better seal!

Re-Threading Tool

- Repair worn or damaged threads.
- Multiple uses (injection ports, fittings, etc.).
- Built-in guide to prevent cross-threading.



- 1) Worn & damaged threads can allow oxygen into the system—compromising analytical results and destroying columns.
- 2) Screw the tool completely onto the injection port in a clockwise direction.
- 3) Unscrew the tool and inspect the threads, repeat as necessary, and, when done, wipe threads with methanol to remove any debris.

Description	qty.	cat.#
Re-threading Tool for 1/8" compression fitting for Agilent split/splitless injection ports	ea.	23018

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Replacement Inlet Seals

- Special grade of stainless steel that is softer and deforms more easily, ensuring a completely leak-free seal.
- Increases column lifetime because oxygen cannot permeate into the carrier gas.
- Reduced noise benefits high-sensitivity detectors (e.g., ECDs, MSDs).
- Silcosteel® seal offers the inertness of glass.
- All seals include washers.

Replacement Inlet Seals for Agilent 5890/6890/6850 Split/Splitless Injection Ports

The inlet seal at the base of the Agilent 5890/6890 GC injection port contacts the sample and must be changed frequently to prevent adsorption of active compounds. In addition, septum fragments and sample residue accumulate on the disk surface, requiring disk replacement.

The inlet seal design increases column lifetime because oxygen cannot permeate into the carrier gas. Detector noise also is reduced with high-sensitivity detectors (e.g., ECDs or MSDs). To reduce breakdown and adsorption of active compounds, use the gold-plated or Silcosteel® seals. The gold surface offers better inertness than standard stainless steel, and the Silcosteel® treatment offers inertness similar to that of fused silica capillary columns.

Single-Column Installation, Opening Size 0.8mm ID*		0.25/0.32mm ID Dual-Column Installation, Opening Size 1.2mm ID		0.53mm ID Dual-Column Installation Opening Size (1/16-inch hole)	
2-pk.	10-pk.	2-pk.	10-pk.	2-pk.	10-pk.
Stainless Steel Inlet Seal					
21315	21316	20390	20391	20392	20393
Gold-Plated Inlet Seal					
21317	21318	21305	21306	—	—
Silcosteel® Inlet Seal					
21319	21320	21307	21308	—	—

*0.8mm ID stainless steel inlet seal is equivalent to Agilent part #18740-20880,
0.8mm ID gold-plated inlet seal is equivalent to Agilent part #18740-20885.

Replacement Inlet Cross-Disk Seal for Agilent GCs

- Ideal for high-flow split applications on Agilent 5890 GCs.
- All seals include washers.

(Similar to Agilent part # 5182-9652.)




















0.8mm ID Cross-Disk Inlet Seal for Agilent GCs		2-pk.	10-pk.
Gold-Plated		20477	20476
Silcosteel®		20475	20474
1.2mm ID Cross-Disk Inlet Seal for Agilent GCs		2-pk.	10-pk.
Gold-Plated		21009	21010
Silcosteel®		21011	21012



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all liners are 100% deactivated Liners for Varian GCs

C O L U M N I N S T A L L S T H I S E N D

Splitless Liners for Varian 1075/1077 GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to Varian part #	ea.	cat.# 5-pk.	25-pk.
	2mm Splitless	trace samples <2µL	2.0 ID 6.3 OD x 74	01-900109-05	20721	20722	20723
	4mm Splitless	trace samples >2µL	4.0 ID 6.3 OD x 74	01-900109-05	20904	20905	20906
	Double Gooseneck	trace, active samples up to 4µL	4.0 ID 6.3 OD x 74	-	20847	20848	20849
	Cyclo Double Gooseneck	trace, dirty, active samples up to 4µL	4.0 ID 6.3 OD x 74	-	20897	20898	-
Split Liners for 1075/1077 Varian GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to Varian part #	ea.	cat.# 5-pk.	25-pk.
	1mm Split	purge & trap inlet splitting or samples <1µL	1.0 ID 6.3 OD x 72	-	20970	20971	-
	Splitter with Wool*	universal, use with rapid autosamplers	4.0 ID 6.3 OD x 72	01-900109-01	20792	20793	20794
	Laminar Cup Splitter	high MW compounds	4.0 ID 6.3 OD x 72	01-900109-02	20803	20804	-
	Cup Splitter	high & low MW compounds	4.0 ID 6.3 OD x 72	-	20724	20725	-
	Cyclosplitter®	dirty samples, many injections before cleaning required	4.0 ID 6.3 OD x 72	-	20727	20728	-
	Frit Splitter	dirty samples, non-active compounds	4.0 ID 6.3 OD x 72	01-900109-03	20715	20716	20717
	Baffle Splitter	close boiling compounds	4.0 ID 6.3 OD x 72	01-900109-04	20718	20719	20720
	Split Precision™ Liner	dirty samples, active samples	4.0 ID 6.3 OD x 72	-	21030	21031	-
DI Liners for Varian 1075/1077 GCs (0.32/0.53mm ID)		Benefits/Uses:	ID**/OD & Length (mm)	Similar to Varian part #	ea.	cat.# 5-pk.	25-pk.
	Uniliner®	trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 72	-	20345	20346	-
	Cyclo-Uniliner®	trace, dirty, high MW, active samples, linearity	4.0 ID 6.3 OD x 72	-	20347	20348	-
	Open-top Uniliner® with Wool*	trace, dirty, active samples, high recovery & linearity	4.0 ID 6.3 OD x 72	-	20845	20846	-
SPI Liners for Varian GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to Varian part #	ea.	cat.# 5-pk.	25-pk.
	0.5mm SPI	high linearity for 0.25 & 0.32mm ID columns	0.53 ID 4.6 OD x 54	01-900109-06	20775	20776	20777
	Siltek™ 0.5mm SPI	high linearity for 0.25 & 0.32mm ID columns	0.53 ID 4.6 OD x 54	01-900109-06	20775-214.1	20776-214.5	20777-214.25
	0.8mm SPI	high linearity for 0.53mm ID columns	0.80 ID 4.6 OD x 54	01-900109-07	20778	20779	20780
	SPI with Buffer	dirty samples >1µL, fits 0.25, 0.32 & 0.53mm ID columns	2.4 ID 4.6 OD x 54	01-900109-08	20850	20851	20852

*Prepacked with fused silica wool. For glass wool instead, add the suffix "-202" to the liner catalog number.












**Nominal ID at syringe needle expulsion point.

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all liners are
100%
deactivated

Liners for Varian GCs

COLUMN INSTALLS THIS END

Liners for Varian 1177 GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to Varian part #	ea.	cat.# 5-pk.	25-pk.
	4mm Split	universal	4.0 ID 6.3 OD x 78.5	39-26119-36	21045	21046	-
	2mm Splitless w/wool*	trace samples <2µL	2.0 ID 6.5 OD x 78.5	39-26119-38	-	21077	-
	4mm Split w/wool*	universal	4.0 ID 6.3 OD x 78.5	39-26119-37	-	21079	-
1078/1079 Liners for Varian GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to Varian part #	ea.	cat.# 5-pk.	25-pk.
	1078/1079 Split	dirty samples, non-active compounds	3.4 ID 5.0 OD x 54	03-918464-00	21708	21709	-
	1078/1079 Splitless	trace samples <2µL	2.0 ID 5.0 OD x 54	03-918466-00	21711	21712	-
	Siltek™ 1078/1079 Splitless	trace samples <2µL	2.0 ID 5.0 OD x 54	03-918466-00	21711-214.1	21712-214.5	-
	Open 0.5mm ID	trace samples <1µL	0.5 ID 5.0 OD x 54	03-925331-00	20992	20993	-
	1078/1079 Split-No Frit	active samples	3.4 ID 5.0 OD x 54	03-918464-00	20859	20901	20909
	Siltek™ 1078/1079 Split-No Frit	active samples	3.4 ID 5.0 OD x 54	03-918464-00	20859-214.1	20901-214.5	20909-214.25
	Open 0.75mm ID	trace, low volume samples	0.75 ID 5.0 OD x 54	03-925330-00	21714	21715	21716
	1078/1079 Split Precision™ Liner	trace samples, dirty samples	3.4 ID 5.0 OD x 54	-	21024	21025	-

*Prepacked with fused silica wool. For glass wool instead, add the suffix "-202" to the liner catalog number.

**Nominal ID at syringe needle expulsion point.

Inlet Liner Seals for Varian 1177 Injectors

Meets original equipment specifications.

(Similar to Varian part # 39-26119-40.)

Description	qty.	cat.#
Inlet Liner Seals for Varian 1177 Injectors	10-pk.	20298

5mm Liner Seals for Varian 1078/1079 GCs

Description	qty.	cat.#
5mm Liner Seals for Varian 1078/1079 GCs	10-pk.	22683

Inlet Liner Removal Tool

- Easily remove liners from injectors.
- Made from high-temperature silicone.
- Won't chip or crack the liner.

Description	qty.	cat.#
Inlet Liner Removal Tool	3-pk.	20181























No more burned fingers!

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all liners are
100%
deactivated

Liners for PerkinElmer GCs

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Split Liners for PerkinElmer GCs	Benefits/Uses:	ID**/OD & Length (mm)	Similar to PE part #	ea.	cat.# 5-pk.	25-pk.
 Baffle Splitter	universal, for most common analyses	3.5 ID 5.0 OD x 100	0330-5181	20736	20737	-
 Cup Splitter	high & low MW compounds	3.5 ID 5.0 OD x 100	-	20739	20740	-
 Cyclo splitter®	dirty samples, max. injections before cleaning required	3.5 ID 5.0 OD x 100	-	20745	20746	-
 Laminar Cup Splitter	high MW compounds	3.5 ID 5.0 OD x 100	-	20805	20806	-
 Auto SYS Splitter with Wool*	universal for most common analyses	4.0 ID 6.2 OD x 92.1	N6101052	20832	20833	20834
 <small>featuring Siltek™ deactivation</small> Siltek™ Auto SYS Splitter w/ Siltek™ Glass Wool	universal for most common analyses	4.0 ID 6.2 OD x 92.1	N6101052	20832-213.1	20833-213.5	20834-213.25
 Auto SYS Cup Splitter	high & low MW compounds	4.0 ID 6.2 OD x 92.1	-	20835	20836	-
 Auto SYS Cyclo splitter®	dirty samples, max. injections before cleaning required	4.0 ID 6.2 OD x 92.1	-	20910	20911	-
 Auto SYS Laminar Cup Splitter	high MW compounds	4.0 ID 6.2 OD x 92.1	-	20827	20828	-
 Auto SYS Split Precision™ Liner	dirty samples, trace samples	4.0 ID 6.2 OD x 92.1	-	21026	21027	-
Splitless Liners for PerkinElmer GCs	Benefits/Uses:	ID**/OD & Length (mm)	Similar to PE part #	ea.	cat.# 5-pk.	25-pk.
 Splitless (2mm ID)	trace samples	2.0 ID 5.0 OD x 100	0330-5180	20730	20731	20732
 Auto SYS Splitless w/Wool (2mm ID)*	trace samples	2.0 ID 6.2 OD x 92.1	N6101372	20829	20830	20831
 <small>featuring Siltek™ deactivation</small> Siltek™ Auto SYS Splitless w/Siltek Glass Wool (2mm ID)	trace samples	2.0 ID 6.2 OD x 92.1	N6101372	20829-213.1	20830-213.5	20831-213.25
 Auto SYS Double Gooseneck	trace, active samples up to 4µL	4.0 ID 6.2 OD x 92.1	-	20853	20854	-
 Auto SYS Cyclo Double Gooseneck	trace, dirty, active samples, up to 4µL	4.0 ID 6.2 OD x 92.1	-	20899	20900	-
DI Liners for PerkinElmer GCs (0.32/0.53mm ID)	Benefits/Uses:	ID**/OD & Length (mm)	Similar to PE part#	ea.	cat.# 5-pk.	25-pk.
 Uniliner®	trace, active samples, high recovery & linearity	3.5 ID 5.0 OD x 100	-	20855	20856	-
 Cyclo-Uniliner®	trace, dirty, active samples, high linearity	3.5 ID 5.0 OD x 100	-	20857	20858	-
 Auto SYS Open-top Uniliner® w/Wool*	trace, dirty, active samples, high recov- ery & linearity	4.0 ID 6.2 OD x 92.1	-	20837	20838	-
 Auto SYS Cyclo-Uniliner®	trace, dirty, high MW active samples, high linearity	4.0 ID 6.2 OD x 92.1	-	20839	20840	-
 Auto SYS Drilled Uniliner®	allows direct injection when using an EPC-equipped GC	4.0 ID 6.2 OD x 92.1	-	20819	20822	-

*Prepacked with fused silica wool. For glass wool instead, add the suffix "-202" to the liner catalog number.

**Nominal ID at syringe needle expulsion point.

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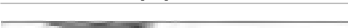



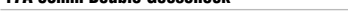
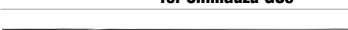
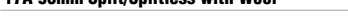
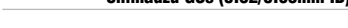




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all liners are
100%
deactivated

Liners for Shimadzu GCs

NEW SPLITLESS LINERS

Split Liners for Shimadzu GCs	Benefits/Uses:	ID**/OD & Length (mm)	Similar to Shimadzu part #	ea.	cat.# 5-pk.	25-pk.
 17A 1mm Split	purge & trap & fast GC	1.0 ID 5.0 OD x 95	-	20976	20977	20978
 128mm Split	universal, for most common analyses	3.5 ID 5.0 OD x 128	221-25822-01	20751	20752	20753
 128mm Cycloplitter®	dirty samples, many injections before cleaning required	3.5 ID 5.0 OD x 128	-	20754	20755	-
 128mm Cup Splitter	high & low MW compounds	3.5 ID 5.0 OD x 128	-	20757	20758	-
 128mm Laminar Cup Splitter	high MW compounds	3.5 ID 5.0 OD x 128	-	20807	20808	-
 99mm Split	universal, for most common analyses	3.5 ID 5.0 OD x 99	221-32544-01	20860	20861	20862
 99mm Cycloplitter®	dirty samples, many injections before cleaning required	3.5 ID 5.0 OD x 99	-	20870	20871	-
 99mm Cup Splitter	high MW compounds	3.5 ID 5.0 OD x 99	-	20866	20867	-
 99mm Laminar Cup Splitter	high MW compounds	3.5 ID 5.0 OD x 99	-	20868	20869	-
 17A Split Precision™ Liner	trace samples, dirty samples	3.5 ID 5.0 OD x 95	-	21020	21021	-
Splitless Liners for Shimadzu GCs	Benefits/Uses:	ID**/OD & Length (mm)	Similar to Shimadzu part #	ea.	cat.# 5-pk.	25-pk.
 128mm Splitless (3mm ID)	trace samples	3.5 ID 5.0 OD x 128	221-25440-03	20748	20749	20750
 99mm Splitless (3mm ID)	trace samples	3.5 ID 5.0 OD x 99	221-32544-00	20863	20864	20865
 17A 95mm Double Gooseneck	reduces backflash and catalytic decomposition	3.5 ID 5.0 OD x 95	-	20958	20959	20960
 17A 95mm Single Gooseneck	reduces backflash, also operates in DI mode	3.5 ID 5.0 OD x 95	221-41599-00	20961	20962	20963
Split/Splitless Liners for Shimadzu GCs	Benefits/Uses:	ID**/OD & Length (mm)	Similar to Shimadzu part #	ea.	cat.# 5-pk.	25-pk.
 17A 95mm Split/Splitless with Wool*	universal, for most common analyses	3.5 ID 5.0 OD x 95	221-41444-00	20955	20956	20957
 Siltek™ 95mm Split/Splitless w/ Siltek™ Glass Wool	universal, for most common analyses	3.5 ID 5.0 OD x 95	221-41444-00	20955-213.1	20956-213.5	20957-213.25
DI Liners for Shimadzu GCs (0.32/0.53mm ID)	Benefits/Uses:	ID**/OD & Length (mm)	Similar to Shimadzu part #	ea.	cat.# 5-pk.	25-pk.
 128mm Uniliner®	trace, active samples, high recovery & linearity	3.5 ID 5.0 OD x 128	-	20872	20873	-
 128mm Cyclo-Uniliner®	trace, dirty, high MW active samples, high linearity	3.5 ID 5.0 OD x 128	-	20874	20875	-
 99mm Uniliner®	trace, active samples, high recovery & linearity	3.5 ID 5.0 OD x 99	-	20876	20877	-
 99mm Cyclo-Uniliner®	trace, dirty, high MW active samples, high recovery & linearity	3.5 ID 5.0 OD x 99	-	20893	20894	-
 95mm Uniliner® with Wool*	trace, dirty, high MW active samples, high recovery & linearity	3.5 ID 5.0 OD x 95	-	21713	21719	-







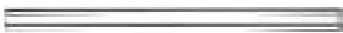









*This liner is prepacked with fused silica wool. To order glass wool instead, add the suffix "-202" to the liner catalog number.

**Nominal ID at spring needle penetration point

all liners are
100%
deactivated

Liners for Thermo Finnigan

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Split Liners for 5000-6000 Series GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to TF part #	ea.	cat.# 5-pk.	25-pk.
	Laminar Cup Splitter	high MW compounds	4.0 ID 5.4 OD x 79.5	-	20809	20810	-
	Cyclosplitter®	dirty samples, many injections before cleaning required	4.0 ID 5.4 OD x 79.5	-	20817	20818	-
	Cup Splitter Gooseneck	high & low MW compounds	4.0 ID 5.4 OD x 79.5	-	20885	20886	-
Splitless Liners for 5000-6000 Series GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to TF part #	ea.	cat.# 5-pk.	25-pk.
	Splitless (2mm ID)	trace samples	2.0 ID 5.4 OD x 79.5	-	20811	20812	20813
	Splitless (4mm ID)	trace samples	4.0 ID 5.4 OD x 79.5	-	20814	20815	20816
DI Liners for 5000-6000 Series GCs (0.32/0.53 ID)		Benefits/Uses:	ID**/OD & Length (mm)	Similar to TF part #	ea.	cat.# 5-pk.	25-pk.
	Open-top Uniliner® w/Wool*	trace, dirty, active samples, high recovery & linearity	4.0 ID 5.4 OD x 79.5	-	20841	20842	-
Split Liners for 8000 & TRACE™ Series GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to TF part #	ea.	cat.# 5-pk.	25-pk.
	1mm Split	purge & trap & fast GC	1.0 ID 8.0 OD x 105	453 20075	20916	20917	-
	3mm Split	universal	3.0 ID 8.0 OD x 105	453 20031	20936	20937	20938
	5mm Split	universal	5.0 ID 8.0 OD x 105	453 20030	20939	20940	20941
	Laminar Cup Splitter	high MW compounds	4.0 ID 8.0 OD x 105	-	20948	20949	-
	Cup Splitter	high & low MW compounds	4.0 ID 8.0 OD x 105	-	20950	20951	-
	5mm Split Precision™ Liner	trace samples, dirty samples	5.0 ID 8.0 OD x 105	-	21028	21029	-
Splitless Liners for 8000 & TRACE™ Series GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to TF part #	ea.	cat.# 5-pk.	25-pk.
	Splitless (3mm ID)	trace samples	3.0 ID 8.0 OD x 105	453 20032	20942	20943	20944
	Siltek™ Splitless (3mm ID)	trace samples <small>featuring Siltek™ deactivation</small>	3.0 ID 8.0 OD x 105	453 20032	20942-214.1	20943-214.5	20944-214.25
	Splitless (5mm ID)	trace samples	5.0 ID 8.0 OD x 105	453 20033	20945	20946	20947
	Double Gooseneck	trace active samples up to 4µL	4.0 ID 8.0 OD x 105	-	20952	20953	-




*Packed with fused silica wool. For glass wool instead, add the suffix "-202" to the liner catalog number.

**Nominal ID at syringe needle expulsion point.

all liners are
100%
deactivated

Liners for Thermo Finnigan

COLUMN INSTALLS THIS END

DI Liners for 8000 & TRACE™ Series GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to TF part #	ea.	cat.# 5-pk.	25-pk.
 Uniliner® w/Wool		trace, active samples, high recovery, & linearity	5.0 ID 8.0 OD x 105	-	21005	21006	-
Split Liners for TRACE™ 2000 GCs		Benefits/Uses:	ID**/OD & Length (mm)	Similar to TF part #	ea.	cat.# 5-pk.	25-pk.
 1mm ID Trace 2000 Glass Liner		trace samples, high recovery & linearity	1mm ID 2.95 OD x 120	-	21114	21115	-
 2mm ID Trace 2000 Glass Liner		universal	2mm ID 2.95 OD x 120	-	21116	21117	-

**Nominal ID at syringe needle expulsion point.

Inlet Liner Seal for TRACE™ 2000 GCs

Description	qty.	cat.#
Inlet Liner Seal	2-pk.	21392



Graphite Sealing Ring and Washer for 8000 Series and TRACE™ GC Inlet Liners

(Similar to Thermo Finnigan part # 290-03406)

Description	qty.	cat.#
Graphite Sealing Ring and Washer	ea.	21898
Graphite Sealing Rings and Washers	2-pk.	21899



Graphite Ferrules for M4 Fittings

- High-purity, high-density graphite.
- Smoother surface and cleaner edges than conventional graphite ferrules.
- Contain no binders that can off-gas or adsorb analytes.
- Stable to 450°C.

Graphite Ferrules for M4 Fittings for QCQ Thermo Finnigan 8000 & TRACE™ 2000

Ferrule ID	Fits Column ID	Graphite 2-pk.	Graphite 10-pk.
0.4mm*	0.18–0.25mm	20280	20281
0.5mm*	0.28/0.32mm	20282	20283
0.8mm*	0.45/0.50 & 0.53mm	20284	20285

*0.4mm ID ferrule is similar to Thermo Finnigan part #290-13488, 0.5mm ID ferrule is similar to Thermo Finnigan part #290-13487, and 0.8mm ID ferrule is similar to Thermo Finnigan part #290-13486.



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handy Septum Size Chart

Instrument	Septum Size
Agilent (HP)	
5880A, 5890, 6890, 6850	11mm
5700, 5880	9.5/10mm
On-Column Injection	5mm
CE Instruments (TMO)	
TRACE GC	17mm
Finnigan (TMO)	
GC 9001	9.5mm
GCQ	9.5mm
GCQ w/TRACE	17mm
QCQ™	9.5mm
TRACE 2000	9.5mm
Fisons/Carlo Erba (TMO)	
8000 series	17mm
Gow-Mac	
6890 series	11mm
All other models	9.5mm
PerkinElmer	
Sigma series	11mm
900, 990	11mm
8000 series	11mm
Auto SYS	11mm
Auto SYS XL	11mm
Pye/Unicam	
All models	7mm
Shimadzu	
All models	Plug
SRI	
All models	Plug
Tracor	
540	11.5mm
550, 560	9.5mm
220, 222	12.5mm
Varian	
<i>Injector type:</i>	
Packed column	9.5/10mm
Split/splitless	
1078/1079	10/11mm
1177	9mm
1075/1077	11mm

Thermolite® Septa

- Usable to 340°C inlet temperatures.
- Each batch tested on FIDs, ECDs, and MSDs to ensure lowest bleed.
- Excellent puncturability.
- Preconditioned and ready to use.
- Do not adhere to hot metal surfaces.
- Packaged in non-contaminating glass jars.



Septum Diameter	25-pk.	50-pk.	100-pk.
5mm ($\frac{3}{16}$ "	20351	20352	20353
6mm ($\frac{1}{4}$ "	20355	20356	20357
7mm	20381	20382	20383
8mm	20370	20371	—
9mm	20354	20358	20362
9.5mm ($\frac{3}{8}$ "	20359	20360	20361
10mm	20378	20379	20380
11mm ($\frac{7}{16}$ "	20363	20364	20365
11.5mm	22385	22386	22387
12.5mm ($\frac{1}{2}$ "	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Plug	20372	20373	20374

InfraRed™ Septa

- Usable to 325°C inlet temperatures.
- Preconditioned and ready to use.
- Excellent puncturability.
- Do not adhere to hot metal surfaces.
- Low bleed.
- Packaged in non-contaminating glass jars.



Septum Diameter	25-pk.	50-pk.	100-pk.
9mm	21417	21418	21419
9.5mm ($\frac{3}{8}$ "	21421	21422	21423
10mm	21424	21425	21426
11mm ($\frac{7}{16}$ "	21427	21428	21429
11.5mm	21430	21431	21432
12.5mm ($\frac{1}{2}$ "	21433	21434	21435
17mm	21436	21437	21438
Shimadzu Plug	21439	21440	21441

IceBlue™ Septa

- Usable to 250°C inlet temperatures.
- General-purpose septa.
- Excellent puncturability.
- Preconditioned and ready to use.
- Do not adhere to hot metal surfaces.
- Packaged in non-contaminating glass jars.
- Ideal for SPME.



Septum Diameter	50-pk.	100-pk.
9mm	22381	22382
9.5mm ($\frac{3}{8}$ "	22388	22389
10mm	22390	22391
11mm ($\frac{7}{16}$ "	22392	22393
11.5mm	22383	22384
12.5mm ($\frac{1}{2}$ "	22394	22395
17mm	22396	22397
Shimadzu plug	22398	22399

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Siltek™ Press-Tight® Connectors

- Siltek™ deactivation for inert pathways to maintain sample integrity.
- Ideal for connecting guard columns to analytical columns.
- Angled Press-Tight® connector designed at an angle approximating the curvature of a capillary column to reduce strain on column-end connections.
- Fits 0.18, 0.25, 0.32, & 0.53mm ID columns.

Siltek™ Press-Tight™ Connectors

Press-Tight® Connector	ea.	3-pk.	5-pk.	25-pk.	100-pk.
Universal Press-Tight® Connector	—	—	20480	20449	20481
Universal Angled Press-Tight® Connector	—	—	20482	20483	20484
Universal “Y” Press-Tight® Connector	20485	20486	—	—	—
Universal Angled “Y” Press-Tight® Connector	20487	20469	—	—	—

Universal Angled Press-Tight® Connectors

- Designed at an angle approximating the curvature of a capillary column.
- Reduces strain on column-end connections.
- Ideal for connecting guard columns to analytical columns.
- Seals all common sizes of fused silica tubing (0.18 to 0.53mm ID, outside diameters from 0.3 to 0.75mm).
- Made from inert fused silica.

Description	qty.	cat.#
Universal Angled Press-Tight® Connectors	5-pk.	20446
Universal Angled Press-Tight® Connectors	25-pk.	20447
Universal Angled Press-Tight® Connectors	100-pk.	20448

Universal Press-Tight® Connectors

- Connect guard columns to analytical columns.
- Repair broken columns.
- Connect column outlets to transfer lines.

Description	qty.	cat.#
Universal Press-Tight® Connectors	5-pk.	20400
Universal Press-Tight® Connectors	25-pk.	20401
Universal Press-Tight® Connectors	100-pk.	20402

Deactivated, Universal Press-Tight® Connectors

- High-temperature silanization for excellent inertness.
- Ideal for trace analysis of active compounds.
- Ideal for analysis of pesticides, semivolatile pollutants, or clinical/forensic samples.

Description	qty.	cat.#
Deactivated, Universal Press-Tight® Connectors	5-pk.	20429
Deactivated, Universal Press-Tight® Connectors	25-pk.	20430
Deactivated, Universal Press-Tight® Connectors	100-pk.	20431

Universal “Y” Press-Tight® Connectors

- Split sample flow onto two columns.
- Split a single column flow into two detectors—perform confirmational analysis with a single injection.
- Fit 0.18, 0.25, 0.32, & 0.53mm ID columns.

Description	qty.	cat.#
Universal “Y” Press-Tight® Connector	ea.	20405
Universal “Y” Press-Tight® Connectors	3-pk.	20406



*Siltek™—the most
inert deactivation available!*



Polyimide Resin

- Permanently connects a Press-Tight® connector to a fused silica column.
- 350°C maximum operating temperature.



Description	qty.	cat.#
Polyimide Resin	5 grams	20445

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Use for fused silica-to-fused silica or fused silica-to-metal connections!



Ideal for MXT® stainless steel to fused silica capillary connections!

MXT®-Union Connector Kits—For Fused Silica Columns

- Low-dead-volume, leak-free connection.
- Reusable.
- Silcosteel® treatment ensures maximum inertness.
- Ideal for connecting guard columns and transfer lines.
- Use to oven temperatures of 350°C.
- Available in union and “Y” configurations.

Previously, easy-to-use MXT® connectors could only be used with metal tubing. Now MXT® connectors can be used with fused silica capillary columns, because of a Valcon polyimide $\frac{1}{32}$ -inch one-piece fused silica adaptor. This unique graphite-reinforced composite allows capillary columns to slide into and be locked in place simply by loosening and tightening the MXT® union $\frac{1}{32}$ -inch fitting.

MXT®-Union Connector Kits—For Fused Silica Columns

Each kit contains the MXT® union, two $\frac{1}{32}$ -inch nuts and two one-piece fused silica adaptors.

Description	qty.	cat.#
For 0.25mm ID Fused Silica Columns	kit	21386
For 0.32mm ID Fused Silica Columns	kit	21385
For 0.53mm ID Fused Silica Columns	kit	21384

MXT® “Y”-Union Connector Kits—For Fused Silica Columns

Each kit contains the MXT® union, three $\frac{1}{32}$ " nuts and three one-piece fused silica adaptors.

Description	qty.	cat.#
For 0.25mm ID Fused Silica Columns	kit	21389
For 0.32mm ID Fused Silica Columns	kit	21388
For 0.53mm ID Fused Silica Columns	kit	21387

$\frac{1}{32}$ -Inch Replacement Nut

Description	qty.	cat.#
$\frac{1}{32}$ " Replacement Nut	5-pk.	20389

Valco® Connectors—One-Piece Fused Silica Adaptor Ferrule

We recommend a one-piece adaptor ferrule for use in fittings where the ferrule will not be removed. Connections are made and disconnected by loosening the fitting nut and sliding the tube out. Fused silica adaptor ferrules are available in Valcon polyimide for use up to 350°C. Valcon polyimide is a unique graphite-reinforced composite, specially prepared to maximize mechanical stability at high temperatures. The determining factor in adaptor ferrule size selection is the fused silica tubing outer diameter (OD).

$\frac{1}{32}$ -Inch Adaptor Ferrule

Tubing OD	Tubing ID	Valco® #	Valcon Polyimide	
			qty.	cat.#
<0.25–0.4mm	0.25mm	FS.4-5	5-pk.	20137
0.4–0.5mm	0.32mm	FS.5-5	5-pk.	20140
0.5–0.8mm	0.53mm	FS.5V-5	5-pk.	20141
$\frac{1}{32}$ " Replacement Nut			5-pk.	20389

Gerstel GRAPHPACK® 3D/2 Connectors

GRAPHPACK® technology provides a complete system that quickly and reliably makes leak-free, low-dead-volume connections. The central component is a metal-jacketed graphite ferrule—the ideal seal for GC applications. GRAPHPACK® ferrules eliminate all the disadvantages and shortcomings associated with previous sealing systems.

Description	qty.	cat.#
GRAPHPACK® 3D/2 Connector** (0.25mm to 0.32mm ID)	ea.	20272
GRAPHPACK® 3D/2 Connector** (0.45mm to 0.7mm ID)	ea.	20273

**Use only with GRAPHPACK® 3D/2 ferrules.

GRAPHPACK® 3D/2 Ferrules

Ferrule ID	Fits Column ID	qty.	cat.#
0.4mm	0.25mm	10-pk.	20271
0.5mm	0.32mm	10-pk.	20270
0.8mm	0.45/0.53mm	10-pk.	20274

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Intermediate-Polarity Deactivated Guard Columns & Transfer Lines

- Useful for a wide range of applications.
- Compatible with most common solvents.

Fused Silica Guard Columns/Transfer Lines

Nominal ID	Nominal OD	1-Meter	5-Meter	5-Meter/6-pk.
0.025mm*	0.363 ± 0.012mm	10097		
0.05mm	0.363 ± 0.012mm	10098	10040	10040-600
0.075mm*	0.363 ± 0.012mm	10099		
0.10mm	0.363 ± 0.012mm	10100	10041	
0.15mm	0.363 ± 0.012mm	10101	10042	
0.18mm	0.37 ± 0.04mm	10102	10046	
0.25mm	0.37 ± 0.04mm		10043	10043-600
0.28mm	0.37 ± 0.04mm		10003	10003-600
0.32mm	0.45 ± 0.04mm		10044	10044-600
0.45mm	0.69 ± 0.04mm		10005	10005-600
0.53mm	0.69 ± 0.05mm		10045	10045-600

Nominal ID	Nominal OD	10-Meter	10-Meter/6-pk.	30-Meter**	60-Meter**†
0.25mm	0.37 ± 0.04mm	10049	10049-600	10012	10013
0.32mm	0.45 ± 0.04mm	10048	10048-600	10022	10023
0.53mm	0.69 ± 0.05mm	10047		10032	10033

MXT® Guard Columns/Transfer Lines

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.	10-Meter
0.28mm	0.53 ± 0.025mm	70044	70044-600	70046
0.53mm	0.74 ± 0.025mm	70045	70045-600	70047

Siltek™-Deactivated Guard Columns

- Revolutionary deactivation process lowers analyte breakdown to less than 1%.
- Minimizes bleed.
- Ideal for chlorinated pesticide analysis.
- Analyze tough samples quickly and accurately.
- Maximum temperature of 380°C.

Siltek™-Deactivated Guard Columns

Nominal ID	Nominal OD	5-Meter	10-Meter
0.25mm	0.37 ± 0.04mm	10026	10036
0.32mm	0.45 ± 0.04mm	10027	10037
0.53mm	0.69 ± 0.05mm	10028	10038

Let Restek Make the Connection for You!

Restek will connect a Siltek™ guard column to any analytical column using a universal Siltek™ Press-Tight® connector and polyimide sealing resin. To order a preconnected guard column, add the three-digit suffix from the chart below to any analytical column catalog number when ordering.

5m Siltek™ Guard Column/Transfer Line

ID	cat.# suffix
0.25mm	-364
0.32mm	-365
0.53mm	-366

Example:

A 5m, 0.32mm ID Siltek™ guard column connected to a 30m, 0.32mm ID, 1.0µm Rtx®-5 column is cat.# 10254-365.

Restek Trademarks: Siltek, Press-Tight, MXT, CarboFrit, Rtx, Uniliner, Silcosteel, Stx, Leak Detective, Stabilwax, Cyclosplitter, mini-Lam, Precision, InfraRed, IceBlue, Plus 1.

Other Trademarks: Valco (Valco Instruments Co., Inc.), GRAPHACK (Gerstel GmbH), Carbowax (Union Carbide Corp.), TRACE (ThermoQuest Corp.), Velcro (Velcro Industries BV), Scotty (Scott Specialty Gases, Inc.), Viton & VESPEL (E.I. du Pont de Nemours & Co., Inc.).

*Not tested with the Grob test mix because of a high pressure drop.

**30- and 60-meter lengths are banded in 5-meter sections.

†Recommendation: Cut 60m guard columns into shorter lengths. Using full length may cause peak distortion.

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Reach for Restek

Plus 1 *Restek's Customer Commitment*

Plus 1™ Service means we will surpass your expectations every time you contact us! You'll get Plus 1™ service when you ask our experienced Technical Service team to help solve a difficult analytical problem. Our efficient Customer Service Team will provide Plus 1™ service even when you place a late-day order. Keep reaching for Restek products and service, and we will provide you with Plus 1™ quality and attention.



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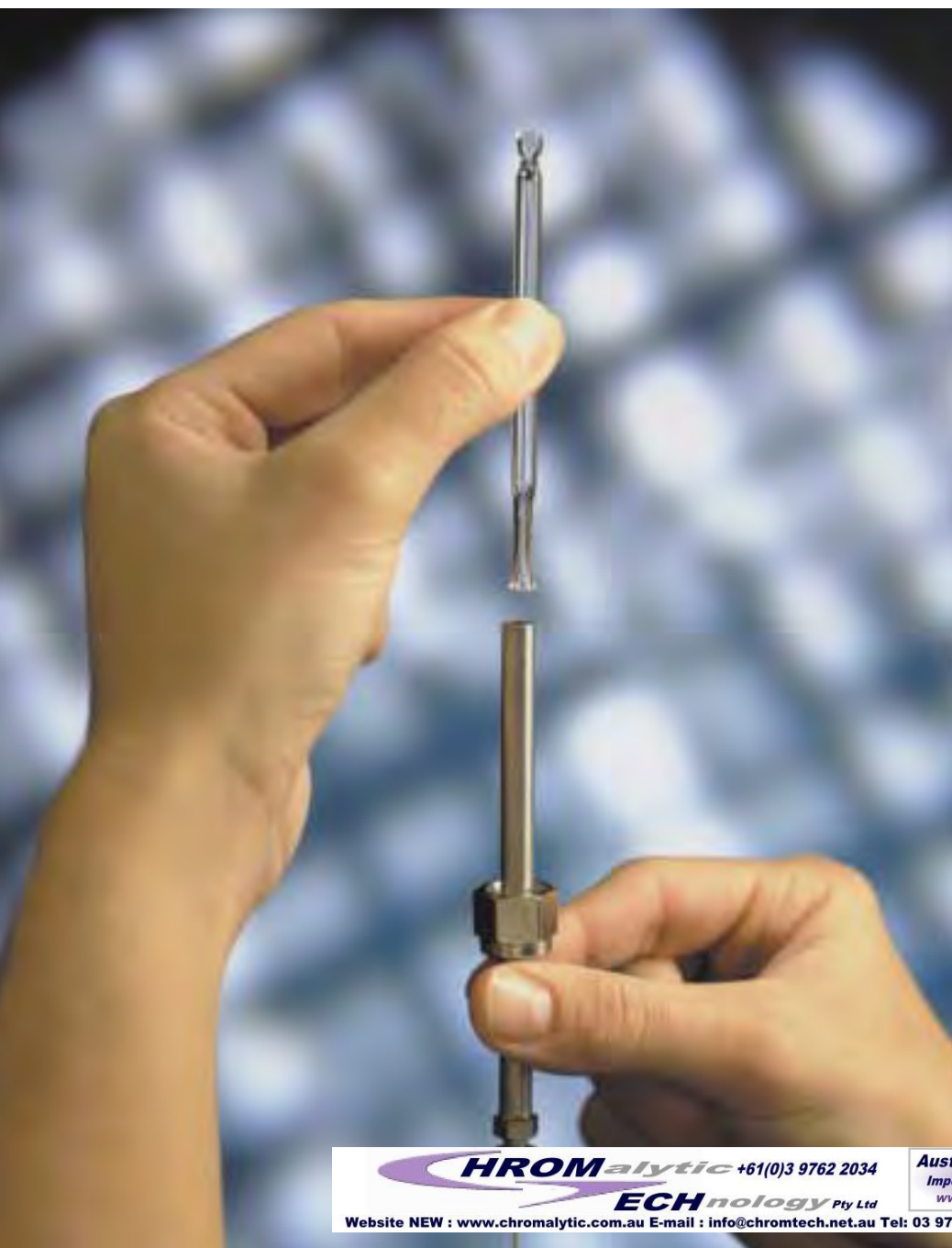


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Lit. Cat. #59880A

A Guide to Direct and On-column Flash Vaporization Injection



Inside:

Overviews of direct and on-column flash vaporization injection techniques

Advantages of direct injection over splitless injection

Practical guidelines to maximize analysis accuracy and reproducibility

Recommendations for converting a split/splitless injection port for direct injections

Alternatives for converting a packed column injection port for direct injections onto 0.32mm ID or 0.53mm ID capillary columns

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Introduction to Direct Injection

While splitless injection is still the most popular trace sample introduction technique, direct injection is rapidly gaining in popularity as analysts search for better ways to analyze trace-level compounds or for simpler conversions from packed to capillary systems. Compared to splitless analysis, direct injection offers higher sensitivity, reduced adsorption of active compounds, and operational simplicity. In addition, the high performance and low cost of the equipment required to perform direct injection has accelerated its acceptance as a mainstream sample introduction technique.

This guide discusses the many important aspects of direct flash vaporization injection on 0.32mm or 0.53mm ID capillary columns. Advantages and disadvantages of direct injection are outlined and compared to the performance of other injection techniques. Considerations such as modifying splitless inlets, retrofitting packed column injection ports, and the need for make-up gas are discussed. Finally, operational parameters such as flow rates and sample injection volume versus injection speed are reviewed.

What is direct flash vaporization injection?

In direct flash vaporization injection, a liquid sample is injected via a syringe into a heated injection port. The sample is rapidly vaporized in the injection port, then transferred to the column. This injection technique is very popular with packed columns but, when used with the first-developed capillary columns, the highly concentrated samples caused overloaded peaks and poor resolution. Subsequently, split systems were developed to provide on-column sample volumes and analyte quantities that were compatible with the low flow rates and limited capacities of capillary columns. Split injections produced symmetrical peaks, while allowing concentrated samples to be injected in the same manner as in packed column systems. Later, split systems were modified to include a splitless injection mode to direct the entire sample into the column for analysis of trace analytes. Direct injection parallels splitless injection at the beginning of the sample introduction process ("purge off" mode or split vent closed), but in splitless injection gas flow in the injection port is switched to the "purge on" mode after a short period of time, thereby flushing excess solvent vapor from the injection port. (See Restek's *Operating Hints for Using Split/Splitless Injectors* Lit. cat.# 59880A for more information.) Because direct injections do not include a "purge on" mode, the entire sample vapor cloud is swept into the column.

Today, direct flash vaporization injections are better understood and are recognized as offering many advantages over splitless injections for the analysis of difficult components. These advantages include less adsorption of active compounds, less discrimination against high-boiling compounds, and better sensitivity for trace components. Direct injection also can be used for concentrated samples commonly analyzed on split/splitless systems, provided the sample is first diluted with a solvent and the injection volume is kept small to prevent column overload. Superior responses for active, high-boiling, or trace compounds, combined with its simplicity and low cost have led to the resurgence of direct injection in many laboratories.

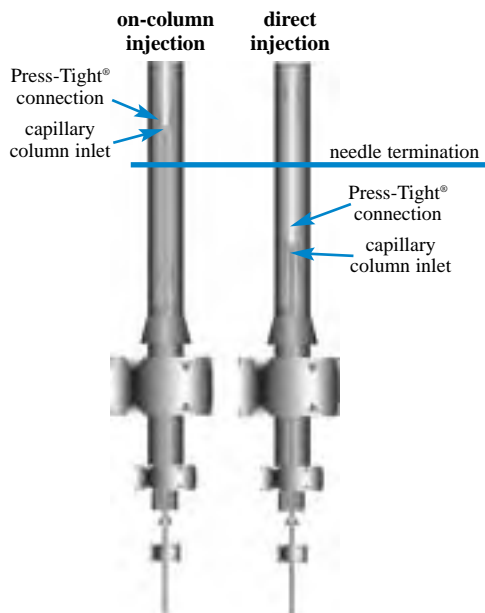
What is the difference between direct and on-column flash vaporization injection?

Both direct injection and on-column injection deliver the entire sample to the column. The difference between the two techniques is denoted by the termination point of the syringe needle during the sample injection and vaporization process (Figure 1). In an on-column injection, the tip of the needle enters the column and the liquid sample is deposited directly inside the column. In a direct injection, the tip of the needle enters a glass injection port liner and the sample is vaporized in the buffer volume of the injection port liner. Thus, in a direct injection, the sample is a vapor before it reaches the column.

Of the two, direct injection is the preferred mode of sample introduction because the injections are less problematic. On-column injections usually are made by introducing the tip of a 26-gauge needle (0.48mm OD) into the bore of a 0.53mm ID column. This tight fit leaves little space for sample expansion during the explosive vaporization process, and solvent peak tailing is a common result. On-column injections cannot be made into a 0.32mm or smaller ID column because the syringe needle is too wide to introduce into the column.

Figure 1.

Direct injection and on-column injection differ in where the syringe needle terminates during sample vaporization.



In contrast, a direct injection is made into a 2mm or 4mm ID injection port liner, and the large buffer volume of the liner ensures adequate space for sample vaporization. The vaporized sample is transferred to a 0.32mm or 0.53mm ID column. Larger sample volumes can be used with direct injections, with minimal sample backflash and solvent peak tailing.

Direct injection also reduces the need for column maintenance relative to on-column injection. In on-column injections, the tip of the syringe needle enters the column bore and there is a potential for chipping the edge of the column or damaging its inner surface. Fragments of fused silica and scratches in the stationary phase coating in the interior of the column inlet can produce adsorptive sites for active sample components, thereby requiring portions of the inlet side of the column to be removed periodically.

Direct injection, however, reduces the frequency of maintenance cycles because there is less physical damage to the column inlet. On the other hand, direct injection dictates that the injection port liner be changed or cleaned during routine maintenance.

When samples are dirty or contain non-volatile residue, direct injection should be used instead of on-column injection. The injector liner will trap the non-volatile sample residue and keep it from entering the column. Maintenance is minimal—simply clean or replace the injector liner. In contrast, column maintenance as a consequence of on-column injections of dirty samples requires removing the inlet end of the column from the injection port, discarding several loops of the column, then reinstalling the column. The only potential advantage to on-column injection over direct injection is that delivery of the sample into the bore of an inert fused silica column might reduce the possibility of analyte adsorption, compared to depositing the sample in an injector liner. However, using deactivated injector liners (all Restek injector liners are fully deactivated) helps minimize the likelihood of analyte adsorption during the sample vaporization and transfer process. Because direct injection results in significantly less column maintenance and allows a larger injection volume than on-column injection, direct injection is becoming much more popular as a sample introduction technique in capillary GC.¹

¹ Most factory-installed GC injectors are designed for split or splitless injections, but these injectors can be adapted for direct injections simply by purchasing liners designed for direct injection. For information about the availability of direct injection liners for your GC model, refer to pages 14-16, or call Restek's Technical Service Group at 800-356-1688 or 814-353-1300, ext. 4.

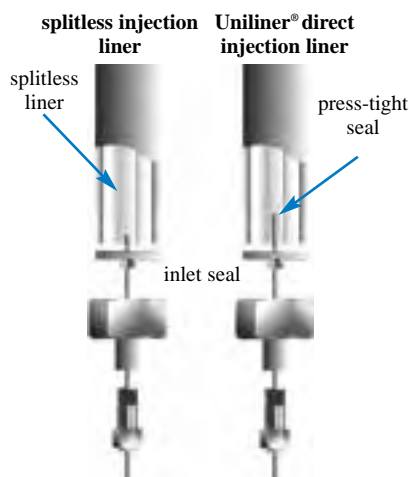
What are the advantages of direct injection over splitless injection?

Because both direct and splitless injections attempt to deliver the entire sample vapor cloud onto the column in the narrowest band possible, both techniques are used primarily for trace-level sample analysis. Only in a direct injection system, however, does the injector liner make a positive seal with the column inlet. This leak-tight connection between the liner and the column ensures that the entire sample enters the column, thereby enhancing the overall sensitivity of the analysis. Adsorption of active species is minimized and responses are greater for higher molecular weight compounds.

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Figure 2.

Splitless and direct injection liners installed in a splitless capillary injector.



A Uniliner® direct injection liner prevents the sample from contacting metal surfaces in an injection port, so sample adsorption and catalytic decomposition are reduced and responses for high molecular weight compounds are greater.

We compared a splitless injection liner and a direct injection liner installed in a capillary GC injector (Figure 2). Notice that the splitless injection liner allows the sample vapor to contact the metal inlet seal at the base of the injection port. Sample vapor potentially can adsorb onto this metal surface, or can condense in the area below the column inlet. Either occurrence can cause low response for active or high molecular weight compounds. In contrast, because the direct injection liner makes a positive seal with the column inlet, all sample vapor is directed onto the column and cannot interact with metal injection port surfaces. Therefore, sensitivity for active and high molecular weight compounds is significantly improved.

Table I summarizes the chromatographic results obtained when a mixture containing active and high molecular weight compounds was injected into three different splitless injection liners and two different direct injection liners. The most typical injection port liner used for splitless injections is a straight liner with a 4mm internal diameter, packed with deactivated fused silica or glass wool (A). The benefit in using a straight liner packed with wool is that the wool enhances sample vaporization and thereby improves the responses for high molecular weight compounds (represented in this analysis by benzo(b)- and benzo(k)fluoranthene). However, when the relative responses obtained by using a straight liner packed with wool (A) are compared to responses from two splitless injection liners with a gooseneck restriction of the internal diameter at each end (B and C), it is apparent that the active probes (2, 4-dinitrophenol, nitrophenol, and pentachlorophenol) were completely adsorbed in the injection port when the straight liner packed with wool was used. A double gooseneck liner (B) improves the response for active compounds by confining the sample cloud within the buffer volume of the liner. This reduces sample contact with the metal support disk in the injector and, thus, reduces adsorption. A double gooseneck liner with an internal glass spiral (C) not only provides good response for active compounds, but also provides better vaporization of high boiling analytes compared to a double gooseneck liner without a glass spiral. Because a

Table I.

Responses for active and high molecular weight compounds are greater with direct injection, compared to the same injector configured for splitless injection.

Analyte	Response Relative to C14				
	Splitless Injection			Direct Injection	
	A 4mm ID wool-packed	B Double Gooseneck	C Cyclo Double Gooseneck	D Uniliner®	E Cyclo-Uniliner®
benzoic acid	NA	0.90	1.23	1.06	1.21
C14	1.00	1.00	1.00	1.00	1.00
2,4-dinitrophenol	NA	0.33	0.46	0.68	0.58
nitrophenol	NA	0.73	0.93	1.24	1.17
nitroaniline	1.03	0.78	0.93	1.20	1.20
pentachlorophenol	NA	0.45	0.55	0.70	0.66
carbazole	2.01	1.43	1.69	2.17	2.06
C20	1.13	0.89	0.98	1.16	1.09
C21	1.08	0.81	0.92	1.10	1.04
C22	1.13	0.81	0.94	1.15	1.09
benzo(b)fluoranthene	2.18	1.18	1.90	2.22	2.47
benzo(k)fluoranthene	2.09	1.15	1.84	2.27	2.36

NA – Peak not quantifiable; data not available.

The data illustrate that direct injections improve responses in general, but especially improve responses for active high molecular weight compounds compared to splitless injections. A double gooseneck splitless injection liner provides better responses than an open, wool-packed splitless injection liner, but it is not until a positive seal is made between the liner and the capillary column (D and E) that significant improvements are observed.

Analysts who use direct injections instead of splitless injections can expect better overall sensitivity and greater response factors for both active and high molecular weight compounds.

30m, 0.32mm ID, 0.25µm XTI-5 column (cat.# 12224), splitless or direct injection of 1µL XTI Mix, concentration = 29ng/µL.

Oven temp.: 100°C to 285°C @ 6°C/min., then to 360°C @ 30°C/min. (hold 5 min.)
 Inj./Det. temp.: 250°C/360°C
 Carrier gas: hydrogen
 Linear velocity: 40cm/sec. set @ 100°C
 FID sensitivity: 8 x 10⁻¹¹ AFS
 Splitless hold time: 0.75 min.

- A: splitless injection, 4mm ID injector liner packed with glass wool (cat.# 20781)
- B: splitless injection, double gooseneck injector liner (cat.# 20784)
- C: splitless injection, cyclo double gooseneck injector liner (cat.# 20895)
- D: direct injection, Uniliner® injector liner (cat.# 20335)
- E: direct injection, Cyclo-Uniliner® injector liner (cat.# 20337)

All analyses conducted with an Agilent 5890 II GC equipped with an autosampler and a dirty inlet seal.

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Uniliner® direct injection liner (D or E) connects directly to the column inlet, responses for both active and high molecular weight compounds are greater than for any of the splitless injection liners. There is little difference between responses when using a Uniliner® direct injection liner (D) or a Cyclo-Uniliner® liner (E). Both liners direct all of the sample onto the column while minimizing contact between the vaporized sample and other injection port surfaces. However, a Cyclo-Uniliner® liner or a Uniliner® containing deactivated wool always should be used when injecting dirty samples.

Injection Port Setup for Direct Injection

Types of injector liners used in direct injection systems

Restek carries three styles of direct injection liners (Figure 3). Of these, the buffer volume chamber in standard Uniliner® injection port liners (A) will accommodate the largest sample vapor cloud. Because of the open design, samples should be relatively clean, otherwise contaminants could be delivered into the column inlet. For extremely dirty samples, open-top Uniliner® injection port liners (B) can be packed with deactivated fused silica wool to trap dirt and non-volatile sample residue. Contaminated wool can be replaced easily and the liner cleaned with solvent and a nylon brush. The glass spiral in a Cyclo-Uniliner® injection port liner (C) provides a surface for vaporizing high and low molecular weight compounds. Non-volatile sample residue is trapped on the first turn of the spiral, reducing subsequent interaction between that contamination and the rest of the sample. In comparison with liners packed with wool, a Cyclo-Uniliner® injection port liner will accept up to five times as many injections of dirty samples before calibration curves degrade. Each of these Uniliner® designs incorporates a gradual, press-tight taper in the base of the liner, which forms a positive seal with the column end and prevents sample components from interacting with heated metal surfaces in the injection port. The seal of the column into the press-tight taper of the injection port sleeve is the key to obtaining maximum responses for all analytes and minimizing solvent peak tailing.

Converting a split/splitless injection port to direct injection

The most common problems associated with splitless injections are caused by the absence of a direct, physical connection between the injection port liner and the column inlet (Figure 2). Sample vapor that accumulates in the space around the inlet of the column is exposed to hot, catalytic metal surfaces of the injection port. Excess sample vapor and less volatile high molecular weight compounds also can be swept out the split vent during the purge on mode. By making a leak-free connection between the injection port liner and the inlet end of the column via the press-tight taper, contact between the vaporized sample and the metal surfaces of the injection port is eliminated and the loss of sample out of the split vent is prevented.

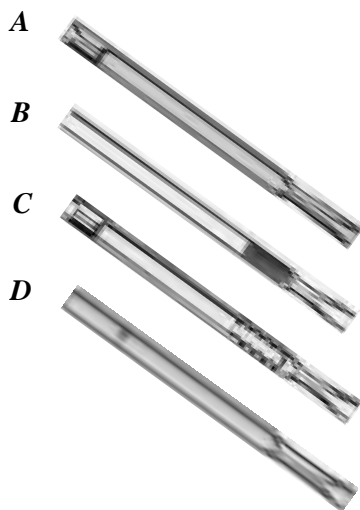


Figure 3.

Choose a direct injection liner based on sample characteristics. The buffer volume chamber accommodates the sample vapor cloud and prevents sample components from contacting metal injection port surfaces.

A) Standard Uniliner® Injector Liner

Accommodates large, relatively clean samples.

B) Open-Top Uniliner® Injector Liner with Wool

Ideal for extremely dirty samples.

C) Cyclo Uniliner® Injector Liner

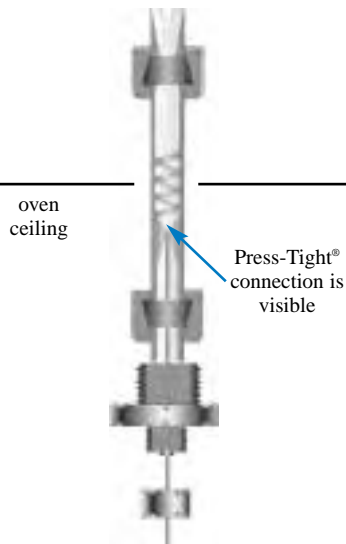
Excellent for high and low molecular weight compounds; accepts many injections of dirty samples.

D) Drilled Uniliner® Inlet Liner

The drilled hole in a Uniliner® injection port liner makes direct injection possible with EPC systems by equalizing pressure in the injection port.

Figure 4.

Vu-Tight® injection liner—in a 1/4-inch packed column injection port—allows visual confirmation of the liner-column connection.

**Figure 5.**

Good vs. poor column installation in a Vu-Tight® liner.

good installation

A brown ring indicates a good press-tight seal.



poor installation

Failure to seat the ferrule before connecting the column leads to a crushed column end.



Always seat the ferrule before connecting the column

A Uniliner® direct injection port liner can be directly substituted for a split or splitless injection port liner, quickly and easily adapting the injection port for use in the direct injection mode. A Uniliner® direct injection port liner is installed in the same manner as a splitless liner, but it must be operated continuously in the purge off mode. Uniliner® injection port liners are available for a wide variety of instruments, and are manufactured to the exact external dimensions specified for the split/splitless liners they are designed to replace.

Converting a packed column injector to direct injection

A 1/4-inch packed column injection port configured for on-column injections easily can be converted for direct injections onto 0.32mm or 0.53mm ID capillary columns by using a specially designed glass liner that forms a seal with the column inlet. Conversion of a packed column inlet is accomplished in less than 15 minutes. *Note that only packed column injectors with an on-column configuration can be retrofitted with these liners.*

We offer two injection port conversion kits in our chromatography products catalog: 1) A glass Vu-Tight® injection port liner fits directly into a 1/4-inch packed column injection port and allows visual confirmation of the connection between the injection port liner and the inlet of the capillary column (Figure 4). 2) A 1/4-inch Uniliner® sleeve adapter is fitted into a 1/4-inch injection port. A 5mm Uniliner® injection port liner can be inserted into the adapter in either direction to allow direct or on-column injections (Figure 1 on page 3). Both conversion kits incorporate the press-tight connection between the injection port liner and the inlet of the column. The combination of a sample expansion chamber and a leak-free connection between the injection port liner and the column delivers superior chromatographic performance. Direct connection of the liner to the column inlet also minimizes dead volume, reducing solvent peak tailing and sharpening peaks for early-eluting components.

Either the Vu-Tight® injection port liner or the Uniliner® sleeve adapter with liner will fit Agilent, Varian, or other common GCs with 1/4-inch packed column injection ports. Either 0.32mm or 0.53mm ID fused silica columns can be used with either conversion kit, as long as the tubing OD is 0.4mm or greater. A special high-temperature deactivating procedure creates an inert surface in the liners, so they will not absorb or react with active compounds such as pesticides, phenols, acids, or basic compounds. Liners that incorporate a glass spiral are available for either conversion system and should be used for the injection of dirty samples.

Careful – don't crush the column end!

Frequently, analysts setting up a direct injection system for the first time will crush the column end into the Press-Tight® taper. When a column is installed with a new graphite ferrule, excessive movement of the column into the Press-Tight® taper will crush the end of the column and force fused silica particles into the column bore. In order to limit the movement of the column and prevent damage, the ferrule must be seated in the fitting before the column is installed all the way into the Press-Tight® taper. To properly install a column in a direct injection liner, withdraw the column end several millimeters away from the Press-Tight® taper and tighten the ferrule into the fitting until the column end no longer moves forward into the liner. The ferrule is now seated and conforms to the interior dimensions of the fitting. Next, loosen the ferrule and push the column into the liner until it seals against the Press-Tight® taper. A dark brown ring at the tip of the column where it contacts the inside surface of the injection port liner indicates a good seal. Now, when the ferrule is retightened, the column end will not be crushed against the liner. Figure 5 shows a column properly installed in a Vu-Tight® liner, and a column that has been crushed because the ferrule was not seated first.

Detector Setup for Direct Injection

Use of make-up gas with a direct injection system

Most flame ionization detectors (FIDs) do not function optimally unless they are supplied with approximately a 1:1 ratio of carrier gas and hydrogen fuel. Because most columns used in direct injection systems operate at a carrier gas flow of between 5 and 10cc/min., make-up gas must be added to augment the low carrier gas flow.² Make-up gas improves peak shape, and may reduce detector noise and improve linearity. Similarly, electron capture detectors (ECDs) require up to 100mL of make-up gas to improve sensitivity and linearity.

PerkinElmer FIDs do not use make-up gas.

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To minimize adsorption and prevent peak tailing, always make sure that the outlet end of the column is correctly positioned inside the FID jet tip (Figure 6), close to the jet orifice or close to the entrance of the ECD detector cell. Read and carefully follow the instrument manufacturer's specifications. In an instrument designed for packed column GC, the FID jets should be replaced with jets designed to function with capillary columns or peaks might tail.

Optimizing Direct Injection

When using direct injection as the sample introduction technique, the sample is injected in a liquid form via a syringe. The sample is vaporized in the heated injection port and the carrier gas transfers the vapor to the head of the column. The two most common problems associated with hot vaporization injections in the direct injection mode are broad, tailing peaks and sample backflash. Choosing the proper carrier gas flow rate and sample size will allow you to achieve optimum chromatographic performance.

In the direct injection mode, all of the sample and carrier gas is directed into the head of the column. In order to produce the narrowest sample bandwidth possible, the vaporized sample must be transferred from the injection port liner to the head of the column as quickly as possible. If the sample is not transferred quickly, a broad, tailing solvent front will be produced and resolution of early-eluting compounds will be compromised.

Direct injection requires higher carrier gas flow rates than split or splitless injection

Direct injection systems perform best when high column flow rates are used. When a 30m, 0.32mm ID capillary column is operated with helium at a linear velocity of 20cm/sec., a column flow rate of approximately 1cc/min. is obtained. When 1µL of a sample diluted in methylene chloride is injected into a heated injection port, the sample expands to more than 400µL of gas volume. At a column flow rate of 1cc/min., it takes more than 24 seconds to transfer the vaporized sample from the buffer volume in the direct injection liner to the head of the column. If the carrier gas flow is increased to a linear velocity of 80cm/sec. (approximately 7cc/min.), the 400µL of methylene chloride vapor will be transferred from the liner to the head of the column in less than four seconds. This rapid transfer of the sample cloud ensures narrow initial sample bandwidths.

Significant differences in chromatography arising from the difference in sample delivery time are evident in Figure 7. Figure 7a shows a mixture of phenols injected using an optimum carrier gas linear velocity (hydrogen, 40 cm/sec.). Notice the tailing of the solvent peak, coelution of 2-nitrophenol and 2,4-dimethylphenol (peaks 3 and 4), and minimal baseline resolution of 2,4-dinitrophenol and 4-nitrophenol (peaks 8 and 9). Figure 7b shows the same sample delivered and separated using a faster carrier gas linear velocity (80 cm/sec.). The solvent peak is significantly sharper, and 2-nitrophenol / 2,4-dimethylphenol and 2,4-dinitrophenol / 4-nitrophenol are fully resolved. For Figure 7c, the carrier gas linear velocity was increased to 120cm/sec. (approximately 120 cm/sec.). The solvent peak shape is further improved and the two pairs of phenols are well resolved. Further increases in flow rate are counterproductive, however, because they decrease the efficiency of the column and reduce resolution of the early-eluting compounds.

³Restek offers high performance make-up gas kits to fit most GCs. For details, see our current chromatography products catalog.

Figure 6.

Restek designed the flared jet tip (arrows) to make capillary column insertion easy.



Replacement FID Jets

- Standard Version: Engineered with a fluted tip to guide the capillary column into the jet.
- High-Performance Version: Identical to the standard version, except that it has been Silcosteel®-treated. Extremely inert, use with active compounds.

Capillary Adaptable FID Replacement Jet for Agilent 5890/6890/6850 GCs (0.011-inch ID tip) (Similar to Agilent part # 19244-80560.)

Description	qty.	cat.#	qty.	cat.#
Standard	ea.	20670	3-pk.	20671
High-Performance Silcosteel®	ea.	20672	3-pk.	20673

Capillary Dedicated Replacement FID Jets for Agilent 6890/6850 GCs (Similar to Agilent part # G1531-80560.)

Description	qty.	cat.#	qty.	cat.#
Standard	ea.	21621	3-pk.	21682
High-Performance Silcosteel®	ea.	21620	3-pk.	21683

Capillary FID Replacement Jets for Agilent 5880 GCs (Similar to Agilent part # 19301-80500.)

Description	qty.	cat.#
Standard	ea.	21637
High-Performance Silcosteel®	ea.	21638

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Use only high-quality septa when making direct injections

Because all of the carrier gas flow is directed into the inlet of the column, high-quality, low-bleed septa must be used with direct injection systems that are not equipped with a septum purge. Volatile compounds that are released from septa material after heating in an injection port can make their way onto the column if not vented off using a septum purge. These compounds will focus at the head of the column when using low flow and low temperature conditions and will be displayed as ghost peaks in the chromatogram, or as increased background or column bleed.

Figure 7.

When used with direct injections, 0.32mm or 0.53mm ID columns perform better at elevated flow rates.

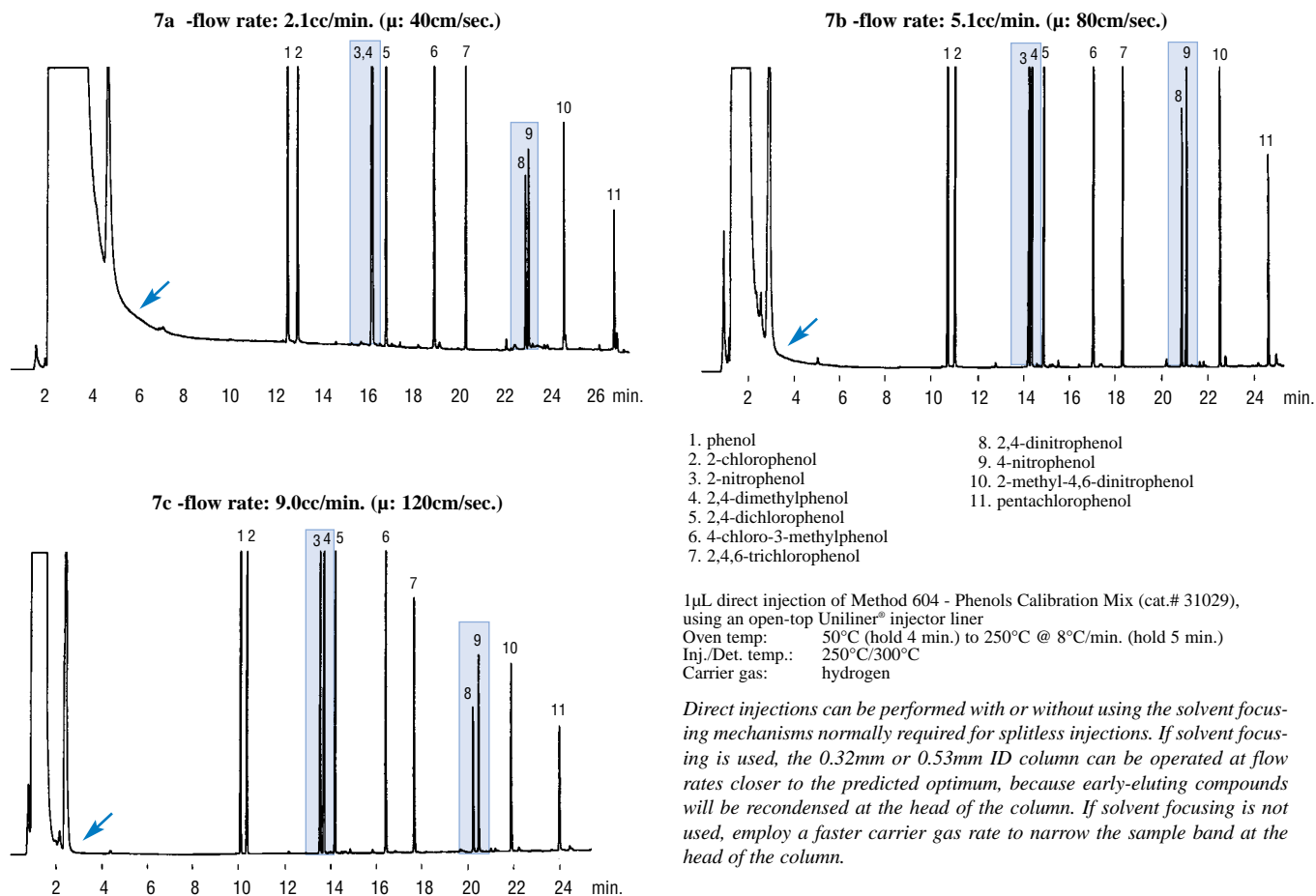


Figure 8 shows a pesticide analysis performed on a direct injection system fitted with a low-quality septum, then with a high-quality Thermolite® septum. Notice that outgassed products from the low-quality septum interfere with peaks 3, 4, and 5, and with peaks 18 and 19. Use of a packed purged injector or a splitless injector (either of which incorporates a septum purge system) greatly diminishes this problem. To ensure that your analyses are free of septum contaminants, however, it is wise to always use high-quality septa, even with a septum purge system. For more information on the advantages of using high-quality septa, request our *Guide to Minimizing Septa Problems* (Lit. cat. # 59886).

Keep injection volumes small when making direct injections

In general, analysts should strive to keep sample injection volumes as small as possible when using direct injections, ideally less than 1µL. Sample sizes greater than 1µL increase the probability of backflash, in which a portion of the vaporized sample can spill out of the top of the injection port liner. Sample backflash can lead to tailing peaks, split peaks, and poor resolution. If the sample volume must exceed 1-2µL, higher injection port pressures or slower rates of injection can minimize backflash.

Figure 8.

Low-quality septa will cause ghost peaks when used with direct injections.

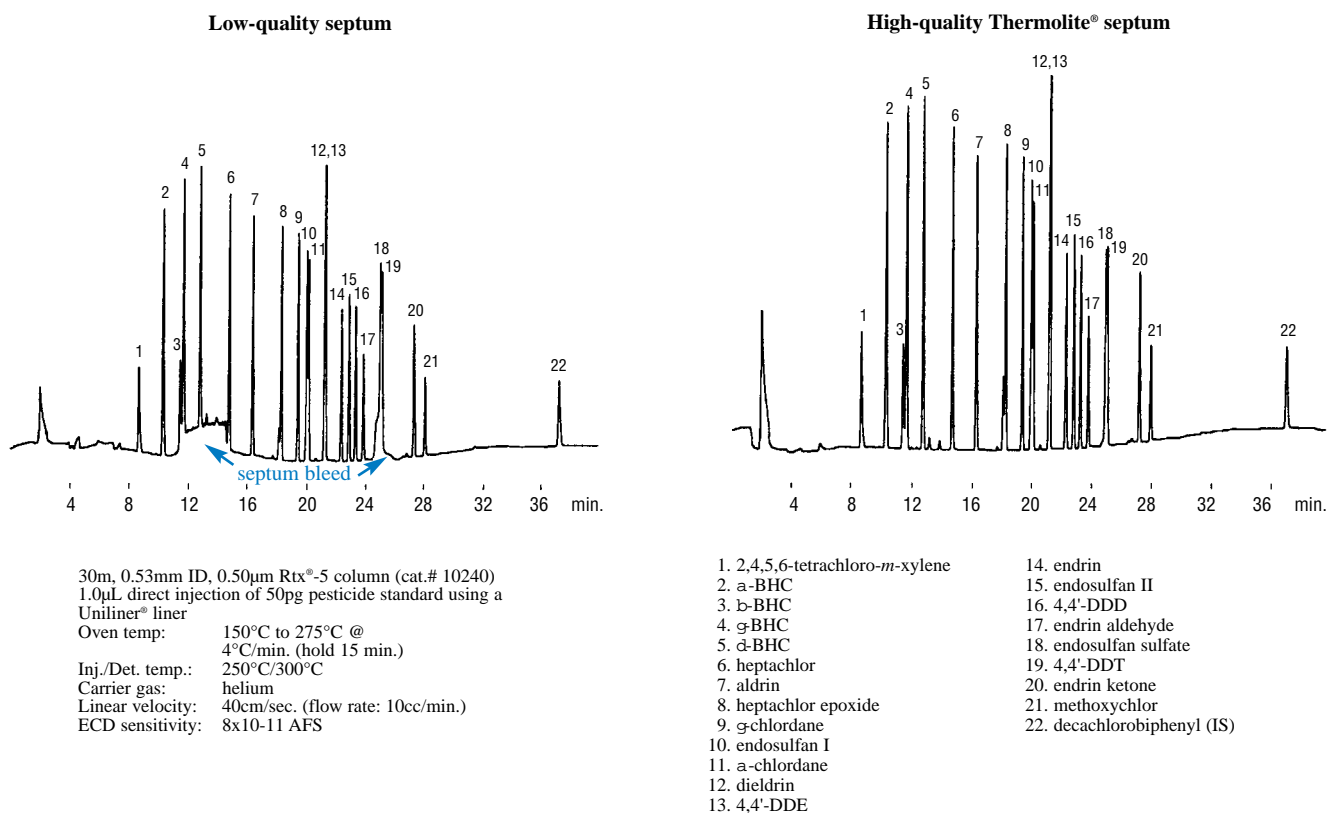


Table II shows typical expansion volumes for common sample solvents at a pressure of 10psig. These expansion volumes can be compared to the injector liner volumes in Table III to determine whether a specific combination of solvent, sample volume, and liner ID is compatible at this pressure. For example, 1 μ L of liquid methylene chloride will expand to more than 400 μ L of vapor in a heated injection port, and 2 μ L of liquid methylene chloride will expand to more than 800 μ L of vapor. A 4mm ID Uniliner[®] direct injection liner can accommodate as much as 900 μ L of vapor before the vapor will backflash out of the injector liner and cause gross solvent tailing. The liner can accept only half this volume of solvent vapor, however, because it already contains carrier gas. Thus, the 400 μ L of vapor produced by injecting 1 μ L of methylene chloride can be accommodated by a 4mm ID Uniliner[®] liner, but the 800 μ L of vapor produced by 2 μ L of methylene chloride cannot be accommodated. Solvents with a larger expansion volume than methylene chloride also will exhibit backflash. For example, 1 μ L of a water-based sample would expand to more than 1400 μ L of vapor and would exceed the buffer volume in any of the direct injection liners.

Compress the sample vapor cloud when making large injections

Table IV further illustrates the importance of minimizing sample volume to avoid backflash. Values in this table show that when a 2 μ L injection of methylene chloride is made in combination with a typical column flow rate of 10cc/min., it would take approximately 1.7 seconds for the entire vaporized sample to be carried from the injection port liner and into a 0.53mm ID column. If a 5 μ L sample is used instead of a 2 μ L sample, it would take almost nine seconds for the entire sample to enter the column.

In order to accommodate the size of the vaporized sample, a slower rate of injection could be used to allow the carrier gas flow to transfer the sample onto the column as the vapor cloud is formed. Keep in mind that slow rates of injection cannot be reproduced consistently when performing manual injections.

A better alternative for making injections larger than 1–2 μ L is to use higher carrier gas pressures in the injection port to compress the vaporized sample cloud. Table V shows how solvent density, solvent molecular weight, and pressure affect sample vapor cloud volume. Note that pressure (“P”) is in the denominator of the ideal gas law. This indicates that an increase in column head pressure will reduce the volume of the sample vapor cloud. Thus, by increasing the injection port pressure from a typical 10psig to a pressure of 15psig or more, the size of the sample vapor cloud can be reduced, allowing rapid injections of large volume samples to be made without resulting solvent peak tailing or backflash.

Table II.

Typical expansion volumes for sample solvents.

Injection Volume (liquid) (μ L)	Expansion Volume (vaporized, μ L)*				
	water	carbon disulfide	methylene chloride	hexane	isooctane
0.1	142	42	40	20	16
0.5	710	212	200	980	78
1.0	1420	423	401	195	155
2.0	2840	846	802	390	310
3.0	4260	1270	1200	585	465
4.0	5680	1690	1600	780	620
5.0	7100	2120	2000	975	775

*Expansion volumes based on a 250°C injection port temperature and a 10psig head pressure.

Table III.

Internal volumes of injector liners.†

Liner ID (mm)	Internal Volume (μ L)	
	Theoretical*	Effective**
0.53	16	
1.0	59	30
2.0	236	118
3.0	530	265
4.0	942	471

*Total internal volume for a typical 75mm-long liner.

**Liner volume available to accommodate an injection (carrier gas present in liner).

†From Grob, *Split and Splitless Injection*, 3rd ed.

Electronic pressure control and direct injection

Some GCs offer electronic pressure control (EPC), which can be used to momentarily pulse the pressure in the injection port, briefly causing an increase in carrier gas velocity during the initial injection period. Notice the reduction in solvent tailing when a 4 μ L sample of a hydrocarbon mix in methylene chloride was analyzed using EPC (Figure 9). A rapid injection was made using an Agilent 7673 autosampler into an injection port capable of EPC. The injection port pressure was increased from 8psig to 23psig just prior to the injection and was held at 23psig for 10 seconds after the injection was made. The solvent peak shape, and separation of early-eluting compounds from the solvent, are greatly improved by EPC.

Table IV.

Injection speed must be based on volume of solvent and column flow rate.

$$\text{Injection Rate} = \frac{[\text{solvent/sample expansion volume (cc)} - \text{injector liner buffer volume (cc)}]}{\text{column flow rate (cc/min.)}}$$

Injection Volume (liquid)	Expansion Volume (vaporized)	Flow rate: Injection:	Injection Time (sec.) for 0.53mm ID Columns							
			5cc/min.		10cc/min.		20cc/min.		30cc/min.	
			DI	OC	DI	OC	DI	OC	DI	OC
0.1 μ L	38 μ L		0.5	0.5	0.5	.23	0.5	.11	0.5	.08
0.5 μ L	194 μ L		0.5	2.3	0.5	1.2	0.5	.58	0.5	.39
1.0 μ L	388 μ L		1.3	4.7	0.67	2.3	0.5	1.16	0.5	.78
2.0 μ L	779 μ L		3.3	9.3	1.7	4.7	0.84	2.3	0.6	1.6
5.0 μ L	1952 μ L		17.4	23.4	8.7	11.7	4.4	5.9	2.9	3.9

DI = direct injection

OC = on-column injection

Table V.

The ideal gas law indicates that an increase in pressure greatly reduces sample expansion volume.

Solvent	Density (g/mL)	Molecular Weight	Expansion Volume (μ L)* at various column headpressures		
			5psig	10psig	15psig
heptane	0.68	100	219	174	145
hexane	0.66	86	245	196	163
pentane	0.63	72	280	224	186
toluene	0.87	92	303	242	201
ethyl acetate	0.90	88	328	261	217
chloroform	1.49	119	400	319	266
methylene chloride	1.33	85	500	399	332
methanol	0.79	32	792	629	525
water	1.00	18	1776	1418	1179

*Expansion volumes determined using a 1.0 μ L injection volume, a 250°C injection port temperature, and a headpressure of 5, 10, or 15psig (common operating pressures for 30m columns having IDs of 0.53, 0.32, or 0.25mm, respectively). For 2 μ L injections, double the volumes.

Use these formulas to calculate values not listed in Table IV or V:

Solvent/sample expansion volume (v) = nRT / P

n = number of moles of solvent and sample.

[volume (mL) x density (g/mL)] / mol wt (g/mole)

R = gas law constant

82.06cc atm/mole °K

T = absolute temperature of injector (°K)

(°K = °C + 273)

P = absolute column headpressure (atm) + 1 atm

Injector liner volume** = $\pi r^2 L$

π = 3.14

r = liner internal radius (cm)

L = liner length (cm)

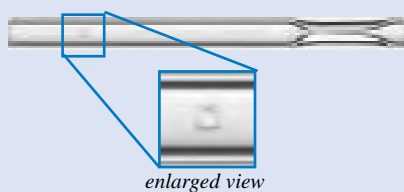
**Also use this formula to determine capillary column internal volume.

HOTtechtip

If large samples must be used for direct injection, an injection port system that incorporates a septum purge line is preferred over one without a septum purge. A septum purge will sweep away any excess solvent or sample vapor that flashes out of the top of the injection port liner. Problems associated with backflash, such as peak tailing and ghost peaks, will be minimized. However, low molecular weight or low-boiling point sample components also can be swept away in the septum purge if they experience backflash problems similar to the sample solvent. This can lead to irreproducible peak areas for early-eluting compounds, and poor quantitative accuracy.

Figure 10.

Uniliner® injector liner for Agilent 6890 GCs (cat.# 21054).



Optimizing Direct Injection When Using Agilent 6890 EPC GCs

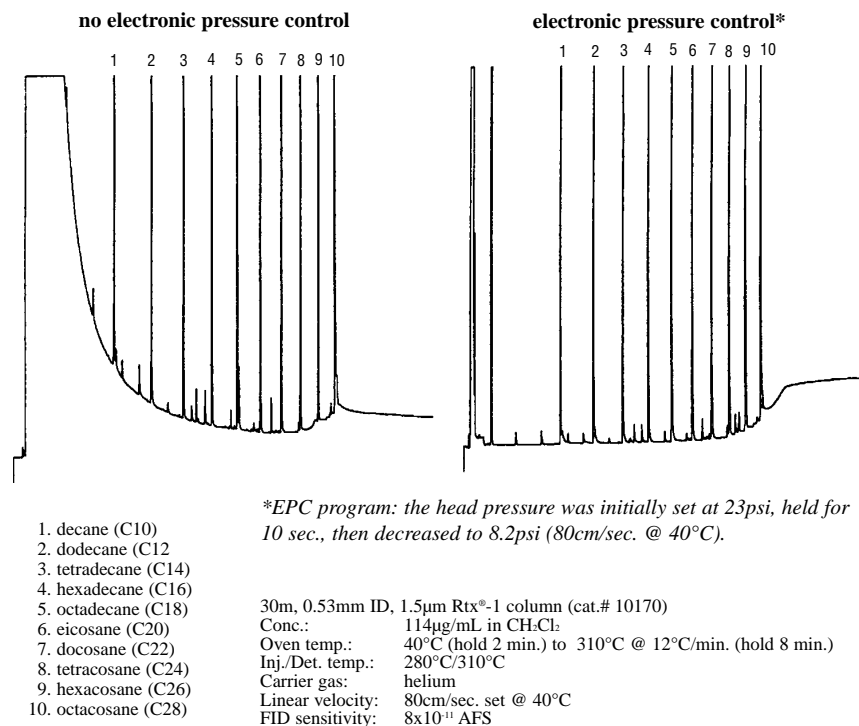
The pneumatics in an Agilent 6890 EPC GC are not the same as in previous models of Agilent GCs. The EPC design includes one pressure sensor upstream of the injection port and a second pressure sensor downstream of the injection port, at the split vent. Because a traditional Uniliner® direct injection liner seals to the analytical column, there is no downstream flow to the split vent and there will be a difference in the pressure that is measured between the two sensors when setting up for direct injection. The upstream sensor will over-compensate for the difference and a high-pressure malfunction will result.

To prevent this problem, we have designed a Uniliner® liner with a unique geometry (Figure 10) that allows you to make direct injections using your Agilent 6890 EPC GC. A small hole in the upper part of the Uniliner® liner allows a portion of the carrier gas to escape from the liner and equalize the pressure at the second sensor, thereby eliminating pressure malfunctions. The design of the Uniliner® liner for Agilent 6890 GCs requires no software or hardware modifications, or flow adjustments. For optimum performance, keep the purge on and use a very low split flow (3 to 5mL/min. or less).

Figure 9.

4µL samples can be rapidly injected via an autosampler if EPC is used to compress the sample vapor cloud—note the improved solvent peak shape.

Direct injection of 4µL hydrocarbons (EPA/Wisconsin DRO Mix, cat.# 31064) using an autosampler.



Summary

Direct injection offers higher sensitivity, less potential for adsorption of active compounds, and greater simplicity, relative to splitless injection. Less adsorption of sample components, in turn, reduces the need for column maintenance. Excellent performance and low cost of the equipment needed also are strong arguments for adopting this technique.

Products for Direct Injection Analyses

Inlet and FID Maintenance Kits for Agilent GCs

- Include the most common types of consumable supplies.
- All parts meet or exceed instrument manufacturer's specifications.
- Include parts list that makes reordering easy.

The Inlet Maintenance Kit includes these tools and many others.



Dislodge ferrules or remove silica deposits with the **Jet Reamer/Ferrule Remover**.



The **Capillary Installation Gauge** makes seating the ferrule and installing the column consistent and easy.



The **Inlet Liner Removal Tool** safely removes an inlet liner from a hot injection port without cracking the liner—and you won't burn your fingers!



Inlet kits include:

- Viton® o-rings.
- Capillary nuts.
- Inlet seals.
- Reducing nut.
- Scoring wafer.
- 11 mm Thermolite® septa.
- 4.0mm single gooseneck liner.
- 0.4, 0.5, and 0.8mm ID graphite ferrules.
- 4.0mm split liner with wool.
- Capillary column caps.
- 1/4- to 5/16-inch wrench.
- Septum puller.
- Installation gauge.
- Wire cleaning brush.
- Jet reamers/ferrule removers.
- Inlet liner removal tool.

The FID Maintenance Kit includes these tools and many others.



FID maintenance made easy with tools and replacement components specifically matched to your instrument.



The **FID Ignitor** meets original equipment specifications.



The **High-Performance Silcosteel® FID Jet** will stay clean longer—even when exposed to highly active compounds.









FID kits include:

- 1/4-inch, 0.4, 0.5, and 0.8mm ID graphite ferrules.
- FID/NPD capillary adaptor.
- Capillary nuts.
- Jet reamers/ferrule removers.
- 1/4-inch nut.
- Scoring wafer.
- Ignitor for either Agilent 5890 or 6890/6850 GCs.
- Capillary column caps.
- FID flow measuring adaptor.
- 1/4- to 5/16-inch wrench.
- Installation gauge.
- Wire cleaning brush.
- High-performance Silcosteel® FID jet for either Agilent 5890 or 6890/6850 GCs.
- 1/4-inch nut driver for jet removal.




Description	qty.	cat.#
Inlet Maintenance Kit for Agilent 5890/6890/6850 GCs	kit	21069
FID Maintenance Kit for Agilent 5890 GCs	kit	21070
FID Maintenance Kit for Agilent 6890/6850 GCs	kit	21071

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Direct Injection Liners for Agilent & Finnigan GCs (for 0.32/0.53mm ID columns)

Description	Benefits/Uses:	ID**/OD & Length (mm)	cat.# ea.	cat.# 5-pk.
 Siltek™ 1mm Uniliner****	trace, active samples, samples <1µL	1.0 ID 6.3 OD x 78.5	21052-214.1	21053-214.5
 Uniliner****	trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	20335	20336
 Siltek™ Uniliner****	trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	20335-214.1	20336-214.5
 Cyclo-Uniliner****	trace, dirty, high MW active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	20337	20338
 Siltek™ Cyclo-Uniliner****	trace, dirty, high MW active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	20337-214.1	20338-214.5
 Open-top Uniliner® with Wool***	trace, dirty, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	20843	20844

Direct Injection Liners for use with EPC Systems (for 0.25/0.32/0.53mm ID columns)

Description	Benefits/Uses:	ID**/OD & Length (mm)	cat.# ea.	cat.# 5-pk.
 Drilled Uniliner®	allows direct injection when using an EPC-equipped GC	4.0 ID 6.3 OD x 78.5	21054	21055
 Siltek™ Drilled Uniliner®	allows direct injection when using an EPC-equipped GC	4.0 ID 6.3 OD x 78.5	21054-214.1	21055-214.5
 Siltek™ 1mm Drilled Uniliner®	allows direct injection when using an EPC-equipped GC	1.0 ID 6.3 OD x 78.5	21390-214.1	21391-214.5

CIS4 and PTV Liners for Agilent GCs

Description	Benefits/Uses:	ID**/OD & Length (mm)	Similar to Agilent part#	cat.# 10-pk.
 Straight Glass Inlet Liner	general use	2.0 ID 3.0 OD x 71	5181-2036	21157
 Baffled Glass Inlet Liner	active compounds, drugs, pesticides	1.5 ID 3.0 OD x 71	5183-2037	21704
 Siltek™ Baffled Glass Inlet Liner	active compounds, drugs, pesticides	1.5 ID 3.0 OD x 71	5183-2037	21704-214.10
 Glass Inlet Liner with Wool*	large volume injections	2.0 ID 3.0 OD x 71	5183-2039	21156

**This liner is prepacked with fused silica wool. To order glass wool instead, add the suffix "-202" to the liner catalog number.*

*** Nominal ID at syringe needle expulsion point.*

**** These Uniliner® liners are for split/splitless injection ports.*

**order
prepacked
liners**

Prepacked Liners




Order liners packed with fused silica wool, fused silica beads, glass wool, or CarboFrit™ inserts by adding the appropriate suffix to the inlet liner catalog number.

Prepacked Inlet Liners Suffix Numbers				
qty.	FS Wool	FS Beads	Glass Wool	CarboFrit™
ea.	-200.1	-201.1	-202.1	-209.1
5-pk.	-200.5	-201.5	-202.5	-209.5
25-pk.	-200.25	-201.25	-202.25	-209.25





†CarboFrit™ inserts require a neck greater than 2mm.

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Direct Injection Liners for Varian GCs (for 0.32/0.53mm ID columns)

Description	Benefits/Uses:	ID**/OD & Length (mm)	Similar to Varian part #	ea.	cat.# 5-pk.	25-pk.
 Uniliner®	trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 72	-	20345	20346	-
 Cyclo-Uniliner®	trace, dirty, high MW, active samples, high linearity	4.0 ID 6.3 OD x 72	-	20347	20348	-
 Open-top Uniliner® with Wool*	trace, dirty, active samples, high recovery & linearity	4.0 ID 6.3 OD x 72	-	20845	20846	-


SPI Liners for Varian GCs (for 0.32/0.53mm ID columns)

Description	Benefits/Uses:	ID**/OD & Length (mm)	Similar to Varian part #	ea.	cat.# 5-pk.	25-pk.
 0.5mm SPI	high linearity for 0.25 & 0.32mm ID columns	0.53 ID 4.6 OD x 54	01-900109-06	20775	20776	20777
 Siltek™ 0.5mm SPI	high linearity for 0.25 & 0.32mm ID columns	0.53 ID 4.6 OD x 54	01-900109-06	20775-214.1	20776-214.5	20777-214.25
 0.8mm SPI	high linearity for 0.53mm ID columns	0.80 ID 4.6 OD x 54	01-900109-07	20778	20779	20780
 SPI with Buffer	dirty samples >1µL, for 0.25, 0.32 & 0.53mm ID columns	2.4 ID 4.6 OD x 54	01-900109-08	20850	20851	20852


Direct Injection Liners for Shimadzu GCs (for 0.32/0.53mm ID columns)

Description	Benefits/Uses:	ID**/OD & Length (mm)	Similar to Shimadzu part #	ea.	cat.# 5-pk.	25-pk.
 128mm Uniliner®	trace, active samples, high recovery & linearity	3.5 ID 5.0 OD x 128	-	20872	20873	-
 128mm Cyclo-Uniliner®	trace, dirty, high MW active samples, high linearity	3.5 ID 5.0 OD x 128	-	20874	20875	-
 99mm Uniliner®	trace, active samples, high recovery & linearity	3.5 ID 5.0 OD x 99	-	20876	20877	-
 99mm Cyclo-Uniliner®	trace, dirty, high MW active samples, high recovery & linearity	3.5 ID 5.0 OD x 99	-	20893	20894	-
 95mm Uniliner® with Wool*	trace, dirty, high MW active samples, high recovery & linearity	3.5 ID 5.0 OD x 95	-	21713	21719	-

17A PTV Liners for Shimadzu GCs

Description	Benefits/Uses:	ID**/OD & Length (mm)	Similar to Shimadzu part #	ea.	cat.# 5-pk.	25-pk.
 17A PTV Liner with Wool*	trace, dirty, high & low MW active samples	1.6 ID 4.0 OD x 95	225-09212-01	21705	21706	21707

Make-up Liners for Shimadzu GCs

Description	Benefits/Uses:	ID**/OD & Length (mm)	Similar to Shimadzu part #	ea.	cat.# 5-pk.	25-pk.
 Detector Make-up Liner	FIDs, ECDs & misc. detectors	1.0 ID 5.0 OD x 75	-	20760	20761	-






See pg. 17 for details on
Siltek™ deactivation.

This liner is prepacked with fused silica wool. To order glass wool instead, add the suffix "-202" to the liner catalog number.
***Nominal ID at syringe needle expulsion point.*

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Direct Injection Liners for PerkinElmer GCs (for 0.32/0.53mm ID columns)

Column Installs This End


DI Liners for PerkinElmer GCs (0.32/0.53mm ID)	Benefits/Uses:	ID**/OD & Length (mm)	Similar to PE part#	ea.	cat.# 5-pk.	25-pk.
 Uniliner®	trace, active samples, high recovery & linearity	3.5 ID 5.0 OD x 100	-	20855	20856	-
 Cyclo-Uniliner®	trace, dirty, active samples, high linearity	3.5 ID 5.0 OD x 100	-	20857	20858	-
 Auto SYS Open-top Uniliner® w/Wool*	trace, dirty, active samples, high recovery & linearity	4.0 ID 6.2 OD x 92.1	-	20837	20838	-
 Auto SYS Cyclo-Uniliner®	trace, dirty, high MW active samples, high linearity	4.0 ID 6.2 OD x 92.1	-	20839	20840	-
 Auto SYS Drilled Uniliner®	allows direct injection when using an EPC- equipped GC	4.0 ID 6.2 OD x 92.1	-	20819	20822	-

PTV Liners for PerkinElmer GCs

Column Installs This End

Description	Benefits/Uses:	ID**/OD & Length (mm)	Similar to PE part #	ea.	cat.# 5-pk.	25-pk.
 PTV Press-Tight®	high linearity for 0.25, 0.32, & 0.53mm ID columns	1.0 ID 2.0 OD x 88	-	20733	20734	20735
 PTV Injector	high linearity	1.0 ID 2.0 OD x 88	-	20742	20743	20744

Direct Injection Liners for 5000-6000 Series Thermo Finnigan GCs (for 0.32/0.53mm ID columns)

Description	Benefits/Uses:	ID**/OD & Length (mm)	Similar to TF part #	ea.	cat.# 5-pk.	25-pk.
 Open-top Uniliner® w/Wool*	trace, dirty, active samples, high recovery & linearity	4.0 ID 5.4 OD x 79.5	-	20841	20842	-

*This liner is prepacked with fused silica wool. To order glass wool instead, add the suffix "-202" to the liner catalog number.

**Nominal ID at syringe needle expulsion point.

**order
prepacked
liners**

Prepacked Liners

Order liners packed with fused silica wool, fused silica beads, glass wool, or CarboFrit™ inserts by adding the appropriate suffix to the inlet liner catalog number.

qty. ea.	Prepacked Inlet Liners Suffix Numbers			
	FS Wool	FS Beads	Glass Wool	CarboFrit™
5-pk.	-200.1	-201.1	-202.1	-209.1
25-pk.	-200.5	-201.5	-202.5	-209.5
	-200.25	-201.25	-202.25	-209.25

†CarboFrit™ inserts require a neck greater than 2mm.

Siltek™-Deactivated Inlet Liners

- Maximize the inertness of the sample pathway.
- Minimize breakdown.
- Low bleed.
- Thermally stable.
- “Clean and green”—manufactured without the use of harmful organic solvents.

For Siltek™ inlet liners, add the corresponding suffix number to the catalog number for your liner. Refer to pages 14-16, or to the Restek Product Guide, for liner catalog numbers.

qty.	Siltek™	Siltek™ with Siltek™ Wool	Siltek™ with CarboFrit®
each	-214.1	-213.1	-216.1
5-pk.	-214.5	-213.5	-216.5
25-pk.	-214.25	-213.25	-216.25

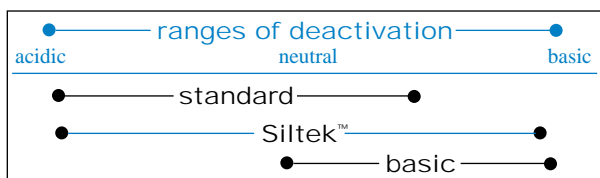
†CarboFrit™ inserts require a neck greater than 2mm.

Standard Deactivation

All Restek Liners Are Deactivated for Superior Inertness



Restek uses unique procedures for deactivating all of our liners. Each lot of liners is evaluated with an endrin breakdown test to ensure inertness. If you need a higher level of inertness for demanding applications such as pesticide analyses, try Siltek™ deactivation, described above. Our base deactivation, described below, is available for amines and basic compound analyses.

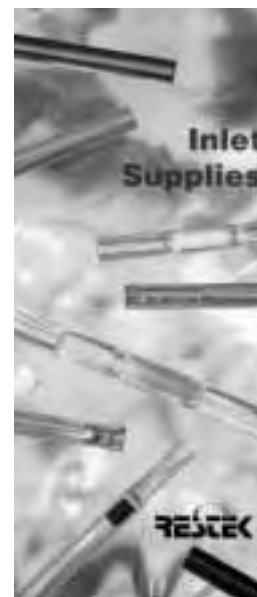


Base-Deactivated Inlet Liners for Agilent GCs

For Amines & Basic Compounds

If the liner you need is not listed here, you can order it on a custom basis by adding the appropriate suffix number. For base deactivation: each (-210.1), 5-pack (-210.5), 25-pack (-210.25). For base-deactivated liners packed with base-deactivated wool: each (-211.1), 5-pack (-211.5), 25-pack (-211.25).

ea.	5-pk.	25-pk.
	4mm Split Straight w/ Wool	
20781-211.1	20782-211.5	20783-211.25
	Cyclosplitter®	
20706-210.1	20707-210.5	—
	4mm Splitless Straight	
20772-210.1	20773-210.5	20774-210.25
	2mm Gooseneck	
20795-210.1	20796-210.5	20797-210.25
	4mm Gooseneck	
20798-210.1	20799-210.5	20800-210.25



Request the handy, pocket-sized Inlet Supplies Guide (lit. cat.# 59893A). It's a must-have for every lab.

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Restek Enhanced Design

For technical support, call
800-356-1688, ext. 4
(814-353-1300, ext. 4)

or call your local
Restek representative.

TIP!

Special dual-taper, 1/4-inch ID Vespel®/graphite ferrules fit over split liners (OD 6.3mm). Ferrules with a slightly enlarged ID are required for splitless liners (OD 6.5mm) and are available from Restek.

Restek Enhanced Split/Splitless Injection Port for Agilent 5890 GCs

Compact, locking
pin-and-slot assembly
prevents inlet lines
from snapping.

Leak-free injection
port seals at
temperatures as high
as 400°C.

Redesigned injector
base improves seal
and simplifies
column installation.

Uses standard
1/4-inch ferrule instead
of sealing disk.

Silcosteel®-treated
for an inert
sample pathway!

Original liner dimen-
sions and column
insertion distances
maintained.

Description	qty.	cat.#
Complete injection port assembly includes: base fitting, split/splitless weldment, stainless steel base screw, septum nut, 1/16" and 1/4" stainless steel nuts, 1/4" graphite ferrule Silcosteel® Injection Port for Agilent 5890 GCs	kit	22675
Ferrules for split liners (6.3mm OD): 1/4" dual-taper Vespel®/graphite ferrules	10-pk.	20290
Ferrules for splitless liners (6.5mm OD): 1/4" dual-taper Vespel®/graphite ferrule	10-pk.	20291
Silcosteel® Base Screws for Restek 5890 Injection Port	10-pk. 50-pk.	21631 21632
Gold-Plated Base Screws for Restek 5890 Injection Port	2-pk. 10-pk.	21629 21630
Septum Nut	ea.	20631
Base Fitting for Restek 5890 Injection Port	ea.	21626
Silcosteel® Split/Splitless Weldment for Restek 5890 Injection Port	ea.	22677
Silcosteel® Shell Weldment for Restek 5890 Injection Port	ea.	22678

Please note: Complete injection port assembly does not include inlet liner, 1/16" capillary ferrule, or split/splitless liner ferrules. Order separately.

Re-Threading Tool

- Repair worn or damaged threads.
- Multiple uses.
- Built-in guide to prevent cross-threading.

Description	qty.	cat.#
For Agilent split/splitless injection ports	ea.	23018

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Due to constant installation, removal, and exposure to extreme temperature changes, threads on GC parts easily become worn and damaged. This can cause a poor seal, and oxygen can enter the system, compromising analytical results and possibly destroying expensive analytical columns.



Screw the rethreading tool completely onto the injection port in a clockwise direction. Depending on the severity of thread damage, this may require force.



Unscrew the rethreading tool and inspect the threads. Repeat as necessary. When done, wipe clean with methanol to remove any debris.

Direct Replacement Split/Splitless Injection Ports for Agilent GCs

Would you like better performance from your injector? Restek's Silcosteel®-coated split/splitless injector is a direct replacement for Agilent 5890 and 6890/6850 GCs. The injector is manufactured from high-quality stainless steel and meets or exceeds Agilent original equipment specifications. Silcosteel® passivates the metal surface to ensure an inert pathway for the sample, delivering increased performance.

Direct Replacement Split/Splitless Injection Port for Agilent 5890 GCs

Description	Similar to Agilent part #	qty.	cat.#
Replacement Weldment with manual flow	19251-60575	ea.	20265
Replacement Shell Weldment	19251-80570	ea.	20266
Silcosteel® Weldment with manual flow	—	ea.	20267
Silcosteel® Shell Weldment	—	ea.	20268

Direct Replacement Split/Splitless Injection Port for Agilent 6890/6850 GCs

Description	Similar to Agilent part #	qty.	cat.#
Replacement Weldment with EPC	G1544-60575	ea.	22674
Replacement Weldment with manual flow	19251-60575	ea.	20265
Replacement Shell Weldment	G1544-80570	ea.	22673
Silcosteel® Weldment with EPC	—	ea.	22672
Silcosteel® Weldment with manual flow	—	ea.	20267
Silcosteel® Shell Weldment	—	ea.	22671

Replacement Inlet Seals for Agilent 5890/6890/6850 Split/Splitless Injection Ports

- Special grade of stainless steel that is softer and deforms more easily, ensuring a completely leak-free seal.
- Increases column lifetime because oxygen cannot permeate into the carrier gas.
- Reduced noise benefits high-sensitivity detectors (e.g., ECDs, MSDs).
- Silcosteel® seal offers the inertness of glass.
- All seals include washers.

Single-Column Installation Opening Size 0.8mm ID*		0.25/0.32mm ID Dual-Column Installation Opening Size 1.2mm ID		0.53mm ID Dual-Column Installation Opening Size (1/16-inch hole)	
2-pk.	10-pk.	2-pk.	10-pk.	2-pk.	10-pk.
Stainless Steel Inlet Seal*					
21315	21316	20390	20391	20392	20393
Gold-Plated Inlet Seal**					
21317	21318	21305	21306	—	—
Silcosteel® Inlet Seal					
21319	21320	21307	21308	—	—

*0.8mm ID stainless steel inlet seal is equivalent to Agilent part #18740-20880.

**0.8mm ID gold-plated inlet seal is equivalent to Agilent part #18740-20885.

Replacement Cross-Disk Inlet Seal for Agilent GCs

- Ideal for high-flow split applications on Agilent 5890 GCs.

(Similar to Agilent part # 5182-9652.)

0.8mm ID Cross-Disk Inlet Seal for Agilent GCs	2-pk.	10-pk.
Gold-Plated	20477	20476
Silcosteel®	20475	20474
1.2mm ID Cross-Disk Inlet Seal for Agilent GCs	2-pk.	10-pk.
Gold-Plated	21009	21010
Silcosteel®	21011	21012

All seals include washers.



Shell weldment for
Agilent 5890



Split/splitless weldment for
Agilent 5890

Septum nut not included



Weldment and shell weldment for
Agilent 6890/6850



Inlet Seals for Agilent 5890/6890
Split/Splitless Injection Port

Note: We recommend the 1.2mm inlet seal for use with VESPEL®/graphite ferrules or when installing two columns using a two-hole ferrule. Use the 0.8mm inlet seal with graphite ferrules and a single capillary column.

VespeI® Ring Inlet Seal
Reliable leak-tight seal for Agilent GCs.
Ask your Restek representative.

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Finger-Tight Column Nuts

- Allows wrench-free column installations.
- Works with standard or compact (Agilent-style) ferrules.
- Made from high-quality stainless steel.

Description	qty.	cat.#
For use with "short" Agilent-style ferrules	2-pk.	21040
For use with standard 1/16" capillary ferrules	2-pk.	21041
For use with standard 1/16" compression fittings	2-pk.	21042



Finger-Tight Nut

- Rapidly tighten columns without wrenches and avoid overtightened stripped threads.
- Two versions available—both can be used with 0.25, 0.32, or 0.53mm ID columns.

Description	qty.	cat.#
For use with "short" Agilent-style ferrules*	ea.	21311
For use with standard ferrules	ea.	21312

*Similar to Agilent part # 5020-8293 and 5020-8292, except that the Restek nut can be used with Vespel® ferrules.

Capillary Nuts (for Agilent 5890/6890 GCs)



Stainless Steel		Similar to Agilent part #	qty.	cat.#
For use with "short" Agilent-style ferrules		5181-8830	2-pk.	21884
For use with standard ferrules—redesigned to fit a wider variety of 1/16" ferrules		—	2-pk.	20883
Brass		Similar to Agilent part #	qty.	cat.#
For use with "short" Agilent-style ferrules		5181-8830	2-pk.	21878
For use with standard 1/16" -type ferrules		—	2-pk.	21879



1/16-Inch Capillary Inlet Adaptor Fitting Kit

(Split/splitless fitting for 0.25 and 0.32mm ID capillary columns)

Restek has specially engineered a high-precision, 1/16-inch fitting that uses standard size, two-hole capillary ferrules. Our design makes it easier to install capillary columns because the nut protrudes farther from the insulated injection port chamber. The column insertion depth is the same as the original equipment. The fitting kit comes with everything needed for dual-column confirmational analysis using 0.25 and 0.32mm ID capillary columns (two-hole ferrules must be ordered separately).

Description	qty.	cat.#
1/16-inch Capillary Inlet Adaptor Fitting Kit	kit	20633
Replacement Inlet Seal (1.2mm hole)	2-pk.	20390
Replacement Inlet Seal (1.2mm hole)	10-pk.	20391

See previous page for gold and Silcosteel® inlet seals.



Direct Replacement Reducing Nut

Restek offers the replacement reducing nut for Agilent 5890/6890/6850 GCs. It is made from high-quality stainless steel and meets original equipment specifications.

Description	qty.	cat.#
Reducing Nut	ea.	22078



1/8-Inch Capillary Inlet Adaptor Fitting Kit

(Split/splitless fitting for 0.53mm ID capillary columns)

Restek has specially engineered a high-precision, 1/8-inch fitting that uses standard 1/8-inch, two-hole capillary ferrules. Our design makes column installation easy because the nut protrudes farther from the insulated injection port chamber. The column insertion depth is the same as the original equipment. The fitting kit comes with everything needed for installation.

Description	qty.	cat.#
1/8-Inch Capillary Inlet Adaptor Fitting Kit	kit	20645
0.53mm ID Dual-Column Installation	2-pk.	20392
Opening Size (1/16-inch hole) Replacement Inlet Seal	10-pk.	20393

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Thermolite® Septa

- Use to 340°C inlet temperatures.
- Each batch tested on FIDs, ECDs, and MSDs to ensure lowest bleed.
- Excellent puncturability.
- Preconditioned and ready to use.
- Do not adhere to hot metal surfaces.
- Packaged in non-contaminating glass jars.



Septum Diameter	25-pk.	50-pk.	100-pk.
5mm ($\frac{3}{16}$ "	20351	20352	20353
6mm ($\frac{1}{4}$ "	20355	20356	20357
7mm	20381	20382	20383
8mm	20370	20371	—
9mm	20354	20358	20362
9.5mm ($\frac{3}{8}$ "	20359	20360	20361
10mm	20378	20379	20380
11mm ($\frac{7}{16}$ "	20363	20364	20365
11.5mm	22385	22386	22387
12.5mm ($\frac{1}{2}$ "	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Plug	20372	20373	20374

InfraRed™ Septa

- Use to 325°C inlet temperatures.
- Preconditioned and ready to use.
- Excellent puncturability.
- Do not adhere to injectors.
- Low bleed.
- Packaged in non-contaminating glass jars.

Septum Diameter	25-pk.	50-pk.	100-pk.
9mm	21417	21418	21419
9.5mm ($\frac{3}{8}$ "	21421	21422	21423
10mm	21424	21425	21426
11mm ($\frac{7}{16}$ "	21427	21428	21429
11.5mm	21430	21431	21432
12.5mm ($\frac{1}{2}$ "	21433	21434	21435
17mm	21436	21437	21438
Shimadzu Plug	21439	21440	21441

IceBlue™ Septa

- Use to 250°C inlet temperatures.
- General-purpose septa.
- Excellent puncturability.
- Preconditioned and ready to use.
- Do not adhere to hot metal surfaces.
- Packaged in non-contaminating glass jars.
- Ideal for SPME.

Septum Diameter	50-pk.	100-pk.
9mm	22381	22382
9.5mm ($\frac{3}{8}$ "	22388	22389
10mm	22390	22391
11mm ($\frac{7}{16}$ "	22392	22393
11.5mm	22383	22384
12.5mm ($\frac{1}{2}$ "	22394	22395
17mm	22396	22397
Shimadzu Plug	22398	22399

handy septum size chart

Instrument	Septum Size
Agilent (HP)	
5880A, 5890, 6890, and 6850	11mm
5700, 5880	9.5/10mm
On-Column Injection	5mm
CE Instruments (TMO)	
TRACE GC	17mm
Finnigan (TMO)	
GC 9001	9.5mm
GCQ	9.5mm
GCQ w/TRACE	17mm
QCQ™	9.5mm
TRACE 2000	9.5mm
Fisons/Carlo Erba (TMO)	
8000 series	17mm
Gow-Mac	
6890 series	11mm
All other models	9.5mm
PerkinElmer	
Sigma series	11mm
900,990	11mm
8000 series	11mm
Auto SYS	11mm
Auto SYS XL	11mm
Pye/Unicam	
All models	7mm
Shimadzu	
All models	Plug
SRI	
All models	Plug
Tracor	
540	11.5mm
550,560	9.5mm
220,222	12.5mm
Varian	
Injector type:	
Packed column	9.5/10mm
Split/splitless	
1078/1079	10/11mm
1177	9mm
1075/1077	11mm

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- Vespel®/graphite ferrules are a 60%/40% blend, offering the best combination of sealing performance and ease of workability.
- Seal with minimal torque, reusable, and preferred for vacuum and high-pressure uses.
- Stable to 400°C.

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- Stable to 450°C.

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50-packs!

Capillary Ferrules-For 1/16-Inch Compression-Type Fittings

Ferrule ID	Fits Column ID	qty.	Graphite	Vespel®/Graphite
0.3mm	< 0.20mm	10-pk.	20233	—
0.4mm	0.25/0.28mm	10-pk.	20200	20211
0.4mm	0.25/0.28mm	50-pk.	20227	20229
0.5mm	0.28/0.32mm	10-pk.	20201	20212
0.5mm	0.28/0.32mm	50-pk.	20228	20231
0.8mm	0.45/0.53mm	10-pk.	20202	20213
0.8mm	0.45/0.53mm	50-pk.	20224	20230
1.0mm	0.75mm*	10-pk.	21058	—
1.6mm	1.00mm*	10-pk.	21060	—

Compact Ferrules for Agilent 5890/6850/6890 GCs

Ferrule ID	Fits Column ID	qty.	Graphite	Vespel®/Graphite
0.4mm	0.25/0.28mm	10-pk.	20250	20238
0.4mm	0.25/0.28mm	50-pk.	20251	20239
0.5mm	0.28/0.32mm	10-pk.	21007	20248
0.5mm	0.28/0.32mm	50-pk.	21008	20249
0.8mm	0.45/0.53mm	10-pk.	20252	20263
0.8mm	0.45/0.53mm	50-pk.	20253	20264
1.0mm	1.00mm	10-pk.	21059	21056
1.6mm	1/16"	10-pk.	21061	21057

Standard Ferrules-For 1/16", 1/8", and 1/4" - Inch Fittings

Fitting Size	Ferrule ID	qty.	Graphite	Vespel®/Graphite
1/4"	3/16"	5-pk.	—	20258
1/16"	1/16"	10-pk.	20207	20218
1/8"	1/8"	10-pk.	20208	20219
1/8"	red. to 1/16"	10-pk.	20209	20220
1/4"	1/4"	10-pk.	20210	20221
1/4"	red. to 1/8"	10-pk.	20225	20222
1/4"	red. to 1/16"	10-pk.	20226	20223

Two-Hole Ferrules-For 1/8- and 1/16-Inch Compression-Type Fittings

Fitting Size	Ferrule ID	Fits Column ID	qty.	Vespel®/Graphite
1/16"	0.4mm	0.25mm	5-pk.	20241
1/16"	0.5mm	0.28/0.32mm	5-pk.	20242
1/8"	0.8mm	0.45/0.53mm	5-pk.	20246

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Reducing Ferrules

Fitting Size	Ferrule ID	Fits Column ID	qty.	Graphite	Vespel®/Graphite
1/8"	0.4mm	0.25mm	5-pk.	20205	20254
1/8"	0.5mm	0.32mm	5-pk.	20205	20255
1/8"	0.8mm	0.53mm	5-pk.	20206	20215
1/4"	0.4mm	0.25mm	5-pk.	20203	—
1/4"	0.5mm	0.32mm	5-pk.	20203	20257
1/4"	0.8mm	0.45/0.53mm	5-pk.	20204	20217

Blank Ferrules-För 1/16-Inch Fittings

Fitting Size	Ferrule ID	qty.	Vespel®/Graphite
1/16"	no hole	10-pk.	20240

Graphite Ferrules for M4 Fittings for GCQ Thermo Finnigan 8000* & TRACE™ 2000

Ferrule ID	Fits Column ID	Graphite 2-pk.	Graphite 10-pk.
0.4mm	0.18–0.25mm	20280	20281
0.5mm	0.28/0.32mm	20282	20283
0.8mm	0.45/0.50 & 0.53mm	20284	20285

Encapsulated Ferrules-För 1/16-Inch Compression Fittings

- Will not deform and stick in fittings.
- Reusable.
- Less torque needed to seal ferrule.
- Restek's unique blend of graphite provides low fragmentation and outgassing.

Ferrule ID	Fits Column ID	qty.	cat.#
0.4mm	0.25mm	10-pk.	21036
0.5mm	0.28/0.32mm	10-pk.	21037
0.8mm	0.45/0.53mm	10-pk.	21038

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- 100% Teflon®; completely inert.
- One-piece design requires no back ferrule.

Fitting Size	Ferrule ID	qty.	cat.#
1/16"	1/16"	10-pk.	21122
1/16"	0.4mm	10-pk.	21123
1/16"	0.5mm	10-pk.	21124
1/16"	0.8mm	10-pk.	21125
1/8"	1/8"	10-pk.	21126
3/16"	3/16"	10-pk.	21127
1/4"	1/4"	10-pk.	21128

HOT techtip

Choosing the Right Ferrule

Although graphite and Vespel®/graphite ferrules each have advantages and disadvantages, the choice of ferrule composition is largely a personal preference. Graphite ferrules are soft, easy to seal, stable to 450°C, and contain no binders that might off-gas. Vespel®/graphite ferrules work better for vacuum and high-pressure applications (e.g., GC/MS) because they will not allow oxygen to permeate into the system, whereas graphite ferrules will. In addition, Vespel®/graphite ferrules do not fragment, which also makes them ideal for GC/MS use. Because Vespel®/graphite ferrules are made from a harder material, they might require retightening after several temperature cycles.

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Optimizing the Analysis of Volatile Organic Compounds



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Introduction

Optimizing the Analysis of Volatile Organic Compounds

One of our standing goals is to provide you with practical technical information to help you obtain reliable data from your chromatographic and peripheral systems. This guide presents information on the common US Environmental Protection Agency (EPA) gas chromatography (GC) methods and procedures used to analyze volatile organic compounds (VOCs). It is a compilation of information based on our experience and that of experts in this field. Much of this guide is dedicated to discussing purge and trap techniques, and showing applications using a variety of configurations and conditions.

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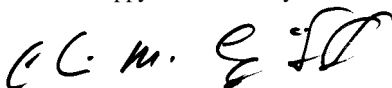
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We hope you enjoy reading this guide and find it useful in your work. If you have any questions, or have input for future editions, please don't hesitate to contact Restek Corporation - we'll be happy to hear from you.



Christopher English
Environmental Innovations Specialist

EPA Method Definitions

Many EPA methods have been developed for the analysis of VOCs. Virtually all VOC methods employ purge and trap techniques to concentrate the volatiles from the sample matrix. The type of sample matrix being analyzed determines which method is used. We will discuss drinking water methods (500 series), wastewater methods (600 series), hazardous waste methods (8000 series), and Contract Laboratory Program (CLP) methods. In addition, we will discuss state gasoline range organic (GRO) methods.

Drinking Water Methods (500 Series)

Proposed in 1973 by the EPA and passed by Congress a year later, the Safe Drinking Water Act (SDWA) establishes national standards for drinking water from surface and ground water sources. These methods regulate the analysis of trace-level organic pollutants in drinking water. Enforcement of the SDWA provides that states shall have the primary authority, while the EPA will oversee activities pertaining to the public water supply system. These methods have evolved over the years, which has resulted in a growing list of compounds of interest in the subsequent revisions.

Method 502.2: This capillary column GC method is used to monitor 60 regulated volatile contaminants in drinking water. It employs a purge and trap concentrator, combined with a photoionization detector (PID) and an electrolytic conductivity detector (ELCD) in series. The PID detects aromatic and double-bond compounds, and the ELCD detects halogenated compounds.

Method 504: This capillary column GC method is used to monitor ethylene dibromide (EDB) and dibromochloropropane (DBCP) in drinking water. It employs microextraction, using hexane, and analysis using an electron capture detector (ECD).

Method 524.2: This capillary column GC/mass spectroscopy (GC/MS) method is used to monitor the same 60 drinking water contaminants listed in Method 502.2. It also employs purge and trap concentration, but uses the MS to determine both aromatic and halogenated compounds.

Method 524.2, Revision IV: This capillary column GC/MS method is used to monitor the 60 compounds listed in Methods 524.2 and 502.2, plus 24 additional compounds. As of Fall 2001, revisions were proposed to replace hydrochloric acid sample preservation with sodium thiosulfate. These revisions, however, were not promulgated at the time of this printing.

Wastewater Methods (600 Series)

In 1977, President Carter signed the Clean Water Act (CWA) allowing the EPA to study and, if necessary, regulate 65 priority wastewater pollutants. A cooperative effort between environmental laboratories and the EPA resulted in the final version of what are now known as the 600 series methods. These methods regulate the analysis of organic pollutants in industrial and municipal wastewater discharges. They were written for packed GC columns, but most environmental laboratories now use capillary column technology.

Method 601: This GC method was developed to monitor 29 halogenated volatile pollutants in wastewater. It employs purge and trap concentration combined with an ELCD.

Method 602: This GC method was developed to monitor seven aromatic volatile pollutants in wastewater. It employs purge and trap concentration combined with a PID. Many laboratories combine Methods 601 and 602 by using a PID and an ELCD connected in series.

Method 624: This GC/MS method uses purge and trap concentration to monitor 35 halogenated and aromatic volatile pollutants in wastewater.

Method 1624: This isotope dilution GC/MS method uses purge and trap concentration to monitor 58 volatile pollutants in wastewater. Stable, isotopically labeled analogs of the target compounds are added to correct for analyte recoveries that might vary due to matrix interference in the analyzed samples.

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Drinking Water Disinfection Byproducts

1996 amendments to the SDWA require the EPA to review and revise existing National Primary Drinking Water Regulations (NPDWR) at least once every six years. Much of this renewed interest in changes to drinking water regulation standards stems from studies suggesting negative reproductive effects, such as spontaneous abortions, resulting from trihalomethanes (THMs) in water. Current studies using compliant levels of THMs in water have revealed adverse reproductive effects, therefore method detection limits (MDLs) will continue to be lowered in methods that address THMs.¹

1. S. Richardson, *Anal. Chem.* 73 (2001) 2719-2734.

Hazardous Waste Methods (8000 Series)

The Resource Conservation and Recovery Act (RCRA) of 1976 was enforced shortly after front-page headlines revealed the presence of serious hazardous waste sites like Love Canal, NY and Times Beach, MO. The analytical methods for determining hazardous waste, known as the 8000 series methods, fall under US EPA SW-846. These methods were designed for monitoring organic pollutants in waste samples prior to disposal at hazardous waste facilities. They also can be used for monitoring groundwater at these facilities.

Method 8010B: This packed column GC method is used to monitor 50 halogenated volatile pollutants in hazardous waste samples. It employs purge and trap concentration and an ELCD.

Method 8011: This capillary column GC method is used to monitor 1,2-dibromoethane (EDB) and 1,2-dibromo-3-chloropropane (DBCP) in hazardous waste samples. It employs microextraction, using hexane, and analysis using an ECD.

Method 8015A: This packed column GC method is used to monitor non-halogenated volatile pollutants in hazardous waste samples. It employs purge and trap concentration and an FID. Total petroleum hydrocarbon analysis, commonly referred to as 8015-TPH, also falls under this method. Method 8015-TPH uses an FID to match a known pattern of gasoline with an unknown sample containing peaks that fall within the gasoline pattern range. If a pattern falls within the gasoline window it may be reported as gasoline.

Method 8020A: This packed column GC method is used to monitor ten aromatic volatile pollutants in hazardous waste samples. It employs purge and trap concentration and a PID. It is common for analysts to combine Methods 8010 and 8020, by using a PID and an ELCD in series.

Method 8021A: This capillary column GC method is used to monitor 60 volatile contaminants in hazardous waste samples. It employs purge and trap concentration, combined with a PID and an ELCD in series. The PID detects aromatic compounds and double-bond compounds, and the ELCD detects halogenated compounds.

Method 8021B: Using the same analytical technique as Method 8021A, the compound list for Method 8021B includes ten additional compounds but does not require the analysis of several branched aromatics and halogenated compounds.

Method 8240B: This packed column GC/MS method is used to monitor 79 volatile pollutants in hazardous waste samples. It employs purge and trap concentration for most analytes, but direct injection can be used for some limited applications.

Method 8260B: This capillary column GC/MS method is used to monitor 98 volatile pollutants in hazardous waste samples. It employs purge and trap concentration for most analytes, but direct injection can be used for some limited applications.

State GRO Methods

Leaking underground storage tanks (LUST) pose significant environmental risks throughout the country. States have the responsibility to develop LUST testing methods. State gasoline range organics (GRO) methods are based on EPA methods such as 602, 8020 and 8015. The most common EPA method used is 8015, which relies on baseline-integrating the total area of the gasoline fingerprint, using marker compounds such as hexane (C6) and dodecane (C12). The 8015-TPH Method analysis uses an FID and pattern recognition—the specific ratio of peaks that make up a particular fuel—to identify the type of fuel. If a pattern falls within the window markers it may be reported as gasoline, then quantified. Difficult matrices can result in misidentification or poor quantitation of the sample, and deterioration in the environment (weathering) further complicates the analysis. Therefore, many states have combined EPA methods, using a PID/FID in series (e.g., Methods 8020/8015-TPH). Specific aromatic compounds are analyzed using PID (Methods 602, 8020), which is connected to the FID (Method 8015-TPH). The common target compounds are benzene, toluene, ethylbenzene, and *m*-, *o*-, and *p*-xylene (BTEX), however many states also have added other compounds to their methods (Table I).

Table I.*State gasoline methods include specific compounds.*

State	Method-Specific Compounds
Alaska (AK101AA)	BTEX, branched aromatics
Arizona	BTEX, C6-C12
California	BTEX, MTBE
California (WIP)	Method 8020, MTBE
Connecticut	GRO
Florida	PVOC
Georgia	GRO Method 8015B
Iowa (OA-1)	GRO, BTEX, MTBE
Louisiana	GRO (C6-C12)
Maryland	GRO Method 8015B
Massachusetts (VPH)	BTEX, <i>m</i> -naphthalene, MTBE, etc.
Michigan (GRO)	BTEX, <i>m</i> -naphthalene, MTBE, etc.
Mississippi	GRO
Missouri (OA-1)	GRO, BTEX, MTBE
Montana	Method 8015
New York	GRO Method 8015B
North Carolina	Massachusetts VPH
Oklahoma	GRO
Oregon	C5, C6, C8, C10, C12, BTEX, MTBE, etc.
Pennsylvania (DEP)	BTEX, MTBE, 1,2-dibromoethane, 1,2-dichloroethane
South Carolina	GRO Method 8015B
Tennessee	GRO
Texas (TNRCC 1005)	hexane, decane (locator mix)
Utah	BTEX, MTBE, naphthalene
Virginia	GRO Method 8015B
Washington (VPH)	C5, C6, C8, C10, C12, BTEX, MTBE, etc.
West Virginia	Method 8015B
Wisconsin	PVOC/GRO BTEX, MTBE, naphthalene, TMB, 1,3,5-TMB

Acronyms:

BTEX - benzene, toluene, ethylbenzene, xylenes

MTBE - methyl-*tert*-butylether

GRO - gasoline range organics

PVOC - petroleum volatile organic compounds

VPH - volatile petroleum hydrocarbons

TMB - trimethylbenzene.

**Where can EPA methods be obtained?****Drinking Water Methods (500 Series)**

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
703-487-4600

Wastewater Methods (600 Series)

Environmental Monitoring and
Support Laboratory
U.S. EPA
Cincinnati, OH 05268
513-569-7562

Hazardous Waste Methods (8000 Series)

U.S. Government Printing Office
Washington, DC 20402
202-783-3238

Websites:

U.S. EPA Homepage
www.epa.gov

Federal Register Link
www.epa.gov/fedrgstr/

Ground Water/ Drinking Water (500 Series)
www.epa.gov/safewater/

Wastewater Office (600 Series)
www.epa.gov/OWM/

Solid and Hazardous Waste (8000 Series)
www.epa.gov/epaoswer/osw/

Updated List of EPA Methods
and Web Locations
www.epa.gov/region01/oarm/testmeth.pdf

Contract Laboratory Program (CLP)

In 1980 the US Congress addressed the cleaning of the most contaminated abandoned and inactive dumpsites. This new legislation was known as the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) and the Superfund Amendments and Reauthorization Act (SARA). These acts required cleanup of the sites and the prosecution of those responsible for the contamination. The methods monitor volatile pollutants at Superfund sites.

Method OLM04.1 (04.2): The US EPA has awarded contracts for organic low-medium (OLM) concentration samples within the Superfund program under the 04.2 revision Statement of Work (SOW). This is a capillary column GC/MS method used to monitor in hazardous waste 50 volatile pollutants that fall under CERCLA and SARA guidelines. While this method employs purge and trap concentration, direct injection can be used for higher concentration samples that require extraction with methanol.

Method OLC03.2: This new EPA Statement of Work (SOW) describes analytical methods for aqueous low concentration organics. This capillary GC/MS method adds nine new volatile compounds to the OLC03.1 target compound list (TCL), for a total of 52 compounds. Deuterated Monitoring Compounds (DMC) are introduced as a sample-by-sample accuracy indicator.

The Love Canal Scandal

In the early 1900s William T. Love started work on his dream—to build a canal between the upper and lower Niagara Rivers to generate power for a planned model city. Before the canal was a mile long, the economy failed—and with it, Love's dream. Hooker Chemical purchased the land in 1920 and for the next three decades the City of Niagara, the US Army, and Hooker dumped waste into the canal. Eventually, the dump was filled and a clay cap was placed over the waste site. Soon after, the city persuaded Hooker to sell the property for \$1 with the threat of the Constitution's imminent domain clause. Although Hooker added a lengthy disclaimer to the property deed detailing the toxic nature of the site, within two years sewer lines were dug into the clay cap that had sealed the waste from leaching to the surface. In the late 1950s, about 100 homes and a school were built near the 20,000 tons of waste (Figure 1). Heavy snow and rainfall in 1975 and 1976 caused high water levels, which exposed the 55-gallon drums (Figure 2).

Figure 2.

Four decades after dumping, toxic waste drums like these were exposed at Love Canal, NY.



Niagara Gazette reporter Michael Brown broke the story, explaining that many residents were living on a toxic waste dump. From the time the families moved in during the '50s they had noticed strange odors, and in the early '70s a tar-like substance was reported in many basements. Analysis using the 8000 series methods, and later the 600 series and CLP methods, identified 248 chemicals, including 2,3,7,8-tetrachlorodibenzo-*p*-dioxin, which is believed to be the most toxic substance known to man. Many VOCs were discovered in the ground, water, and air—most notably benzene—a known carcinogen. There were no toxicological data available for 100 of the 248 compounds. On August 2, 1978 state health officials ordered all pregnant women and children under the age of two to leave the area. A week later, with headlines across the country detailing the Love Canal disaster, President Carter approved the immediate evacuation of 221 families. That number would soar to nearly 900 families by the time this tragedy completely unfolded.

This was the first environmental disaster given daily front-line media coverage. It was a turning point for environmental awareness and ultimately helped to shape the environmental testing methods that are used today for the identification of VOCs in air, water, and soil. The combined efforts of environmental laboratories, engineering firms, and regulatory agencies have evolved since Love Canal to protect the public and ultimately save lives.

Figure 1.



Infrared aerial photo of Love Canal area (spring 1978) showing 99th Street elementary school (center), two rings of homes bordering the landfill, and LaSalle Housing Development (upper right). White patchy areas are barren sections where vegetation will not grow, presumably due to leaching chemicals.

Image courtesy of State University of New York at Buffalo University Archives.

We thank Dan Di Landro, Visiting Assistant Librarian, for help with obtaining the photograph.

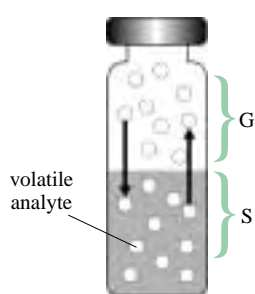
Purge and Trap Theory

Concentration of Volatile Organics

Volatile organic compounds can be concentrated by either static headspace or dynamic headspace (i.e., purge and trap) sampling. In static headspace concentration, a sample is placed in a closed sample chamber. Molecules of the volatile compounds in the sample migrate to the headspace above the sample and equilibrium is established between the concentration of the compounds in the vapor phase and in the liquid phase (Figure 3). Once equilibrium is reached, an aliquot of the headspace above the sample is injected onto the GC column. A major problem with static headspace techniques is that the sample matrix significantly affects equilibrium. Analyses for compounds that show high solubility in the sample matrix often yield low sensitivity as a result of matrix effects. Further, static headspace analysis only samples an aliquot of the volatiles (i.e., 1mL, 2mL, or whatever the size of the sample loop), which also affects sensitivity.

Figure 3.

Volatile analyte in equilibrium between the gas and sample phases.



G=gas phase (headspace)

The gas phase, commonly referred to as the headspace, is above the sample phase.

S=sample phase

The sample phase contains the compound(s) of interest, usually in the form of a liquid or solid in combination with a dilution solvent or a matrix modifier.

Once the sample phase is introduced into the vial and the vial is sealed, molecules of the volatile component(s) diffuse into the gas phase until the headspace reaches a state of equilibrium, as depicted by the arrows. An aliquot is then taken from the headspace.

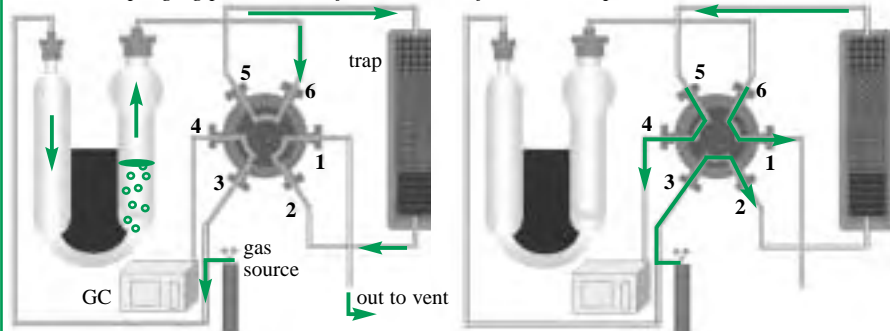


Sample purging in progress in a Tekmar 3100 concentrator.

Purge and trap concentration is a dynamic headspace technique that reduces matrix effects and increases sensitivity, relative to static headspace techniques. Samples containing VOCs are introduced into a purge vessel and a flow of inert gas is passed through the sample at a constant flow rate for a fixed time. Volatile compounds are purged from the sample into the headspace above the sample and are transferred to and concentrated on an adsorbent trap (Figure 4). After the purging process is complete, the trap is rapidly heated and backflushed with carrier gas to desorb and transfer the analytes to the GC column.

Figure 4.

The purging process transfers the VOCs from the sample to the GC column.



The purge and trap concentrator in "purge" mode. The 6-port valve allows carrier gas to bubble through the aqueous sample, transferring volatiles to the adsorbent material.

The purge and trap concentrator in "desorb" mode. VOCs concentrated on the trap are desorbed to the chromatograph for separation, identification and quantification.

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Sequences and Flow Paths of the Purge and Trap Unit

Purge and trap units are designed to have separate flow rates for the purge gas and the desorb (carrier) gas. The recommended gas for both purging and desorption is helium. The purge gas flow typically is set at 30-50mL/min. The desorb gas flow ranges from 10-80mL/min., depending on the column type and GC equipment used (see the Applications section of this guide for example chromatograms). The desorb gas should be controlled using a flow controller. The flow controller from the injection port of the GC commonly is used, but a separate flow controller can be connected to the desorb gas bulkhead fitting on the back of the purge and trap system. Hydrocarbon traps should be installed on the carrier gas line prior to the purge and trap system. This will prevent trace hydrocarbon or solvent "ghost peak" contamination from interfering with the analyses.

Purge and trap techniques involve the following series of steps that must be followed to ensure accurate and reproducible results:

Step 1. Standby

During the standby mode, the purge gas flow is stopped, the trap is cooled, and the system is readied for the start of an analysis. The desorb gas bypasses the trap and is directed onto the column as the carrier gas flow. The gas flow rate through the column can be measured.

Step 2. Purge (wet)

During the wet purge, the purge gas flow passes through the purge vessel, removes volatile analytes from the sample, and sweeps the analytes through the heated valve onto the adsorbent trap. The analytes are collected on the trap and the purge gas exits through the purge vent. The purge gas flow typically is set at 30-50mL/min. and can be measured at the purge vent. Samples usually are purged for 10-15 minutes. During the purge mode, the desorb (carrier) gas is directed onto the column.

Step 3. Purge (dry)

During the wet purge, a large amount of water is removed from the sample and collects on the trap. The dry purge removes the excess water that accumulated. During the dry purge, the purge gas bypasses the purge vessel and is directed to the trap. The dry purge gas removes water and carries it out the exit vent. The desorb (carrier) gas is directed onto the column. Only traps that incorporate hydrophobic adsorbents can be dry purged.

Step 4. Desorb Preheat

Once the analytes have been trapped and excess water removed, the purge gas flow is stopped. During this static period, the trap is rapidly heated to ~5°C below the desorb temperature of the adsorbent materials used. The desorb preheat step uniformly volatilizes the sample to create a narrow sample band and a more efficient sample transfer onto the GC column. Without a desorb preheat step the peaks would tail, resulting in poor chromatography. During the desorb preheat step the desorb (carrier) gas is directed onto the column.

Step 2.

Purge (wet)

- Volatiles in matrix diffuse into carrier gas as gas is bubbled (purged) through the matrix. Volatiles are transferred to the trap.
- Typical flow: 30–50mL/min. for 10–15 min.



Step 3.

Purge (dry)

- Trap is dried by purging with gas only.
- Typical time: 1-4min.



Step 4.

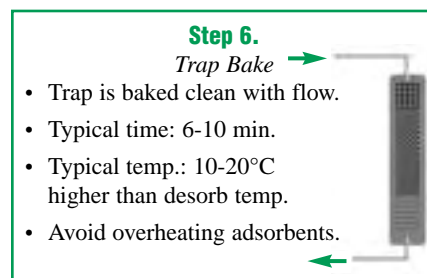
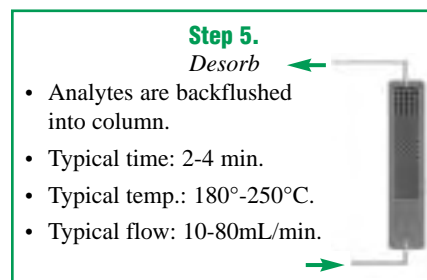
Desorb Preheat

- Trap is heated without flow, to minimize analyte desorb time from packing material.
- Typical temp.: 5°C below desorb temp.



Step 5. Desorb

Once the desorb preheat temperature is reached, the purge and trap unit valve is rotated. This directs the desorb (carrier) gas flow to backflush the adsorbent trap and carry the analytes in a narrow band to the GC system. Figure 4 (page 7) shows the flow path of the desorb mode. While the sample transfer occurs, the trap is heated to its final desorb temperature. Desorb temperatures range from 180°C-250°C, determined by the adsorbent materials and the model of concentrator. The desorb flow rate is extremely important; it must be high enough to ensure that the sample remains in a narrow band during the transfer to the GC column. The optimum desorb flow rate for a purge and trap system is >20mL/min.; however, this flow rate is too high to use with capillary columns and must be reduced to retain column efficiency. The optimum flow rate for 0.53mm ID columns is 8-10mL/min. For narrow bore capillary columns (0.18-0.32mm ID), the desorb flow rate usually is 1-2mL/min. when direct interface is used. This low flow rate requires a longer desorb time due to the slow transfer of the sample from the trap, which, in turn, creates a wide sample bandwidth resulting in broad peak shapes for all early eluting compounds. Cryofocusing (i.e., cold trapping) can be used to reduce band broadening, by installing a secondary cold trap or by cooling the GC column to subambient temperatures. The desorb time is inversely proportional to flow rate and trap temperature, so that as the flow rate/trap temperature increases, the desorb time decreases, due to the analytes flushing off the trap at a higher rate. Also, it is possible to desorb at higher flow rates (25-80mL/min.), when using narrow bore capillary columns, by using a split injector to split the flow prior to the column (for more details on this technique see the GC System Configurations section, page 18).



Step 6. Trap Bake

After the desorb step the trap is baked, with gas flow, to remove any remaining sample components and contaminants from the trap in preparation for its next use. This step generally lasts 6-10 minutes; typical temperatures are 10-20°C above the desorb temperature. To prevent damage to the adsorbent materials, do not exceed the maximum temperature of the trap.

Purge and Trap Components

Purge Vessel

Three types of purge vessels (i.e., spargers) commonly are used in purge and trap systems. Frit spargers (Figure 5) are used for most water samples. The frit creates many small bubbles that travel through the sample to increase purging efficiency. Fritless spargers are used for samples that have high particulate content, or for industrial wastewater samples that may foam. They create fewer bubbles, which decreases purging efficiency but eliminates plugged frits and reduces foaming problems. Needle spargers are used when purging soil, sludge or solid samples. A narrow gauge needle is inserted into the sample and used to release a small stream of purge gas. The two common sizes of spargers are 5mL and 25mL.

Figure 5.

Purge and trap frit spargers.



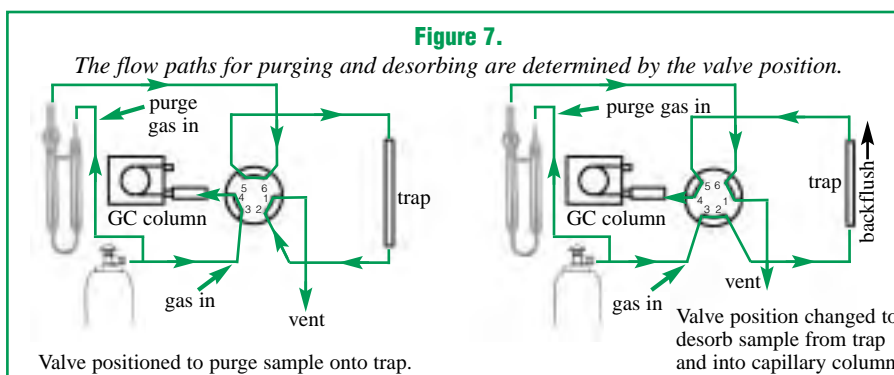
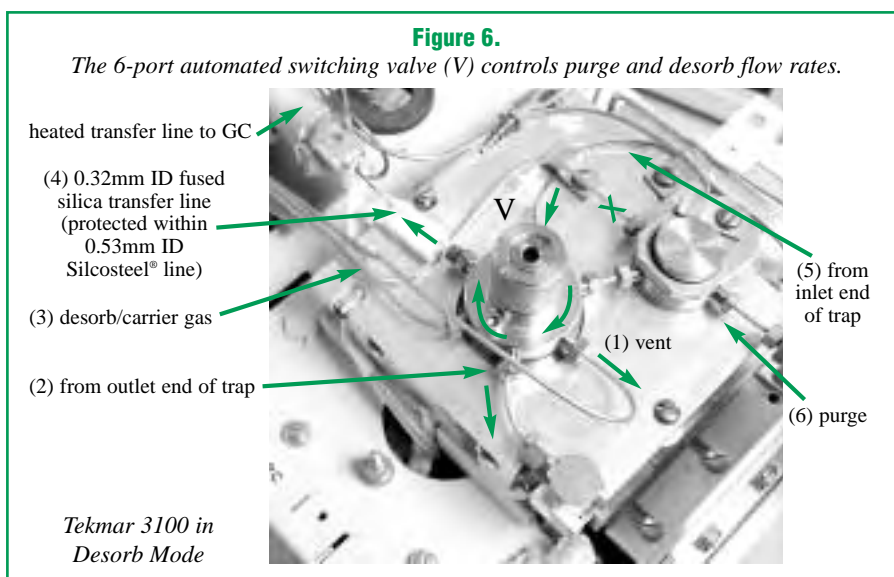
- For Tekmar 2000, 3000, or 3100.
- Available in 5mL and 25mL sizes.
- Uniform frits ensure maximum purge efficiency for water samples.
- Manufactured to tight tolerances to ensure a leak-free seal.

Description	qty.	cat.#
5mL Fritted Sparger, 1/2-inch mount	ea.	21150
25mL Fritted Sparger, 1/2-inch mount	ea.	21151

Not recommended for wastewater samples because the sample might foam or the frit might become plugged.

Valves

The purge and desorb flows are controlled by an automated switching valve (Figure 6). The valve is contained in a heated compartment to prevent sample condensation inside. By rotating the valve, the purge and desorb flow paths can be changed during the purge and trap sequence (Figure 7).



Adsorbent Materials and Traps

Adsorbent materials are used to trap the VOCs that have been purged from the sample. The adsorbent must be able to retain compounds during the entire purging sequence and then rapidly release them during the desorption step. Each adsorbent has a unique trapping capability for a specific class or classes of compounds. Therefore, a trap may have several different beds of adsorbents. The weakest adsorbent material is placed at the inlet end of the trap, then the next strongest adsorbent, and so on. The more volatile compounds pass through the weaker adsorbents and are retained by the stronger adsorbents, while the less volatile compounds are retained on the weaker adsorbents and never reach the stronger adsorbents (from which they would be difficult to desorb). Once the compounds are collected, the trap is rapidly heated and backflushed with carrier gas to drive the sample components into the GC system. Ideally, the adsorbents in the trap retain polar and non-polar analytes without retaining water or methanol, efficiently release the trapped compounds onto the analytical column, and withstand the temperatures required to desorb (i.e., “bake off”) the higher molecular weight contaminants. A list of common traps used in purge and trap concentration can help you choose the best one for your application (Table II, page 13).

Adsorbent Materials

Tenax® Adsorbent (surface area: 50m²/g): Tenax® adsorbent is excellent for trapping non-polar compounds and is hydrophobic so it does not retain water; however, it does have some disadvantages. Very volatile compounds are not retained well and must be trapped on a stronger adsorbent material. In addition, polar compounds like alcohols are poorly retained on this adsorbent. Tenax® adsorbent also has limited thermal stability; the 2,6-diphenyleneoxide polymer thermally decomposes into toluene, benzene, and other aromatics. The particles melt together and permanently adhere to the trap; this then restricts carrier gas flow. As the adsorbent degrades, there often is a loss in response for brominated compounds.

There are two grades of Tenax® adsorbent used as a trapping material: Tenax® GC and Tenax® TA (Trapping Agent) adsorbents. Common background contaminants in Tenax® GC adsorbent include benzene and toluene. Tenax® TA adsorbent is a purer form and is more commonly recommended for thermal desorption applications. The manufacturer's recommended operating temperature is 230°C but, realistically, the material performs best when kept below 200°C. Samples that contain organic acids can degrade Tenax® adsorbent. This effect is more pronounced at higher temperatures; for longer trap life and better consistency do not use traps containing this adsorbent at temperatures above 200°C.

Silica Gel (surface area: 200-800m²/g): Silica gel is a stronger adsorbent than Tenax® adsorbent. Silica gel is commonly used in conjunction with Tenax® adsorbent as a trap for volatile organic pollutants. It is an excellent trapping material for polar and highly volatile compounds that are gases at room temperature; however, silica gel is extremely hydrophilic and will retain large amounts of water. *Be aware that if a trap contains silica gel, dry purging will not reduce the water content.*

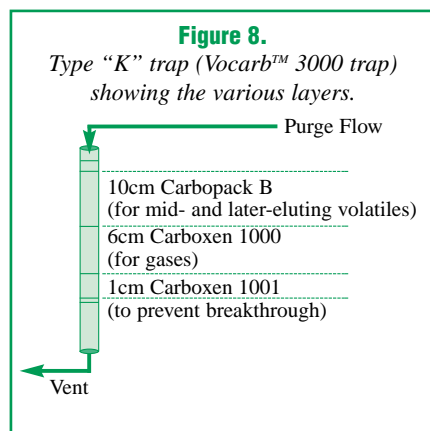
Coconut Charcoal (surface area: 900m²/g): Coconut charcoal is another strong adsorbent material. It is commonly used in series after silica gel for trapping very volatile compounds that might break through the gel. Coconut charcoal is hydrophobic, and does not retain significant amounts of water. It does, however, trap carbon dioxide (CO₂) purged from the sample, and it has been reported that charcoal is a source of CO₂, which can interfere with the quantitation of early-eluting compounds when using GC/MS systems.

Graphitized Carbon Black or Carboxen® Adsorbent (surface area: 10-100m²/g): Graphitized carbon black (GCB) is an alternative to Tenax® adsorbent. GCB is available in many pore sizes and is effective in trapping volatile organics in the same range as Tenax® adsorbent. GCB is hydrophobic and has excellent thermal stability, making it ideal for purge and trap techniques. Highly volatile compounds are not retained well on GCB and must be trapped on stronger adsorbent materials such as carbon molecular sieves.

Carbon Molecular Sieves (surface area: 50-800m²/g): Carbon molecular sieves such as Carbosieve™-SIII are alternatives to silica gel and charcoal. High surface areas make these materials ideal for trapping highly volatile compounds. They are commonly used in series after GCB because they retain compounds that break through the GCB. Carbon molecular sieves are hydrophobic and have excellent thermal stability.

Carboxen®-1000 Adsorbent (surface area: 1200m²/g): Carboxen®-1000 adsorbent is a strong adsorbent designed to be used as the innermost adsorbent bed in the trap (Figure 8, page 12). This material traps Freon® compounds, permanent gases, and light hydrocarbons. It has characteristics very similar to those of Carbosieve® S-III packing material. Carboxen®-1000 adsorbent is stable to temperatures of 300°C. Its only shortcoming is the adsorption of CO₂, which can interfere with early-eluting compounds.² Carboxen®-1001 and Carboxen®-1002 are similar materials.

2. Mosesman, N.H., W.R. Betz, and S.D. Corman. "Alternate Trapping Materials for Purge-and-Trap Analysis of Volatile Compounds." *Proc.-Water Qual. Technol. Conf. Adv. Water Anal. Treat.* 14 (1987): 245-50.



Choosing the Right Trap for Your Analysis

Type “K” Trap (Vocarb™ 3000 Trap): The most effective trap on the market is the Vocarb™ 3000 or type “K” trap (Figure 8). This trap has exceptional ability to retain highly volatile compounds like difluorodichloromethane with minimal bleed, activity, or break-down, yet it works well for trapping higher boiling compounds like naphthalene and trichlorobenzene. The trap resists adsorption of water and methanol, and virtually eliminates the need for moisture control systems (MCS) and the dry purge step on the concentrator. Because this trap contains Carboxen™ 1000 adsorbent (described on page 11), which has a surface area of over 1200m²/g, a desorb temperature of 245°C is required when using Tekmar purge and trap instruments. For OI Analytical sample concentrators, such as the Model 4560, the desorb temperature should be 220°C or lower. The lower temperature will prevent overshooting the maximum temperature of the trap, which would damage the packing materials (caused by the rapid trap heating rate, 800°C/min., of the OI system).³ When using this trap be sure to verify performance. Non-linear response for chloromethane is a sign of breakthrough and an indication that the trap must be changed. Another indication of a defective “K” trap is loss in response for acrolein.

Type “J” Trap (BTEXTRAP™ Trap): The “J” trap is excellent for concentrating gasoline range organics (GRO) because it retains less water and methanol compared to the “K” trap, and can withstand higher temperatures than the Tenax®/silica gel trap. Because many GRO samples have high concentrations of gasoline components, it is necessary to dilute the sample in methanol, and this trap can accept a heavy sample load with percent levels of methanol while still passing continuing calibration check criteria. The disadvantage of the “J” trap is its limited ability to retain more polar analytes like the ethers and alcohols. Laboratories analyzing for *tert*-butyl alcohol will attain lower detection limits by using the “K” trap, compared to the “J” trap. For GRO samples containing methyl-*tert*-butyl ether (MTBE), trap selection will depend on the sample matrix. When analyzing highly contaminated soils for MTBE, it is best to use the “J” trap. For cleaner samples, the “K” trap provides better sensitivity.

Type “B” Trap (Tenax®/Silica Gel Traps): Tenax®/silica gel traps are used for a variety of VOC methods. These traps exhibit better recoveries of polar analytes than the “K” trap, but the silica gel layer adsorbs water, methanol, and carbon dioxide. The Tenax®/silica gel trap also has better lot-to-lot reproducibility compared to the “K” or “I” traps. For laboratories that are not trying to achieve MDLs for gaseous VOCs at concentrations above 10ppb, these traps will work well. To achieve detection limits for gases at concentrations below 10ppb, the lower water and methanol retention of the “K” trap is recommended.

Type “F” Trap (OV®-1 /Tenax®/Silica Gel Traps): Although these traps are recommended in many EPA methods, they exhibit more bleed and activity than the Tenax®/silica gel trap, with no significant improvement in performance. This suggests the bleed originates from the OV-1 (methyl silicone) material.⁴ Therefore, laboratories wishing to adhere more closely to the EPA Method Protocol should choose Tenax®/silica gel traps without OV®-1.

Type “I” trap (Vocarb 4000™): The “I” trap is used for increased response for less volatile compounds such as the chloronaphthalenes and methylnaphthalenes. Generally, it is used only for applications involving analytes of larger molecular size and is not the first choice for ketones or alcohols. Common desorb times of two to four minutes should be increased with the “I” trap, to optimize sensitivity for compounds having high boiling points.

3. OI Analytical, “Volatile Organics Analysis: Building a State-of-the-Art Purge and Trap GC/MS system” Application Note 02971294.

4. OI Analytical, “Proper Trap Selection for the OI Analytical Model 4460A Purge and Trap Sample Concentrator” Application Note 12851098.

Table II.
Compositions and characteristics of common types of traps.

Description	Trap Designation	Dry Purge	Preheat (°C)	Desorb (°C)	Bake (°C)
24cm Tenax®	A	yes	175	180	200
15cm Tenax®/8cm silica gel	B	no	175	180	200
8cm Tenax®/7.7cm silica gel/7.7cm charcoal	C	no	175	180	200
16cm Tenax®/7.7cm charcoal	D	yes	175	180	200
1cm OV®-1/7.7cm Tenax®/7.7cm silica gel/ 7.7cm charcoal	E	no	175	180	200
1cm OV®-1/15cm Tenax®/7.7cm silica gel	F	no	175	180	200
1cm OV®-1/ 23cm Tenax®	G	yes	245	250	260
7.6cm Carboxen® B/1.3cm Carboxen® S-III	H	yes	245	250	260
8.5cm Carboxen® C/10cm Carboxen® B/ 6cm Carboxen® 1000/1cm Carboxen® 1001	I (Vocarb™ 4000)	yes	245	250	260
7.7cm Carboxen® C/1.2cm Carboxen® B	J (BTEXTRAP™)	yes	245	250	260
10cm Carboxen® B/6cm Carboxen® 1000/ 1cm Carboxen® 1001	K (Vocarb™ 3000)	yes	245	250	260

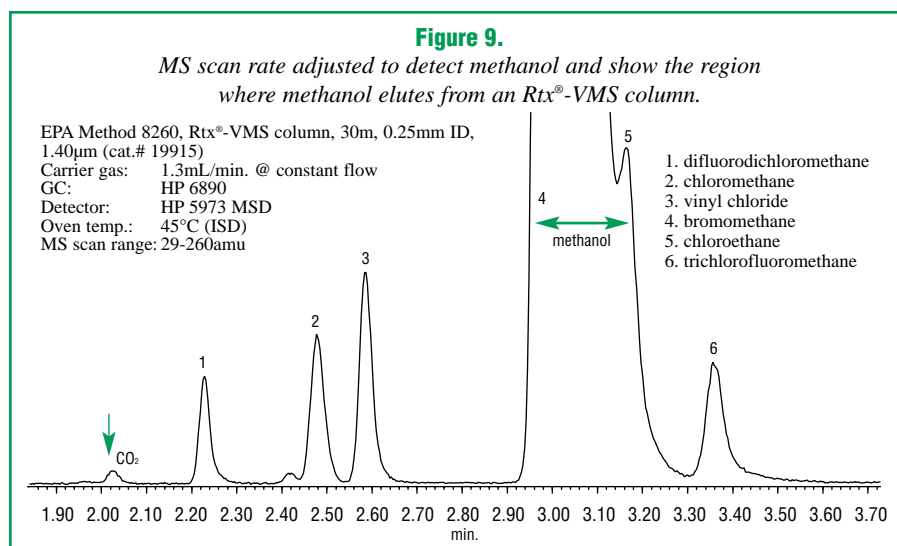
Moisture Control Systems—Water and Methanol Management

Water and methanol can cause the biggest problems in purge and trap concentration. During the desorb step, water and methanol that accumulated on the trap are released into the chromatographic system. As much as 10µL of water can accumulate on a trap containing silica gel during a purge; this expands to 12mL of water vapor during desorption.⁵ Interference caused by excess water is a problem during detection. For example, water vapor passing through a PID can cause a negative dip in the baseline. Water also can saturate a PID, decreasing its sensitivity and interfering with the identification of compounds that coelute with water. Detector saturation also can occur with MS systems. Although the lower end of the scan range typically is adjusted above the molecular weight of water, interference can still occur. If the water plug is very large, the peaks for analytes that elute in the water/methanol region will broaden and sensitivity will be reduced.

In similar fashion, methanol also causes interferences with target analytes. The PID gives a positive signal for methanol as a broad, flat-topped peak that usually interferes with 2-methylpentane, 3-methylpentane, and vinyl chloride. Adjusting the MS scan range to start above 35amu can minimize the effects of methanol (mass/charge ratio of 31amu). When using an Rtx®-VMS column, or a cyanopropylphenyl “624”-type column, methanol and chloroethane will elute simultaneously. This can affect sensitivity and linearity for chloroethane (Figure 9). When using a “502.2” phase column, methanol coelutes with bromomethane. Spiking higher concentration intermediate standards into the purge vessel or autosampler vials can minimize methanol interference. Also note that an increase in methanol added to the purge standard also inevitably increases the amount of water that purges into the system.

In recent years, much work has gone into developing hydrophobic adsorbents that minimize water collection on the trap. Extensive studies recommend incorporating a dry purge cycle to remove water from the trap prior to desorption. Current designs of purge and trap systems have added features to eliminate water prior to delivering the sample to the chromatographic system. Moisture control systems (MCS) remove water by condensation, prior to the desorb step. Such systems typically are composed of a piece of metal tubing that is heated during purge and then cooled to 30°C. The sample, desorbed from the heated trap, travels through the MCS, where a large portion of the water is condensed from the saturated carrier gas. These systems are very effective for GC methods that do not have polar/active compounds, such as ketones, in the analyte list. An older purge and trap system that does not have an MCS can be retrofitted with one. Restek offers an MCS bypass line for Tekmar 3000 and 3100 purge and trap concentrators, to increase response and maintain linearity for ketones, alcohols, and acetates (Figure 10, page 14). When analyzing samples for ketones or other polar compounds, the MCS should be bypassed to maintain linear calibration for these compounds.

5. OI Analytical, “OI Analytical Model 4560 Sample Concentrator Rapid Trap Heating”
Application Note 04521297.



Transfer Line

Once the sample is desorbed from the trap, it travels through the heated transfer line to the GC. This line can be made of nickel, fused silica, or Silcosteel®-treated tubing. A heating jacket surrounds the transfer line to keep it between 120-125°C, which prevents water and analyte condensation in the line. For direct connection, we recommend matching the inside diameter of the transfer line to the inside diameter of the capillary GC column, or use of tubing of a slightly smaller inner diameter than the capillary column. This helps minimize band broadening and poor peak symmetry for sample components. Because transfer lines can be a source of active sites, use deactivated fused silica or Silcosteel®-treated tubing to reduce analyte adsorption. When using a fused silica transfer line, insert the line into a metal tube before installing it into the heated jacket. This will protect the fused silica tubing from nicks and scratches that could cause the line to break. Be sure to use the correct Valco® ferrules to minimize dead volume (see Direct Connection, page 20).

Figure 10.

An MCS bypass line can increase response and maintain linearity for ketones, alcohols, and acetates.

Moisture Control Bypass Line for Tekmar 3000 Purge & Trap

- Increases response for ketones, alcohols, and acetates.
- Suitable for US EPA Methods 8260, 524.2, and OLM4.1.
- Silcosteel® tubing for increased inertness.
- Easily attaches in minutes.

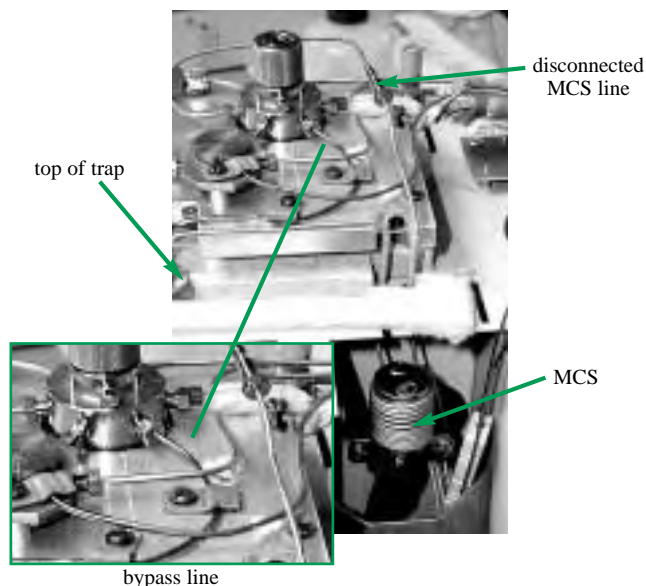


Description

Moisture Control Bypass Line

qty.
ea.

cat.#
21035



Troubleshooting Common Problems Associated with Purge and Trap Units

Water: The most common problem with VOC analysis is water in the sample. Water does not create problems with the purge and trap system, but it will create problems with the GC system. A large quantity of water can quench the PID response, causing a negative dip in the baseline. Water also can saturate the MS and create interference with early eluting gases. Analysts using an MS can observe the water band eluting from the column as a large baseline rise. Since VOC samples typically are aqueous or contain large amounts of water, water vapor will be purged along with the target compounds. Although water cannot be completely prevented from collecting on the trap, the amount transferred to the GC system can be minimized by using a trap that contains hydrophobic adsorbents (see Adsorbent Materials and Traps, page 10). A dry purge step also may remove water from the adsorbent surface (see Sequences and Flow Paths, page 8). Many new purge and trap systems employ water management to minimize the amount of water reaching the GC system, but be aware that these systems can have adverse effects on the recovery of polar compounds such as ketones (page 13). Many laboratories purge 5mL-10mL of sample in 25mL purge vessels (see photo); water condenses on the inner wall of the vessel, reducing the amount of moisture that ultimately gets onto the trap.



Purging a 5mL sample in a 25mL purge vessel, to reduce water transfer to the trap.

Leaks and Active Sites: Another common problem in purge and trap systems is reduced sensitivity caused by leaks or active sites in the system. Reduced sensitivity for all compounds normally indicates a leak. To test for leaks in the purge and trap system, perform a pressure decay test by capping off the purge vent during the purge cycle. The bubbles passing through the purge vessel should stop within 2 to 10 minutes. If the bubbles do not stop, there is a leak in the purge system. To locate the leak, use a leak detector. Start checking for leaks at the purge vessel and work back to the inlet line on the back of the instrument. Leaks most commonly occur at the purge vessel and at the trap fittings.

Reduced sensitivity for specific compounds usually indicates the presence of active sites in the system. Poor response for bromoform or other brominated compounds is a good indicator of active sites in the purge and trap unit or transfer line. However, poor bromoform response also can be caused by high transfer line temperatures ($>130^{\circ}\text{C}$). Reduce the transfer line temperature and determine if bromoform recoveries improve. Another component that decomposes due to active sites is 1,1,2,2-tetrachloroethane (Figure 11).⁶ To reduce or eliminate sources of activity, clean or replace sources of contamination, including internal gas lines and the transfer line. Inert Silcosteel[®]-treated tubing is an excellent choice for re-plumbing purge and trap gas lines. Tekmar's newest purge and trap concentrator, Model 3100, incorporates Silcosteel[®] treatment on all tubing and internal surfaces.

Ghost Peaks: Ghost peaks typically are caused by carryover from sample components that collect within the purge and trap system. This problem is most common when performing total petroleum hydrocarbon (TPH) analysis because these samples often contain high molecular weight components. If the valve oven and transfer line temperatures are set too low, high molecular weight compounds can condense in the line, then bleed onto the column. To eliminate ghost peaks, temporarily increase the purge and trap valve oven and transfer line temperatures to bake out the contaminants. The heated mount feature on some purge and trap instruments can reduce carryover by up to 50%, but this also will increase the amount of moisture entering the trap. The standard mount temperature is 40°C ; increasing the temperature to 70°C significantly reduces sample carryover. For severe contamination, steam cleaning or methanol rinsing can be performed (see instructions on page 11). Unlike in other cleaning procedures, here we do not recommend using water after methanol rinsing because it is very difficult to remove water from the purge and trap system. Ghost peaks also are caused by adsorbent contamination or degradation. Tenax[®] can break down to toluene, benzene, styrene, naphthalene, and other aromatic compounds (see Adsorbent Materials and Traps, page 10). This normally is an indication of trap overheating. To prevent this problem, do not expose a trap containing Tenax[®] adsorbent to temperatures above 200°C .

6. Tekmar-Dohrmann, *Purge and Trap Concentrator Course*, 1989. Cincinnati, Ohio.

EPA Update

The US EPA promulgated update III of Test Methods for Evaluating Solid Waste (SW-846). This 1997 update deleted the previous EPA purge and trap Method 5030A, "Sample Preparation of Volatile Organic Compounds for Purge and Trap Analysis" and replaced it with Method 5035, "Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples." Method 5035 involves extensive fieldwork and raises MDLs for soil samples; however, accuracy is improved.

Previously, soil samples were collected using Teflon®-lined screw-cap containers and stored at 4°C, with a 14-day maximum holding time. Once the samples were in the laboratory, 5g aliquots of soil were added to 5mL of reverse osmosis (RO) water. The volatiles in these samples exit the soil matrix and leak from the container. Method 5035 requires samples to be collected and preserved in the field at the time of sampling, using methanol and a stir-bar. Volatiles dissolved in the methanol are less likely to escape. The seal is not broken until the time of analysis, thus minimizing analyte loss through evaporative mechanisms. Sodium bisulfate is used to prevent biodegradation of VOCs. Unlike HCl preservation, sodium bisulfate does not break down 2-chloroethyl-vinyl-ether. This greatly improves the accuracy of analytical results from soil samples because evaporative loss occurs almost immediately in soils that are not preserved in methanol. Disadvantages include the higher detection limits and the problems associated with purging higher percentages of methanol.



Flushing the trap attachment area with methanol. Repeat several times.

Figure 11.
No measurable response for bromoform (9), combined with a greatly diminished response for 1,1,2,2-tetrachloroethane (10), strongly indicates a contaminated transfer line.

20m, 0.18mm ID, 1.0µm Rtx®-502.2 column (cat.# 40914), 4ppb of VOA standards.

Oven temp.: 35°C (hold 5 min.) to 180°C @ 6°C/min.
to 210°C @ 20°C/min. (hold 5 min.)

Inj. / det. temp.: 100°C / 280°C

Linear velocity: 20cm/sec. set @ 35°C

Purge & trap: Tekmar 3000

Purge: 11 min.

Trap pressure control: 6psi

Desorb preheat: 250°C

Desorb time: 2 min.

Detector: MS

Split ratio: 40:1

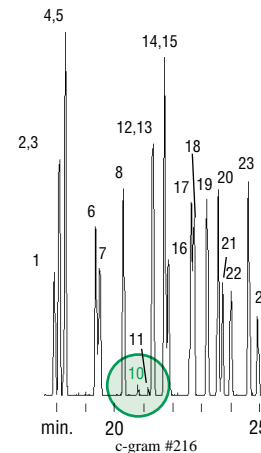
Scan range: 35-260AMU

Trap: Vocab™ 3000

Desorb temp.: 260°C

Desorb flow rate: 20mL/min.

1. chlorobenzene	13. bromobenzene
2. ethylbenzene	14. 1,3,5-trimethylbenzene
3. 1,1,1,2-tetrachloroethane	15. 2-chlorotoluene
4. <i>m</i> -xylene	16. 4-chlorotoluene
5. <i>p</i> -xylene	17. <i>tert</i> -butylbenzene
6. <i>o</i> -xylene	18. 1,2,4-trimethylbenzene
7. styrene	19. <i>sec</i> -butylbenzene
8. isopropylbenzene	20. <i>p</i> -isopropyltoluene
9. bromoform (not detected)	21. 1,3-dichlorobenzene
10. 1,1,2,2-tetrachloroethane	22. 1,4-dichlorobenzene
11. 1,2,3-trichloropropane	23. <i>n</i> -butylbenzene
12. propylbenzene	24. 1,2-dichlorobenzene



Permission to publish this chromatogram
granted by Anne Williams, Tekmar Company.

Instructions for Cleaning Purge and Trap Concentrators

We developed these instructions using Tekmar LSC 2000 and 3000 concentrators. Always remember to use safety glasses when working in the laboratory.

1. Keep the instrument power on and turn the line heaters off. Set all temperatures to the off position. WAIT UNTIL HEATED ZONES HAVE COOLED.
2. Make sure the unit is in standby mode.
3. Disconnect the purge and trap vessel.
4. Flush methanol into the area where the top of the trap attaches, using a 5mL syringe without a needle (i.e., a Luer-lock syringe - see photo). This is the area where the purge vessel attaches to the purge and trap. You should see methanol coming out of the mount.
5. Clean the mount, using a tissue. The mount is either nickel- or gold-plated, so be careful not to scratch the surface. If you cannot clean the mount, it may need to be replaced.
6. Clean the purge vessel with methanol, then with ultra-pure water. Do not use soap. You may use a brush.
7. Increase purge and trap temperatures to normal operating conditions and hold for two hours, with no trap or purge vessel installed.
8. Install an empty trap. Do not use an old trap with the packing removed; particles of trapping material may end up in the concentrator. If an empty trap is not available, refer to the next paragraph. With the empty trap in place, attach all lines, including the line to the purge vessel. Desorb for at least one hour with the transfer line disconnected from the column. This will help to drive any methanol remaining from step 6 out of the system.

If you do not have an empty trap, disconnect the transfer line from the column, connect the purge vessel and all lines, and install an old trap. Desorb for one hour.
9. Install and condition a new trap and run blanks until a clean baseline is achieved.

If you are still having activity problems after following this procedure, please contact the Restek Technical Service Team via email at support@restekcorp.com or via phone at 800-356-1688 or 814-353-1300, ext. 4.

Broad Peaks: Peak broadening is another problem often experienced when analyzing VOCs by purge and trap methods. Broad peaks are caused either by poor sample transfer from the purge and trap to the GC or by dead volume within either system. Broad peaks frequently result from dead volume in the connection between the purge and trap unit and the GC system. (See pages 18-21 for connection methods.) Because trap desorption is a relatively slow process, the sample band transferred to the GC can be very wide. To reduce this bandwidth, the sample must be transferred quickly and/or refocused at the inlet of the GC column through a secondary cold trapping technique such as cryofocusing or subambient cooling. The transfer time can be reduced by using the desorb preheat feature. During this step, the trap is heated to 5°C below the desorption temperature, and the valve is positioned so no flow passes through the trap. This helps the compounds trapped on the adsorbents to rapidly migrate from the trap when backflushing begins.

The desorb flow rate also will affect the bandwidth. If the desorb flow is too low (<9mL/min.), the band becomes broad (Figure 12) and must be refocused at the column inlet. If faster flow rates are used (>9mL/min.) in conjunction with long, thick-film columns, the bandwidth can be reduced enough so that secondary trapping is not required. Ideally, desorbing at a flow rate of 20-30mL/min. yields a very narrow bandwidth. However, when using narrow bore columns, it might be necessary to split the flow at the injection port to maintain column efficiency.

Foaming Samples: Analysts deal with foaming samples in two primary ways: by dilution or by addition of an anti-foaming agent. Diluting the sample compromises the detection limit, but in the end may save instrument downtime. Anti-foaming agents such as polydimethylsiloxane and silicon dioxide methylcellulose are designed to reduce foaming of surfactants in a liquid matrix. These are effective at preventing a sample from foaming, but they generally produce artifact peaks that can interfere with the target analytes. An anti-foam blank must be run prior to samples to determine the contribution of artifact peaks from the anti-foaming agent. If dilution or anti-foaming agents do not reduce foaming or if samples have not been screened for surfactants, use a 5 or 10mL sample in a 25mL purge vessel to prevent the bubbles from entering the fittings and, ultimately, the trap. If you are running an unattended autosampler, you can insert a plug of deactivated fused silica or glass wool into the top of the purge vessel to prevent foam from entering the purge and trap lines. If all else fails consider switching to a fritless sparge tube and increasing the purge time to effectively remove the volatile analytes.



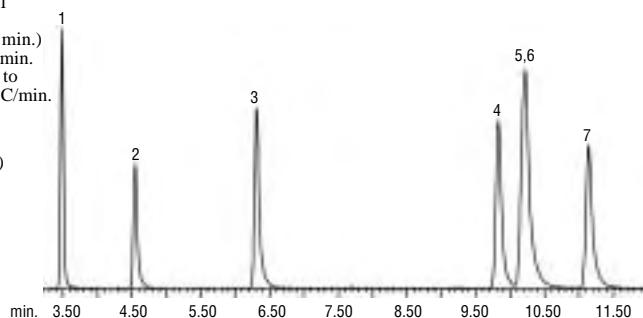
Wastewater samples commonly contain surfactants and other material that can contaminate the concentrator.

Figure 12.

A low desorb flow can produce tailing peaks, as in this example, desorbed at 9mL/min.

EPA Method 8020, Rtx®-5Si MS column, 40m, 0.45mm ID, 1.5µm (cat. #12798)
 Carrier gas: 9mL/min. @ constant pressure
 GC: Finnigan 9001
 Detector: FID
 Oven temp.: 40°C (hold 2 min.)
 85°C @ 4°C/min.
 (hold 1 min.) to
 225°C @ 40°C/min.
 (hold 2 min.)

1. benzene
2. α,α,α-trifluorotoluene (SS)
3. toluene
4. ethylbenzene
5. m-xylene
6. p-xylene
7. o-xylene



GC System Configurations

Wide-bore Systems (0.45mm ID and 0.53mm ID columns)

Wide-bore capillary columns are operated at faster flow rates than narrow-bore columns, and can be connected directly to the purge and trap system with a 10mL/min. desorb flow. Wide-bore columns used for VOCs analyses usually are coated with a thick film of stationary phase to increase retention and separation of the highly volatile analytes (e.g., chloromethane and vinyl chloride, bromomethane and chloroethane), or other closely-eluting sample components.

Wide-bore columns range from 30-105 meters in length. A longer column can refocus early-eluting volatile compounds and greatly improve separation of the gases (see Applications, page 37). Shorter columns require sub-ambient cooling for separating the gases; this increases the cost of the analysis and adds laboratory time associated with handling tanks of liquid nitrogen. For best overall results, we recommend using a 75m, 0.45mm ID capillary column for analyzing the volatile compounds listed in US EPA Methods 502.2 and 8021B (see Applications, page 37).

Resolution of the early-eluting gaseous analytes increases significantly with a decrease in temperature. Use a starting temperature of 35°C-50°C, depending on the target list and the purge and trap conditions (see Applications, page 37). A longer column can be used to increase the pressure within the column, which, in turn, will increase the solubility of the analytes in the stationary phase. Using optimized temperature programs and narrower bandwidths, reasonably fast analysis times can be achieved (see page 37). However, the higher flow rates through wide-bore columns prevent the analyst from directly connecting the column to the vacuum system of an MS. A jet separator or open split interface must be used to reduce the amount of carrier gas flowing into the MS (see Figure 31, page 32).

The method for connecting a purge and trap transfer line to a wide-bore GC column should be carefully considered. The three connection methods are: 1) through the existing GC injection port; 2) using a low volume injector; and 3) with a direct column connection. These alternatives are described below.

Injection Port Connection: In this connection option, the purge and trap transfer line is connected to the GC injection port that accepts the carrier gas line. The carrier gas line is cut close to the injection port body and a deactivated union (e.g., cat.# 20510, see our catalog) is used to connect the purge and trap transfer line to the injection port (Figures 13 through 16). This allows the analyst to make manual injections when troubleshooting, and to inject bromofluorobenzene when tuning the MS in accordance with EPA methods. The injection port can be a source of dead volume, however. Dead volume causes band broadening, resulting in poor peak shape and loss of resolution for the most volatile target compounds. The severity of the problem is determined by the inside diameter of the injection port liner and the total desorb flow through the port. To reduce the dead volume in the injection port, use a 1mm ID split liner (e.g., cat. #20972; see products section). If the injection port is designed for

Vu-Tight® Direct Injection Liner

- Visually observe the Press-Tight® connection between the column end and liner.
- 1/4-Inch OD: accepts 0.32 or 0.53mm ID capillary column (column OD from 0.5mm to 0.8mm).
- Slotted top prevents obstruction of carrier gas flow.
- Two designs available.*
- Operate in the direct injection mode.
- Can easily be packed with wool for dirty samples.



Description	qty.	cat.#
Vu-Tight® DI Liner	ea.	20342
Vu-Tight® DI Liner	5-pk.	20343
Vu-Tight® DI Liner	25-pk.	20344

* Refer to our catalog for information about Cyclo Vu-Tight® liners, for use with dirty samples.

Figure 13.

Plumbing a purge and trap interface to a GC injection port allows flow adjustment via the GC flow controller. This is the most common way of analyzing volatile compounds by MS because the flow can be split, allowing 1mL/min. into the MS source.

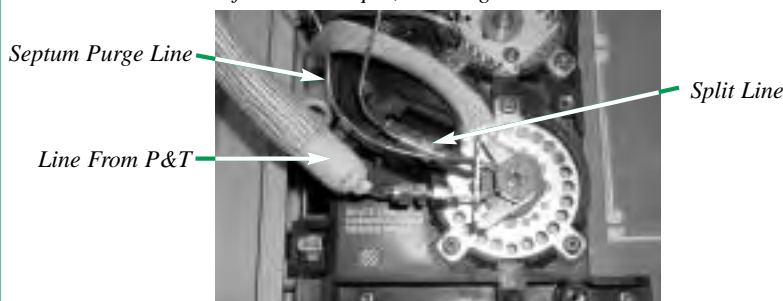
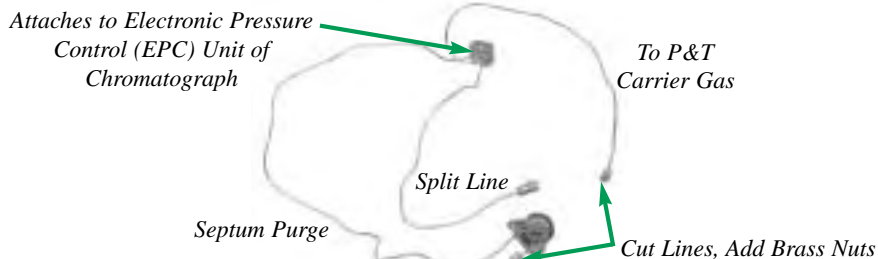


Figure 14.

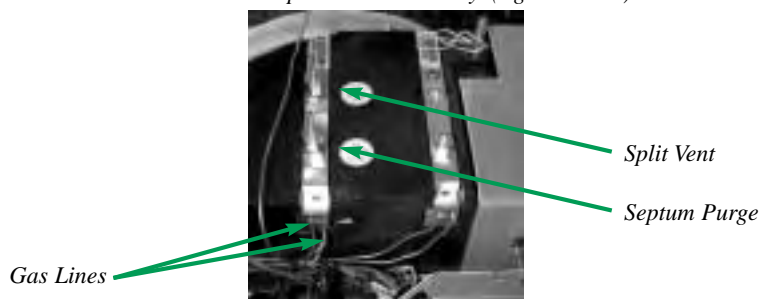
Configuring your GC is simple! Prepare the lines by cutting them as shown. (Agilent 6880)

**Figure 15.**

Carrier gas flow is adjusted through the injection port regulator. Carrier gas sweeps analytes from the trap, through the transfer line, onto the column. (Tekmar 3100)

**Figure 16.**

Reconnect the weldment lines to the GC and check for leaks. Be sure to set up the lines correctly. (Agilent 6890)



Low Volume Injectors



Description	cat.#
Low-Volume Injector for Agilent Split/Splitless GC Inlets	21692
Low-Volume Injector for Agilent 5890 Septum Packed Purge Port	21698
Low-Volume Injector for Varian Split/Splitless GC Inlets	21693

For descriptions of low-volume injectors, see page 63.

packed columns, we recommend using a Vu-Tight® inlet liner (cat. #20342, page 18). The wide-bore capillary column is sealed into the tapered restriction in the liner, ensuring direct transfer of the sample to the column. In addition, the Vu-Tight® inlet liner allows visual inspection of the column-to-liner seal.

Low-Volume Injectors: A low-volume injector (LVI) will reduce dead volume, yet allow limited manual injections. Such a system can be used to convert a packed column or a capillary split/splitless injection port for purge and trap applications. The internal volume of the injector is significantly reduced, compared to a conventional injection port, which improves sample transfer from the purge and trap system. The purge and trap transfer line is connected to the LVI, and the wide-bore column is connected at the base of the injector. A septum in the LVI allows manual injections if needed.

Direct Connection

A union between the purge and trap transfer line and the capillary column bypasses the injection port, eliminating the problems associated with the injection port: loss of sample through the septum purge, adsorption of active compounds, bleed from Viton® O-rings or septum, and — most important — dead volume. Two direct connections are described below. The disadvantage of a direct connection is it eliminates the ability to make manual injections when attempting to isolate a chromatographic problem. Therefore, this connection technique works best for experienced analysts and for instruments that undergo regular maintenance.

Metal Transfer Line: This is the easier of the two direct connection methods. Using the transfer line provided by the instrument manufacturer and an MXT® low dead volume connector (cat.# 20394, see our catalog), connect the trap to the capillary column. This configuration significantly improves peak shape, compared to injection port connections, especially with an electrolytic conductivity detector (ELCD).

Fused Silica Transfer Line: A fused silica transfer line further reduces dead volume, relative to the original equipment line. We recommend using Siltek® fused silica tubing for VOC or other sensitive analyses because it is not affected by moisture and is inert to active compounds. To configure the line, disconnect the metal transfer line from the Valco® six-port valve, then remove the metal ferrule and 1/16" nut by cutting the end of the tubing. While wearing insulated gloves, heat the line to 200°C to melt the glue that holds the line in place, then use pliers to pull the line out of the heater jacket. Cool the line, install a piece of metal tubing (cat. #21503, see our catalog) inside the line, then install the Siltek®-treated fused silica transfer line within the metal tubing (cat. #10027, page 63). The metal tubing will prevent the transfer line from being scratched or broken. Base the ID of the metal tubing on the OD of the transfer line: 0.02" ID for a 0.25 or 0.32mm ID fused silica line, 0.30" ID for a 0.45 or 0.53mm ID line. In turn, base the ID of the transfer line on the ID of the analytical column; we recommend using a transfer line with an ID equal to or slightly smaller than that of the column. A transfer line with an ID slightly smaller than that of the column will increase backpressure, enhancing the resolution of early-eluting compounds. Use a Press-Tight® connector (cat. #20400 or 20403, page 20) to connect the fused silica transfer line to the analytical column (Figure 17). Use the correct ferrule for connecting the column to the 6-port valve (Figure 18); we recommend a one-piece fused silica adaptor (cat. #20137, page 64).

Universal Press-Tight® Connectors

Description	cat.#
Universal Press-Tight® Connectors, 5-pk.	20400
Universal Press-Tight® Connectors, 25-pk.	20401
Universal Press-Tight® Connectors, 100-pk.	20402
Universal Angled "Y" Press-Tight® Connector, ea.	20403
Universal Angled "Y" Press-Tight® Connectors, 3-pk.	20404

For additional connectors, see page 64.

Figure 17.

A dual-column configuration splits the sample equally between separate detector systems.



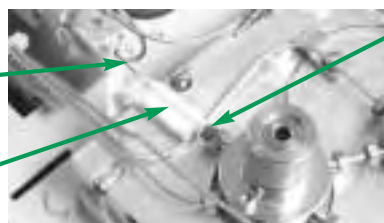
Angled "Y"
Press-Tight®
Connector

Figure 18.

Connect the fused silica line directly to the 6-port valve. Notice a small (5mm) septum helps determine how far the column is inserted into the valve, preventing breakage at the column end that could allow shards of fused silica to enter the valve.

MXT® Metal Tubing
(used to protect fused
silica column)

Fused Silica



Septum

Narrow-bore Systems (0.18mm ID - 0.32mm ID columns)

Narrow-bore columns (0.18mm ID-0.32mm ID) offer higher resolution, compared to 0.45mm ID or 0.53mm ID columns. Because these columns typically are operated at lower flow rates, they are not compatible with the fast desorb flow rates from common purge and trap systems. Splitting the sample at the injection port or cryofocusing (i.e., secondary trapping) will provide compatibility and help focus the sample at the column inlet.

Splitting the Sample: Many environmental laboratories analyzing VOCs by GC/MS use narrow-bore capillary columns and split the sample at the injection port. Higher sensitivity ion trap GC/MS systems (e.g., Varian Saturn 2000™ and Thermo Finnigan GCQplus™ systems)⁷ and recently developed quadrupole MS systems (e.g., the Agilent 5973 system) allow high split ratios in the injection port while maintaining sensitivity adequate to meet the requirements of EPA Method 524.2.⁸ Older quadrupole GC/MS systems require an increase in purge volume (25mL) to compensate for the sample lost due to splitting.

Using a standard split/splitless injection port to split the desorb flow allows a higher desorb flow rate while maintaining a lower column flow. With this technique, the trap is desorbed at a flow rate of 10-60mL/min. and the column flow rate is adjusted to 1.0-1.3mL/min., which is compatible with the vacuum system of an MS. The remaining flow exits through the split vent. The faster desorb flow rate produces a narrow sample bandwidth which, when combined with the high efficiency of a narrow-bore column, allows high split ratios without significant loss in sensitivity.⁸ Surprisingly, a 1:20 split ratio provides more sensitivity than a 1:10 split ratio, because the higher flow from the trap focuses the target compounds more efficiently.

Cryofocusing (secondary trapping): A cryofocusing unit refocuses the volatile compounds at the inlet of the narrow-bore column. This allows the trap to be desorbed at only 1-2mL/min., while improving peak shape and resolution by reducing sample bandwidth. Cryofocusing takes place on a short length of deactivated, uncoated, fused silica tubing that is cooled to -160°C using liquid nitrogen. To increase retention for very volatile gases, or when analyzing Freon® compounds, use tubing coated with a thick film of stationary phase.

While cryofocusing greatly improves peak shapes from narrow-bore columns, the approach consumes large amounts of liquid nitrogen, increasing operating expenses and requiring liquid nitrogen tanks in the lab. If the liquid nitrogen tank empties in the middle of a sample sequence, there can be significant downtime before the tank is replaced.

Capillary Column Phases

Many capillary columns have been designed for the analysis of VOCs. Column selection normally is based on the analytical method (e.g., US EPA method), compound list, and detection system used. This section serves as an overview of the different phases used for VOC analyses. See the Applications section (page 37) for examples of GC and GC/MS separations under specific conditions.

The first columns used for analyzing volatiles were based on diphenyl/dimethyl polysiloxane stationary phases. These include VOCOL®, Rtx®-Volatiles, HP®-VOC, and Rtx®-502.2 columns. The main advantages of these phases are their resistance to oxidative breakdown and their lower bleed, compared to cyanopropylphenyl polysiloxane (i.e., "624") phases. The major drawback of diphenyl/dimethyl polysiloxane phases is the incomplete resolution of bromomethane and chloroethane. Many environmental laboratories still use these columns, especially when analyzing samples for a limited set of compounds.

7. Jessie Crockett Butler, Meredith Conoley, "Analysis of Volatile Organics in Solid Wastes, Soils, and Water Using a Split Injection and the Polaris Q Ion Trap GC/MS." Application Note AN9167. Thermo Finnigan, GC and GC/MS Division, Austin, TX.

8. D.R. Decker, J.J. Harland, and M.J. Feeney, "Comparison of Detection Limits and Analysis Time Using Wide and Narrow Bore Capillary Columns for Purge-and-Trap GC/MS Analyses." OI Analytical. Application Note 02850896.

Another type of column used for VOC analysis is based on cyanopropylphenyl/dimethyl propyl polysiloxane phases, commonly known as the “624” phases. The Rtx®-624 column is designed for EPA Method 624, but also performs well for Methods 524.2, Revision IV, and 8260. The main advantage of the Rtx®-624 column is the complete separation of the highly volatile gases, including dichlorodifluoromethane, chloromethane, vinyl chloride, chloroethane, and bromomethane.

More recently, the Rtx®-VRX column was developed, using computer-assisted stationary phase design (CASPD), to address the expanded list of compounds in EPA Methods 8021 and 502.2. This unique column improves resolution and reduces overall analysis time compared to traditional columns. Like the Rtx®-624 column, the Rtx®-VRX column provides excellent separation of the highly volatile gases. Its only disadvantage is poor resolution of the most common trihalomethanes (THMs), chloroform and bromodichloromethane, from other target analytes. These analytes are frequently found in chlorinated drinking water samples. While the Rtx®-VRX column has been used for MS methods with favorable results, because of this poor resolution it is not recommended for drinking water analysis using PID/ELCD detection.

The most recent innovations for VOC analysis have been the development of the Rtx®-VGC and Rtx®-VMS columns. These columns also were designed using CASPD. Designed for PID/ELCD analyses, the Rtx®-VGC column resolves all compounds listed in EPA Methods 502.2 and 8021, with >80% resolution of each of the four trihalomethanes from the other target compounds, >30% resolution between 2-chlorotoluene/1,1,2,2-tetrachloroethane, and >60% resolution of all other volatile compounds in the two EPA methods. The column resolves the gases and early-eluting compounds well enough that the GC oven program can be started at 50°C.

The Rtx®-VMS column was designed to address the increasing number of analytes listed in EPA Method 8260, and also is a good choice for separating compounds listed in EPA Method 524.2, revision IV. The major difference between the Rtx®-VMS phase and others such as “502.2,” “624,” or “VRX” is its overall selectivity and the distance between members of isomeric pairs, like 2-/4-chlorotoluene. A faster final oven ramp rate is possible because these compounds elute farther apart on the Rtx®-VMS phase, eliminating partial co-elutions that would interfere with quantification. This column offers excellent separation of EPA Method 8260B compounds in less than 18 minutes - the normal cycle time for a purge and trap system. Using the EPA-suggested surrogates the analysis time can be less than 10 minutes with a narrow bore column. Even faster analyses are possible if you replace the internal standard chlorobenzene-d5 with another compound, such as 4-bromofluorobenzene. Sub-10-minute analysis times allow you to connect two purge & trap units to one GC/MS instrument, significantly increasing sample throughput (see page 37).

Metal Columns

In addition to the standard fused silica versions of the analytical columns discussed above, metal MXT® columns coated with the same stationary phases also are available from Restek. To eliminate the activity problems associated with metal tubing, we make these columns from Silcosteel®-treated stainless steel tubing, assuring excellent inertness. Because these columns are much more durable than fused silica columns, and can be coiled to less than 5-inch diameters, they are ideal for portable GC applications. Their durability makes them a popular choice for teaching laboratories at colleges and universities. Analyte resolution on MXT® columns is similar to that on fused silica columns.

Detection Systems

VOCs can be analyzed using a variety of detection systems, including GC/MS, GC/PID, and GC/ELCD. Here, we discuss consequences of using each of these systems, and present tips for maintenance and troubleshooting.

Column Configurations

Single Column: Environmental engineers characterize a contaminated site using MS or dual-column GC, or they might monitor the site solely with single-column GC methods. Injections of standards on a single column, delivered to the detector, can provide tentative identification and quantification. Retention times for analytes listed in a given method are established by injecting a check standard containing all of the target compounds. Retention times for analytes in site samples are compared against retention times for the standard, to verify if unknown compounds match known targets. A single column configuration works well with characterized samples, but retention times are not unique for every analyte, especially for early-eluting compounds that spend little time in the stationary phase (e.g., Freon® compounds). In environmental laboratories coelutions from non-target compounds also are very common, creating very complex chromatograms that are difficult to interpret using a single-column design.

Dual Columns: In a dual-column configuration, the sample passes through a fused silica guard column, then is split between two analytical columns of differing selectivity. Standards are injected to establish retention times on both columns simultaneously. One disadvantage to this configuration is the 50% loss in sensitivity resulting from splitting the sample. This loss can be overcome by increasing the sample volume or by optimizing the detector. Flow rates for the two columns should agree within 20% because uneven splitting will further affect sensitivity.

Detector Configurations

Detectors can be connected in parallel, in series, or in tandem, to double the amount of information about the sample.

Parallel System: In a parallel system the sample is split equally between the two detectors, allowing both detectors to be destructive (e.g., ELCD/FID). This detection system works well but is unsuitable for a dual-column analytical configuration because the sample already will have been split between the two columns.

Series System: Series detection involves connecting two detectors in sequence, using a short length of deactivated metal or fused silica tubing. The sample passes through the first detector, which must be non-destructive (e.g., PID), then through the second detector. This produces two sets of information about the sample with no loss in sensitivity because the sample volume does not change from the first detector to the second. The only disadvantage is dead volume, which can broaden the peaks. Minimize dead volume by minimizing the length and ID of the line connecting the detectors.

Tandem System: The tandem configuration connects two detectors without the dead volume associated with a series system. The non-destructive first detector is the base for the second detector. The units can be connected to a single detector port on one GC. This makes it possible to use a dual-column configuration, with each column connecting to tandem detectors, producing four sets of data per analysis. This approach is used in EPA Method 8021.⁹

Detectors: Method requirements determine the choice of detector(s). The current shift toward analysis by performance-based criteria makes it possible to use detection other than that listed in a method if it can be shown that performance is similar to, or better than, what would be attained by following the guidelines in the method. The most common GC detectors are the PID, the FID, and the ELCD. GC/MS eliminates the need for a confirmation column.

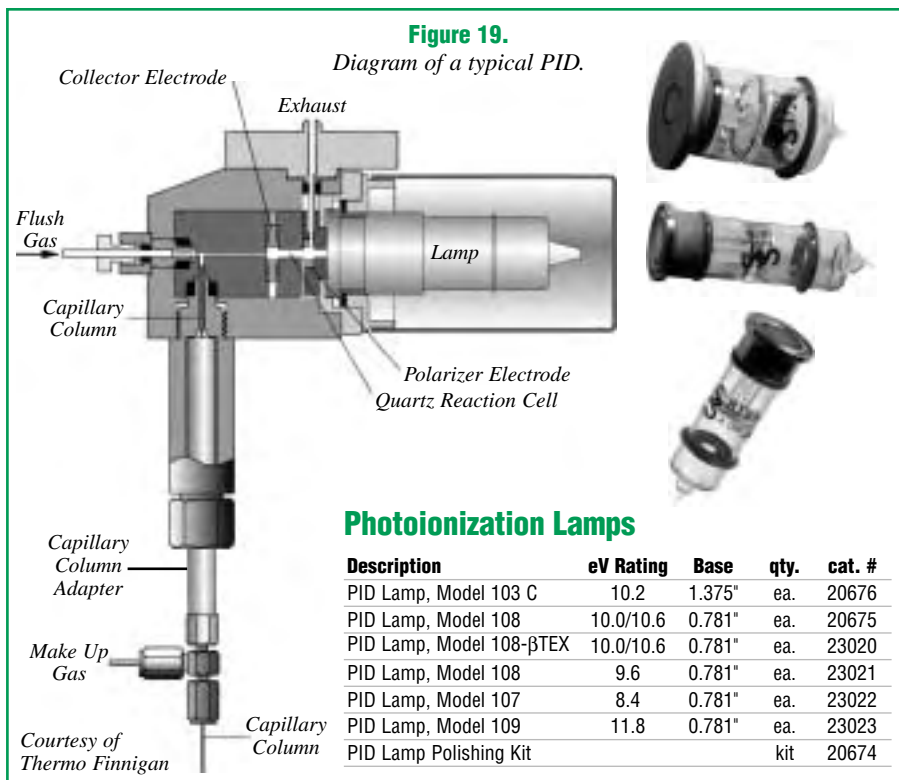
9. R.D. Braun. *Introduction to Instrumental Analysis*. McGraw-Hill Book Company. New York. 1987. pp. 915-916.

PID Operation

The photoionization detector, PID, is a selective, non-destructive detector most commonly used for characterizing aromatic compounds (Figure 19). It has excellent sensitivity (low pg detection) and provides a linear dynamic range of 3 orders of magnitude.

In a PID, a krypton lamp emits photons in the form of light energy at a wavelength of 116.6nm and 123.6nm. The photons excite compounds having an ionization potential of less than 10.2eV. Charged particles produced in this manner pass through a reaction cell with an electrical potential of 50 to 200 volts, producing an electrical charge that is measured as a signal. Sensitivity is a function of the chemical structure of the analyte, including the number of carbon atoms, the nature and position of functional groups, and the position of double or conjugated double bonds.¹⁰ For suitable analytes, a PID is 10-times more sensitive than an FID. Compounds such as benzene (9.3eV ionization potential) have ionization efficiencies of less than 0.1%, allowing the majority of the sample to pass through the detector unaffected. Even with this minute portion of the sample ionized, sensitivity for aromatic compounds is measured at the pg level.

PID Maintenance and Troubleshooting: It is very common for silicone from column bleed to collect on the PID window and reduce transmission from the lamp. Reduced sensitivity for all components is evidence of this condition. We recommend cleaning the window on a regular basis, using a mild abrasive material such as iron oxide. Alumina powder is more abrasive than iron oxide and can scratch the lens. This will reduce sensitivity. Create a slurry with the powder, scrub the window, then rinse with acetone or methanol. Avoid touching the clean window. If sensitivity is not restored, replace the lamp. Dead volume within a PID will produce broad peaks, and might cause peaks to tail. PIDs designed for packed column systems have a cell volume intended for high flow rates. When using these detectors with capillary columns, 20-30mL/min. of make-up gas is required to minimize dead volume and, in turn, reduce peak tailing. Newer PID designs have smaller cell volumes that are compatible with capillary columns.



10. Jessie Crockett Butler, "Tandem Detector Technology for Gas Chromatography." Environmental Analysis Technical Report No. 9110, p3. Thermo Finnigan, GC and GC/MS Division, Austin, TX.

FID Operation

The flame ionization detector, FID (Figure 20), is a selective detector because it only responds to materials that ionize in an air/hydrogen flame. This condition covers a very broad range of compounds, however. An FID / PID combination often is used for petroleum or volatile analyte applications.

In an FID, the combustion of hydrogen and air produces a flame. When an organic compound enters the flame; the large increase in ions produced is measured as a positive signal. Because response is proportional to the number of carbon atoms introduced into the flame, an FID is considered a quantitative counter of carbon atoms burned.¹⁰ Among the detectors discussed here, an FID has the largest linear dynamic range – nearly five orders of magnitude. The FID will detect most carbon-containing analytes at a sensitivity of approximately 0.5ng/ μ L.

Properly set gas flow rates are important to achieving maximum sensitivity with an FID, and preventing the flame from being extinguished (flame-outs). Generally, the total flow to the FID is 300mL/min. to 500mL/min., of which the hydrogen flow plus the carrier gas flow is approximately 30mL/min. The balance of the flow (make-up gas) typically is nitrogen.

The carrier gas and hydrogen gas mix in the FID jet. Capillary columns require a small jet (0.011 inch ID) whereas most packed column applications are compatible with a larger jet (0.018 inch ID). Jets with even larger ID are available for applications involving packed columns that exhibit higher bleed.

Capillary Adaptable FID Jet for Agilent 5890/6890/6850 GCs (0.011-inch ID tip)

(Similar to Agilent part # 19244-80560.)

Description	qty.	cat.#	qty.	cat.#
Standard	ea.	20670	3-pk.	20671
High-Performance (Silcosteel®-Passivated)	ea.	20672	3-pk.	20673

Capillary Dedicated FID Jet for Agilent 6890/6850 GCs

(Similar to Agilent part # G1531-80560.)

Description	qty.	cat.#	qty.	cat.#
Standard	ea.	21621	3-pk.	21682
High-Performance (Silcosteel®-Passivated)	ea.	21620	3-pk.	21683

Capillary FID Jet for Agilent 5880 GCs

(Similar to Agilent part # 19301-80500.)

Description	qty.	cat.#
Standard	ea.	21637
High-Performance (Silcosteel®-Passivated)	ea.	21638

Packed Column FID Jets for Agilent 5890/6890/6850 GCs

0.018-Inch ID

(Similar to Agilent part # 18710-20119.)

(Similar to Agilent part # 18710-20119.)	qty.	cat.#	qty.	cat.#
Standard	ea.	21694	3-pk.	21695
High-Performance (Silcosteel®-Passivated)	ea.	21696	3-pk.	21697

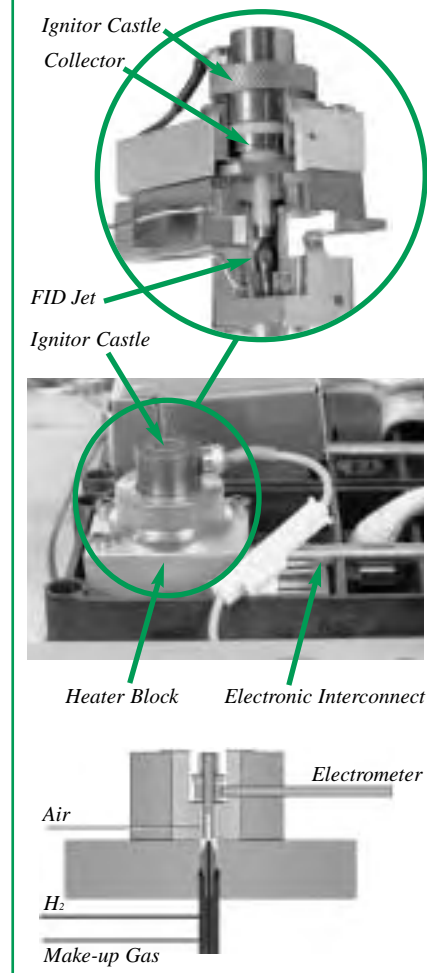
0.030-Inch ID

(Similar to Agilent part # 18789-80070.)

(Similar to Agilent part # 18789-80070.)	qty.	cat.#	qty.	cat.#
Standard	ea.	21688	3-pk.	21689
High-Performance (Silcosteel®-Passivated)	ea.	21686	3-pk.	21687

Figure 20.

Components of a typical FID

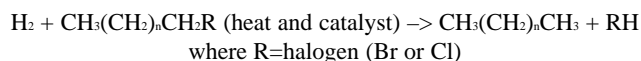


www.restekcorp.com

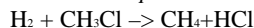
FID Maintenance and Troubleshooting: Contamination and a clogged jet are common problems associated with using an FID in analyses of volatile compounds, such as gasoline range organics (GRO) analyses that involve samples containing diesel fuel or oils. Flame-outs at the beginning of a VOCs analysis usually are the combined result of incorrect gas flows and excessive water from the purge trap. When performing maintenance on an FID always check the gas flows before calibrating the instrument. Water management is discussed on page 13.

ELCD (Hall® detector) Operation

In typical applications, an electrolytic conductivity detector, ELCD (Figure 21), is a chemical detector that catalytically reduces halogenated materials to haloacids, HCl and HBr, by mixing them with high-temperature hydrogen in a heated nickel reaction tube. In other words, this detector pyrolyzes these analytes in the presence of a catalyst and a reaction gas (hydrogen):



Example:



The haloacid molecules flow into the electrolytic conductivity cell via a Teflon® transfer line, and are dissolved in a stream of n-propanol. The conductivity of the alcohol is monitored because the concentration of hydrogen halide is directly proportional to the current. The signals thus produced characteristically have tailing peaks. Although the ELCD is most commonly used for halogenated compounds and, in the halogen mode, it is selective only for these species (Figure 21), it can be configured to detect sulfur, nitrogen, and nitrosamine compounds. Figures 22-25 and Figure 27 show various important parts of the ELCD system.

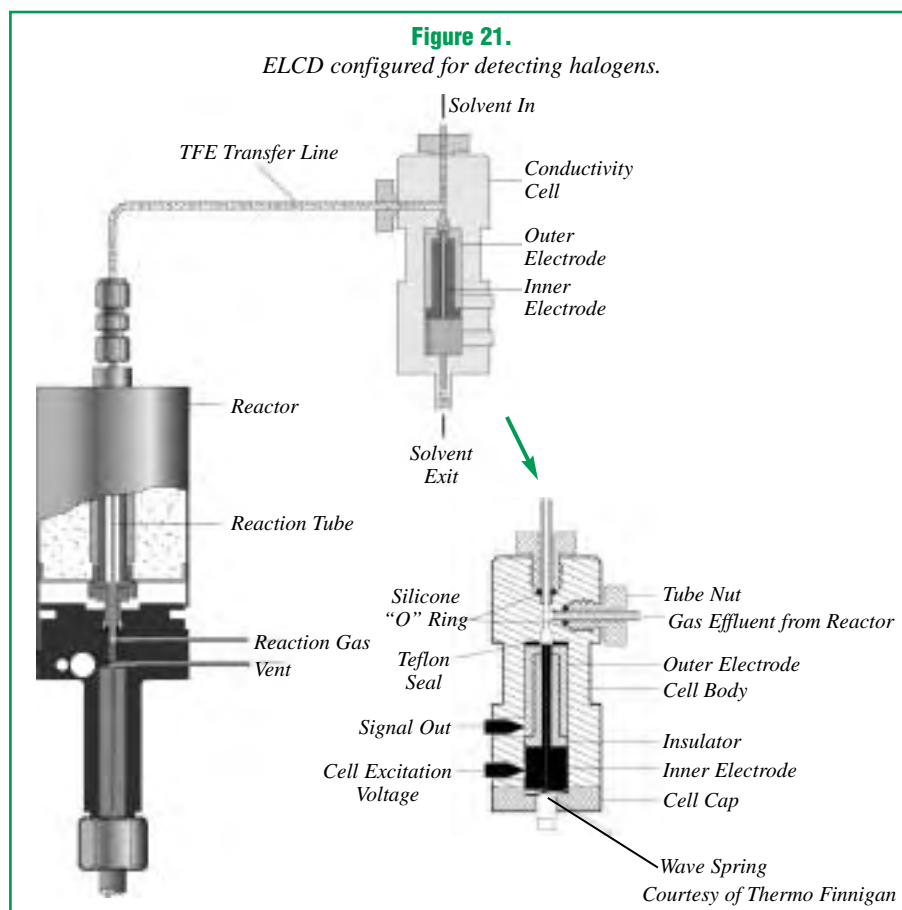
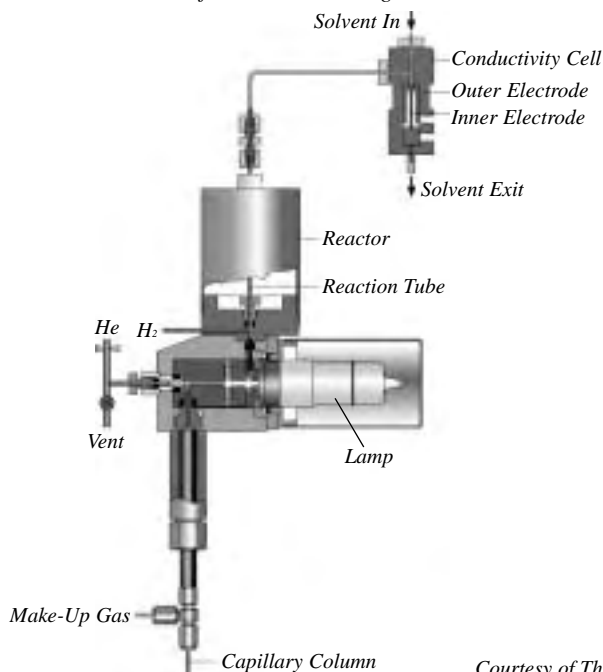
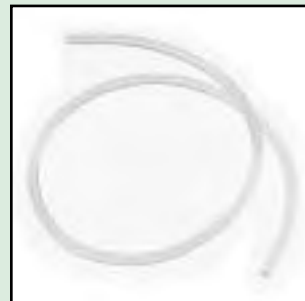


Figure 22.

Cross-section of the Thermo Finnigan PID/ELCD



Courtesy of Thermo Finnigan

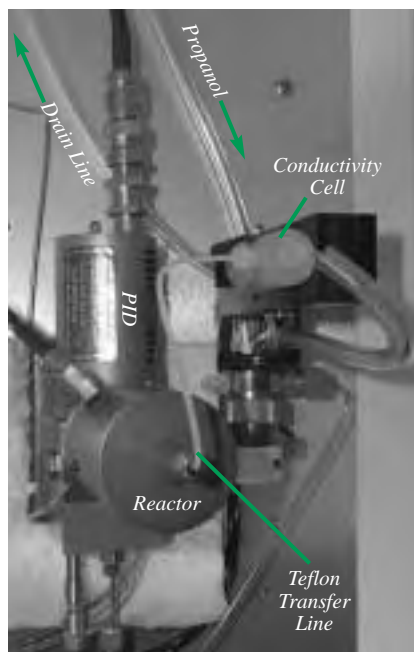
Teflon® Transfer Lines for ELCDs

- Stringently cleaned with HCl.
- Convenient precut pieces.
- Fit Tracor, Tremetics, O.I., many other ELCDs.

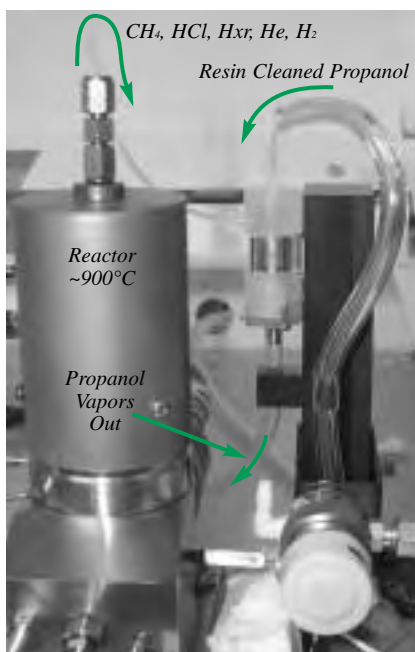
Description	cat.#
Teflon® Transfer Lines for ELCDs (five 6.5-inch lines), 5-pk.	20121

Figure 23.

Electrolytic conductivity detector: top view

**Figure 24.**

Side view of the tandem detectors.

**Chromatographic Detectors:
Design, Function, and Operation**

Comprehensively covers the design, construction, and operation of gas chromatography, liquid chromatography and thin-layer chromatography detectors—all in one convenient, up-to-date source.

R.P.W. Scott, Marcell Dekker, Inc., 1996, 514pp., ISBN 0-8247-9779-5 cat.# 21090



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Ultra-High-Purity Brass Line Regulator

- Use wherever you need to reduce the line pressure by 20psi or more.
- Same purity level as high-pressure cylinder regulators.



Fitting	qty.	cat.#
1/4" female NPT ports*	ea.	21666

* Please order appropriate male connector, pipe-to-tube fittings; see our catalog.

Figure 25.

Electrolytic conductivity detector: propanol tank with pump.

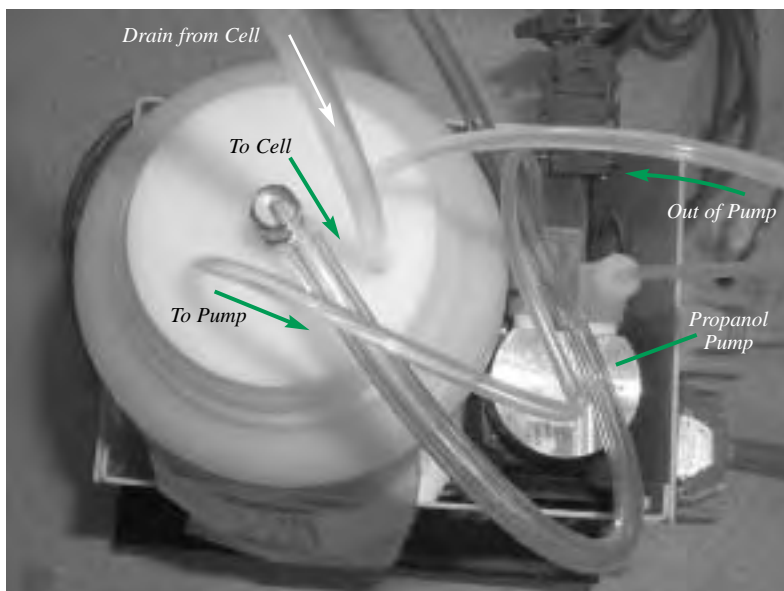


Figure 26.

With any detector, always be sure that gas flows are adjusted properly.



ELCD Maintenance and Troubleshooting: ELCD performance depends on the reactor temperature, the volume of the conductivity cell, the propanol flow rate, the hydrogen gas flow (Figure 26), and the purity of the solvent system. The goal is to minimize tailing as much as possible without losing sensitivity. Depending on the amount of use and the types of samples analyzed, the nickel reaction tube – the reaction catalyst – should be replaced as often as monthly. Hydrocarbons and certain other compounds can “poison” the reaction tube and reduce its catalytic activity. Oxygen and moisture can oxidize the reaction tube, affecting sensitivity. High-purity hydrogen gas is critical for a stable baseline. Use gas regulators with stainless steel diaphragms and the proper purifiers for reaction gases.

A drop in sensitivity (particularly for brominated compounds), baseline instability, or appearance of unknown peaks indicate it might be necessary to replace the reaction tube. Reconditioning the reaction tube might restore baseline stability: disconnect the Teflon® transfer line, then increase the reactor temperature to 1000°C for one hour, then reset the reaction temperature to 900°C for re-calibration.

Similarly, the Teflon® transfer line between the reactor and the conductivity cell requires frequent cleaning or replacement. Flushing the transfer line should remove most of the contamination. To do this, disconnect the line from the reaction tube and plug the drain line leading from the reaction cell. This will force propanol through the transfer line, flushing contamination out. If this does not improve response and peak shape, replace the transfer line (cat# 20121, page 27).

Use only high-purity solvents in the ELCD (only HPLC-grade for halogen mode). The solvent intake line is equipped with a scrubber resin cartridge that removes contaminants from the solvent. To maintain solvent purity and a stable baseline, change this cartridge every six months.

ELCD: Minimizing Peak Tailing: Peak tailing is a characteristic of the ELCD – the key to successful ELCD operation is regular maintenance to minimize the tailing. Most tailing problems are caused by contamination or leaks in the system. Peak tailing also can be caused by contamination in the Teflon® transfer line from the reaction tube to the conductivity cell. Table III lists factors that can contribute to tailing peaks. Reaction tube deterioration can be due to water and/or oxygen corroding the tube surface over time, or to carbon deposits left by the organic solvent. In purge and trap applications, water management can help slow this corrosion.

Poor responses for brominated compounds indicate active sites in the pathway. Isolate the purge and trap system by making a manual injection. If responses for brominated compounds still are poor, the reaction tube probably is deteriorating. A combination of tailing peaks and poor responses for brominated compounds also is an indication that the reaction tube must be replaced. Maintain detailed notes on instrument maintenance to minimize troubleshooting problems in the future.

ELCD performance also depends on the internal volume of the conductivity cell. Older ELCDs have larger cell volumes that cause more tailing. Smaller cells in newer ELCDs significantly reduce peak tailing.

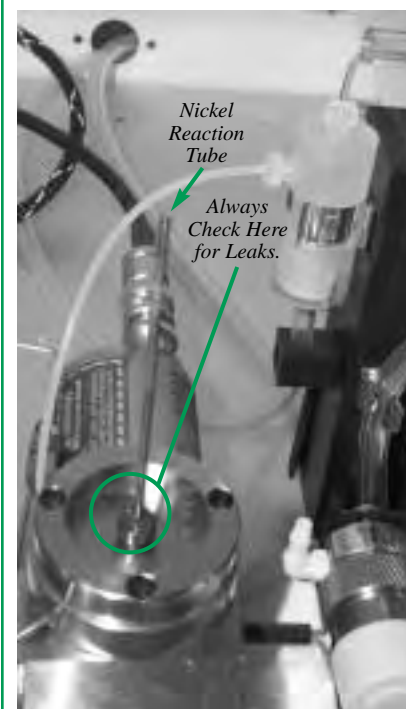
Table III.

Common causes of excessive peak tailing from an ELCD.

Contaminated conductivity cell	Low makeup gas flow
Contaminated reaction tube	Low propanol flow
Contaminated resin cartridge	Low reaction gas flow (hydrogen)
Contaminated Teflon® transfer line	Low reaction temperature (below 850°C)
Dead volume between detectors in series	Unpure gas (carrier / makeup / reaction)
Leak at the base of the reaction tube	

Figure 27.

ELCD reactor removed from detector assembly, reaction tube exposed.



Replacement Nickel Reaction Tubes

- Pretreated for maximum sensitivity.
- Quality-controlled for reliability.
- Available for different models.



To replace these instrument part numbers:

Order these
Restek part
numbers:

ELCD Model #	Tremetrics	Varian	PerkinElmer	Shimadzu	O.I. Analytical	qty.	cat.#
Hall 700A	115439-0003	00-996724-14	0330-2675	—	—	2-pk.	21580
Hall 1000	117459-0003	00-997625-12	N660-1072	220-90435-00	—	2-pk.	21581
O.I. 4420	—	—	—	—	183780	2-pk.	21582

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GC/MS Operation

Mass spectrometry (MS) is the most common detection system used for VOC analysis. The MS provides unique spectral information for accurately identifying components eluting from the capillary column. As a compound exits the column it is bombarded with high-energy electrons and is broken into structurally significant charged fragments. These fragments are separated by their mass-to-charge ratios in the analyzer, to produce a spectral pattern (i.e., fingerprint) unique to the compound. To confirm the identity of the compound the spectral fingerprint is matched to a library of known spectra. By knowing the spectral patterns for compounds in the target list, the appropriate masses for quantification can be chosen.

For analyzing volatile compounds in environmental samples, the most common types of MS operating systems are the quadrupole system and the ion trap system.

Quadrupole Operation

A narrow bore (≤ 0.25 mm ID) capillary column can be inserted into the source of the quadrupole MS in electron impact mode (EI). The carrier gas flowing through the column, approximately 1mL/min., is quickly swept away under the high vacuum of the source while analytes exiting the column are bombarded with a stream of electrons at 70eV.

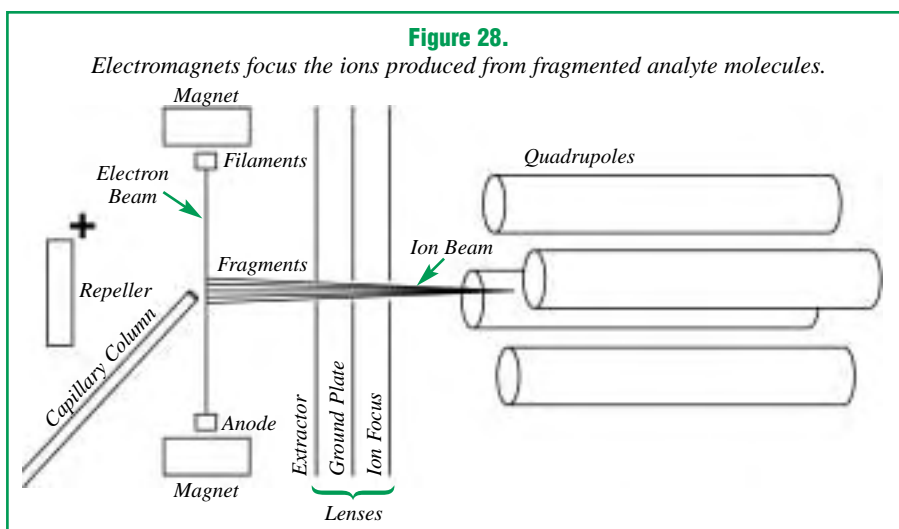
Electromagnets begin focusing the ions (Figure 28). Positively charged fragments are pushed away from the positively charged repeller, toward a series of focusing lenses. The first lens, the draw-out plate or extractor, accelerates the ions, then the ion focus lens further accelerates the ions and squeezes them into a tight beam of charged particles before they enter the mass analyzer – an array of four parallel rods, or quadrupoles. Other plates (i.e., a ground plate), if present, are connected to a ground that discharges the defocused ions, to prevent them from causing charge interference with the ion focus lens. In this way ions that are not correctly directed down the quadrupoles are discarded. Electromagnetic fields produced by a combination of direct current (DC) and an oscillating radio frequency (RF), enables ions that have a specific mass-to-charge ratio to pass through the quadrupoles to the detector, forcing these ions into a spiral, or corkscrew-shaped, three-dimensional sine wave that passes through the center of the quadrupole arrangement (Figure 29). As the DC/RF waves are swept up or down, specific mass-to-charge ions strike the electron multiplier (detector), which translates ions to electrons. The electrons bounce off the dynodes (walls) of the electron multiplier, generating a cascade of electrons. These electrons are exchanged to photons, which are measured as a current by the photomultiplier.¹¹

Gas Chromatography & Mass Spectrometry, A Practical Guide

- Separation conditions for numerous compound types, derivatized and underivatized.
- How to interpret mass-spectral data, with examples.



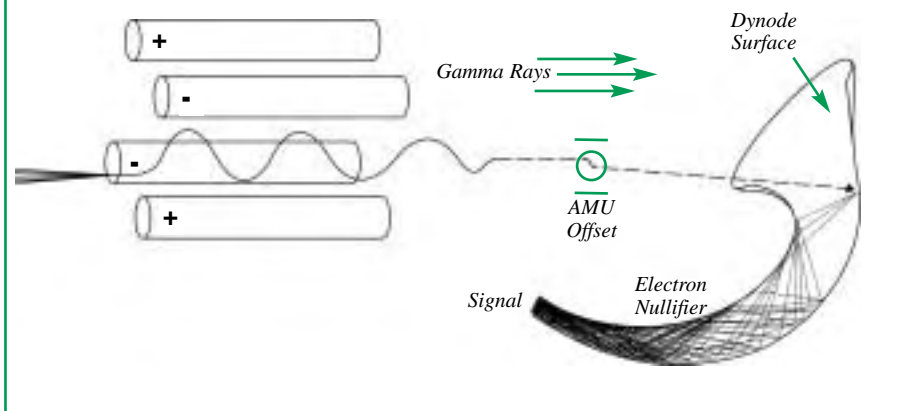
F.G. Kitson, B.S. Larsen and C.N. McEwen, Academic Press, 1996, 381pp., ISBN 0-12-483385-3 cat.# 20497



11. F.G. Kitson, B.S. Larsen & C.N. McEwen. Gas Chromatography and Mass Spectrometry: A Practical Guide, Academic Press, New York. 1996.

Figure 29.

Electromagnetic fields force the ions into a spiral, three-dimensional sine wave through the center of the quadrupole arrangement.



Ion Trap Operation

The major difference between a quadrupole MS and an ion trap MS are the mechanisms of ion focus and scanning. Three hyperbolic electrodes, a ring and two endcaps, form the core of an ion trap MS (Figure 30). In electron impact mode the sample is ionized, fragmented, and introduced into the ion trap through a pulsing electronic gate that opens and closes, controlling the number of ions that enter the trap. Ions that enter the trap are stored in stable orbits. Adjusting the voltage around the ring electrode pushes some of these ions into unstable orbits, causing them to exit to the detector. Because all ions entering the trap are stored temporarily, only a finite amount of sample can be allowed to enter the trap area, otherwise the system would be overloaded.

Interfacing the Capillary Column to the MS

The ion source and analyzer of the MS are under vacuum. To enable the pumping system to maintain this vacuum, the volume of carrier gas entering the MS must be small. Regardless of the pumping capacity of the MS vacuum system, the best sensitivity is achieved if the carrier gas flow rate is approximately 1mL/min. Because a narrow-bore capillary column routinely is operated at near 1mL/min. flow rates, it can be connected directly to the MS without overwhelming the pumping system. Wide-bore capillary columns, however, usually are operated at flow rates that are too high for most MS systems. Consequently, an interface must be used to reduce the flow to a level that is compatible with the MS pumping system. Figure 31 shows the two most common interfaces – the open split and the jet separator.

An open split interface (OSI) functions like an inlet splitter system in a chromatograph. It allows as much as 90% of the carrier gas to be vented away from the MS vacuum system. Correspondingly, this is reflected by a sample loss of up to 90%, which reduces sensitivity by an order of magnitude. Therefore, an OSI is not suitable for trace-level environmental analysis. Splitting the sample at the injection port, combined with analysis on a narrow-bore column, is favored over using an OSI because a high desorb flow rate can be used to ensure better sample transfer from the trap. Also, a 0.25mm ID or narrower column increases efficiency and improves resolution of analytes.

Another alternative to an OSI, the jet separator, reduces the carrier gas flow without significant loss of sensitivity. A jet separator works on the principle of momentum. Very small molecules such as helium (or other carrier gas) do not have sufficient momentum to pass across a small gap in the jet separator and are routed away from the MS, using a vacuum pump. Larger molecules, such as most target components, have the necessary momentum to carry them across the gap and into the MS. Using this device, much of the carrier gas can be eliminated without significant loss of target compounds. Added momentum is required to carry very small analyte molecules, such as gases, across the gap, however. In these situations we recommend adding make-up gas to provide the extra momentum and improve responses for low molecular weight target compounds.

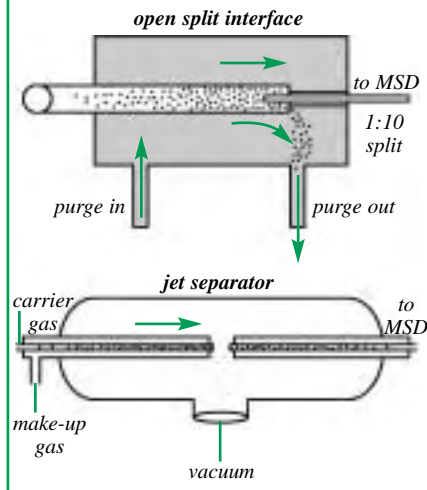
Figure 30.

Ring and end caps form the core of an ion trap MS.



Figure 31.

An open split interface or a jet separator will reduce the gas flow entering a mass spectrometer vacuum system.



MS Calibration and Tuning

Calibration allows the correct identification of masses, whereas tuning adjusts the intensity and peak widths for masses. The MS is calibrated by adjusting the DC/RF frequency so that mass axis points are aligned with expected mass fragments of known spectra. Tuning ensures that target compounds analyzed on the MS will have the same distribution (pattern) of ions, and peak widths for ions will be narrow enough that adjacent mass peaks will not overlap. A compound widely used for calibrating and tuning MS systems is perfluorotributylamine (PFTBA or FC43). Modern instruments introduce PFTBA into the ion source during the autotune procedure. The instrument software adjusts the MS parameters to match the known fragmentation pattern for PFTBA. The ion of greatest abundance in the spectrum is mass 69; the relative abundances of masses 131 and 219 are roughly 50% of that for mass 69 (Figure 32). In analyses of volatiles, mass 502 is less important because its relative abundance is 1% of the mass 69 value. Low peak heights or a loss of mass 502 generally indicate a cleanliness problem at the source.

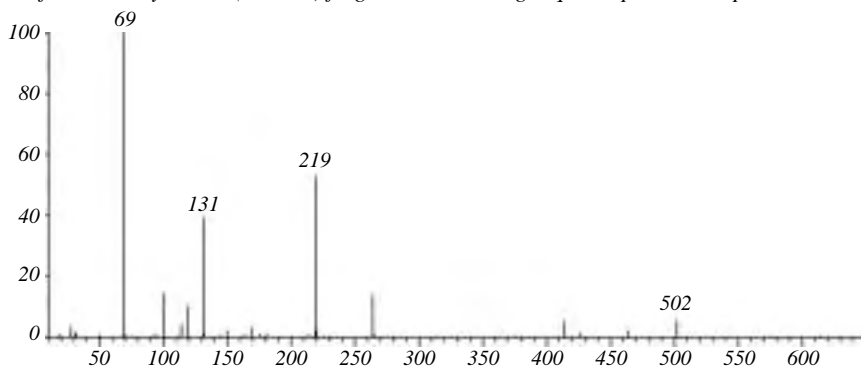
Specific Tuning Requirements: 4-Bromofluorobenzene: After the system is calibrated and tuned, using PFTBA, a 50ng solution of 4-bromofluorobenzene (BFB) is introduced. BFB usually is introduced by injection through the GC injection port but, alternatively, it can be purged from a water blank. Abundance criteria for BFB are listed in Table IV.

If tuning with BFB fails under criteria acceptable for PFTBA, decrease the relative abundance of masses 131 and 219 to 30% of mass 69 by adjusting the entrance lens. If necessary, slightly decrease the repeller voltage. This procedure targets the ions from mass 173 through mass 177. A second tuning failure with BFB may dictate recalibration and tuning with PFTBA. Ion ratios for BFB should be checked every 12 hours. As long as results meet the specifications in Table IV, no further calibration or tuning is required.

Poor tuning can significantly affect the sensitivity of the MS. Figure 33 shows spectra for a sample analyzed twice, first after a failing PFTBA tuning with mass 131 as the base peak (Figure 33, A), then after a passing tuning (Figure 33, B). The second analysis exhibits a three-fold increase in sensitivity.

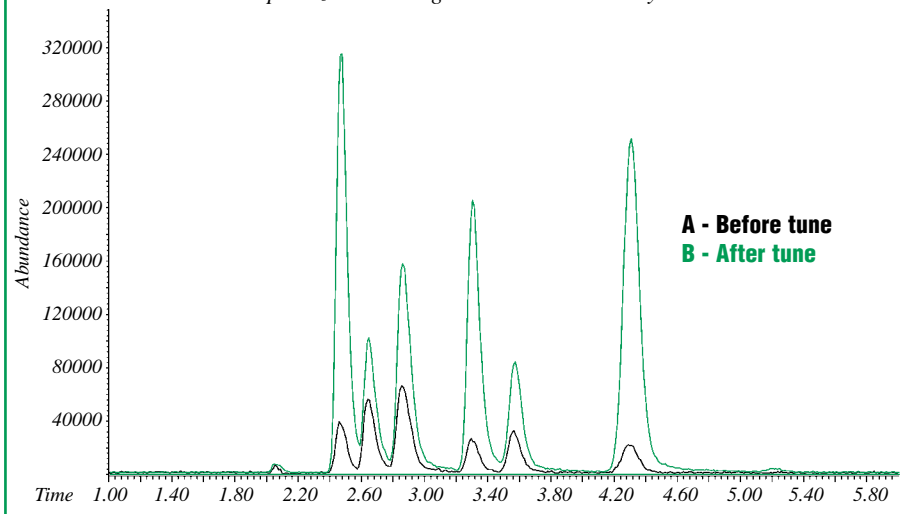
Figure 32.

Perfluorotributylamine (PFTBA) fragmentation, using a quadrupole mass spectrometer.

**Table IV.**

US EPA ion abundance criteria for 4-bromofluorobenzene (BFB).

Mass/Charge Ratio	Relative Abundance Criterion
50	15-50% of mass 95
75	30-80% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5-9% of mass 174
176	>95% but <101% of mass 174
177	5-9% of mass 176

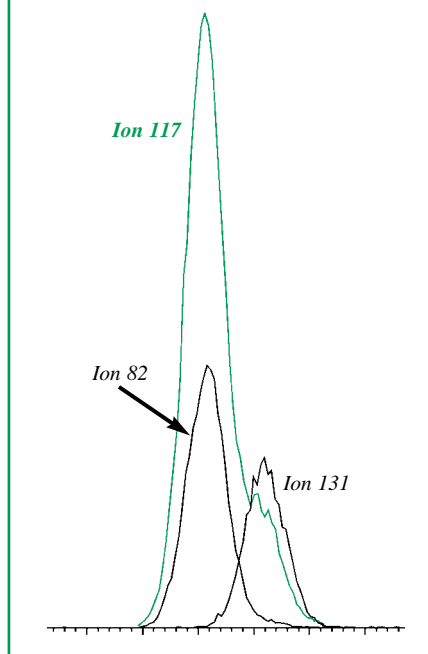
Figure 33.*Optimize MS tuning to increase sensitivity.*

Leak Checks: The MS is a powerful tool for determining the presence of leaks in the GC/MS system because it is able to detect air and water. Check for leaks by turning on the PFTBA valve and scanning for m/z 69, 18, and 28. By using the base peak for PFTBA (m/z 69), a relative concentration of water/nitrogen can be determined. The combined relative abundance of 18/28 to 69 should be between 0.1% and 3%. Figure 34 shows an air/water value of 0.14% (0.05+0.09). If the value is below 0.1%, compare the current total abundance of ion 69 with its abundance at the last leak check. Instruments with large leaks have reduced sensitivity for 69 and may show abundances of 0 for lower ions, suggesting there is no leak. This is due to saturation of the detector. If a leak is present, the instrument will not tune. An MS with a diffusion pump should be allowed more time to equilibrate because it is less efficient at removing low molecular weight contamination. After this preliminary air/water check, begin tuning the instrument. After the instrument passes tuning, check again for air and water.

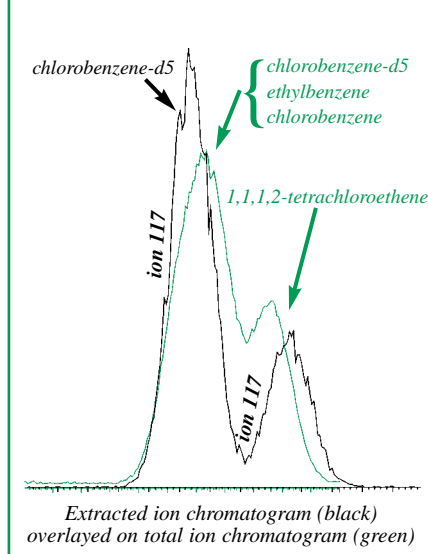
Figure 34.*An initial air/water check on an Agilent 5971A MS, before tuning.*

Figure 35.

The quantification ion for chlorobenzene-d5 can be changed from ion 117 to ion 82, to eliminate the need for chromatographic resolution from 1,1,1,2-tetrachloroethane.

**Figure 36.**

A slower oven temperature program eliminates the need to change the internal standard or the quantification ion.



Identifying Target Analytes

Qualitative identification of a target compound is based on retention time (± 0.06 minutes) and on comparison of the sample mass spectrum to a reference mass spectrum. Compounds are identified from three ions of the greatest intensity. The quantification ion, usually the highest m/z fragment, is used for determining the concentration of a particular analyte. When using any column for GC/MS, attention must be given to coeluting compounds to determine if acceptable quantification ions can be found. It is important that there be no coelution between compounds sharing ions used for quantification. As long as unique ions can be selected for quantifying compounds that share retention time, chromatographic coelution is acceptable. Reducing analysis time without carefully checking for coelutions can lead to problems. For example, internal standard chlorobenzene-d5 and analyte 1,1,1,2-tetrachloroethane, which share quantification ion 117, can coelute from a "VMS," "624," or "VRX" stationary phase. Many laboratories eliminate the need for chromatographic resolution by changing the quantification ion for chlorobenzene-d5 from 117 to 82 (Figure 35). An alternative solution is to replace chlorobenzene-d5 with another internal standard that elutes in the same region of the chromatogram, such as 4-bromofluorobenzene. In performance-based measurement systems (PBMS), surrogates and internal standards may be changed, as long as the analyst can show that the performance will be equivalent or better. Auditors for state or local regulatory agencies might not allow these changes, however. An additional option, the suggested surrogates can be used and the analysis performed using a slower GC oven temperature program that resolves the coelution (Figure 36). This option eliminates the need to change either the internal standard or the quantification ion, but prolongs analysis time. An analysis time of less than 10 minutes, with chromatographic resolution of these compounds, is possible with a 20m x 0.18mm x 1.0 μ m df Rtx®-VMS column (see Application section, page 51).

Identifying Non-Target Analytes

For samples containing analytes that do not match retention time and/or mass spectra for the target compounds, a library search can be used in an attempt to match the unknown spectra with known spectra. Unknown compounds in the sample, referred to as *tentatively identified compounds* (TICs), should be reported only as estimates.

Mass Spectral Clues for Identifying TICs: The MS provides three valuable clues to identifying TICs: parent ion, isotopic composition, and common fragmentation ions. The parent ion (also known as the molecular weight ion) is the ionized form of the neutral compound, but not all compounds are stable enough to produce a molecular weight ion. Most environmental contaminants, except compounds that contain nitrogen, will have an even number mass for a parent ion. The parent ion reveals information about the elemental composition and the distribution of isotopes. The term "isotope" is used to describe atoms of an element with differing numbers of neutrons. Most elements have isotopes in a particular distribution to each other. For example, carbon occurs primarily in two forms, ^{12}C and ^{13}C . ^{13}C is at an abundance of 1.1% relative to ^{12}C .¹² This information can contribute to determining the number of carbons present in the fragmented ion. Carbon is considered an A+1 element because its isotopes vary by 1amu. Compounds such as oxygen, sulfur, silicon, chlorine, and bromine are A+2 elements because their isotopes vary by 2amu or more (Figure 37, page 35). Fragmentation ions also can offer clues to compound composition (Table V).

Leak-Free Column/MS Installation Using an Injection Port Connection

The most common problem associated with volatiles analyses by GC/MS is the presence of leaks. The following procedure will help ensure optimum performance. Do not use this procedure with columns with IDs larger than 0.25mm, because the amount of oxygen that would be introduced into the MS source during the last step will oxidize the metal parts and reduce sensitivity.

12. F.W. McLafferty and F. Turecek. *Interpretation of Mass Spectra*, University Science Books, Mill Valley, 4th edition, 1993, pp. 283-291.

Connect the capillary column, 0.25mm ID or smaller, to the injection port, but not to the MS source, and condition the column. When the column has been conditioned, remove 50cm from the detector end of the column to ensure complete removal of siloxanes and other potential contaminants. Then, with the MS still turned off, insert the column end into the MS source. Cut the column several centimeters from the connection to the injection port. Use septa to cap the short length of column that is left in the injection port and the new, unconnected inlet end of the column. Also cap the split vent and septum purge vent lines on the GC (Figure 38). Perform a pressure decay test on the injection port by setting the pressure to 30psi, then shutting off the gas supply. The pressure should remain constant for at least 1 minute. If the pressure drops in less than 1 minute, turn on the gas supply and begin leak checking, using an electronic leak detector, such as the Restek Leak Detective™ II (cat. #20413, page 36).

Once you have confirmed the GC system is leak-tight, and while the injection port end of the column is capped and there is no flow in the column, evacuate the MS and record the source pressure in your maintenance logbook. After several hours equilibration, perform the instrument leak-check using PFTBA (see MS Calibration and Tuning, page 32). If a leak is present, draw 500µL of methanol into a syringe and apply drops of this solvent on areas where leaks might be suspected, while scanning for mass 31. Alternatively, bathe these areas with argon gas and scan for m/z 40. The brass source nut is the primary place for leaks in an MS, this nut should be replaced every time the column is changed (cat. #20643, page 35). Other areas to examine include the rubber seals and the PFTBA vial.

After you confirm the MS is leak-free, quickly install the inlet end of the column into the injection port. The MS will draw air into the source during this connection time; after another 2-hour equilibration the MS is ready for tuning/leak checking using PFTBA.

Figure 38.

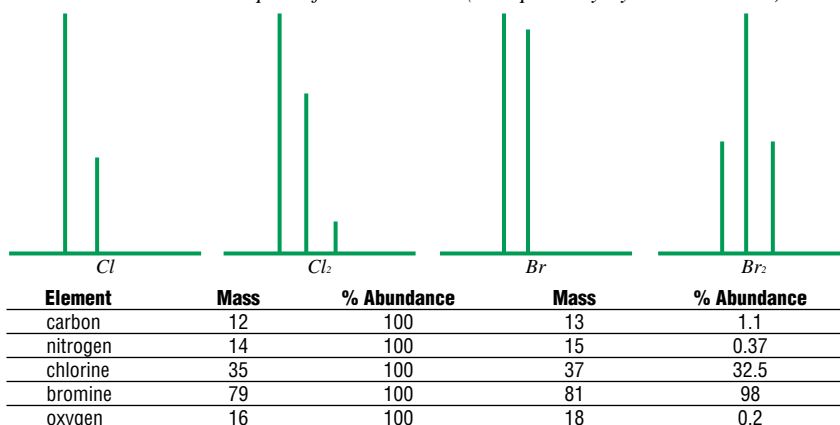
To check for leaks, cap the split vent and septum purge vent lines on the GC and the column, then shut off the gas supply. System pressure should remain constant.



Cap both

Figure 37.

Cl and Br are examples of A+2 elements (isotopes vary by 2amu or more).



MSD Source Nut

(Similar to Agilent part # 05988-20066.)

Description	qty.	cat.#
(Detector) MSD Source Nut	2-pk.	20643

Table V.

Fragment ions can offer clues to compound composition.

Compound Class	Fragment Ions
Aldehydes, amides, amines	44, 58, 72, 59, 30
Aliphatic hydrocarbons	43, 57, 71, 85, 99
Alkylbenzenes	104, 91
Aromatic hydrocarbons	39, 50, 51, 52, 63, 65, 76, 77, 91
Fluorine-containing	50, 69
Methacrylates	41, 69
Methyl ketones	43, 58
Oxygen-containing	31, 45, 59, 73
Sulfur-containing	47, 61
Unsaturated hydrocarbons	41, 55, 69

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Leak Detective™ II Leak Detector

- Affordable thermal conductivity leak detector—every analyst should have one*
- Compact, ergonomic design is easy to hold and operate with one hand.
- Helium, hydrogen, and nitrogen can be detected at 1×10^{-4} cc/sec. or at an absolute concentration as low as 100ppm**
- Fast results—responds in less than 2 seconds to trace leaks of gases with thermal conductivities different than air.
- Micro-chip design improves sensitivity and response time over previous models.
- Auto zeroing with the touch of a button.
- Battery-operated for increased portability (one 9-volt).



Description	qty.	cat.#
Leak Detective™ II Leak Detector	ea.	20413

* *Never use liquid leak detectors on a capillary system. Liquids can be drawn into the system.*

** **Caution:** *NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possibly hazardous conditions.*

MS Contamination

A universal detector, the MS responds to all organic compounds and, consequently, any contamination potentially can interfere with target analyte identification. The common sources of contamination are column bleed and septum bleed. High column bleed can be an indication of oxygen entering the system and degrading the stationary phase in the column. If high column bleed is suspected, conduct a leak check of the system, using the procedure described on page 34 and 35. Septum bleed introduces silicon fragments, characterized by ion 73, into the system. Silicon compounds also are components of GC and MS seals. Table VI lists common contaminants and the ions by which they can be identified.

Water / Methanol: The introduction of water and/or methanol vapor from the purge and trap system can cause problems in an MS system. Excess water vapor entering the MS can decrease the ionization of target analytes eluting at the same time. To overcome problems associated with water vapor, use a trap containing hydrophobic adsorbents, such as the Vocab® 3000 trap. If you are using an ion trap system, increase the split ratio in the injection port. This will prevent overloading the ion trap and will increase overall linearity for the gaseous analytes.

Table VI.

Common contaminants and their identifying ions.

Contaminant	Characteristic Fragmentation Ions
Silicon	73, 147, 207, 221, 281, 355, 429, 503
Rough vacuum pump oil	55-57, 61-67, 81-85, 95-99
Diffusion pump oil	77, 115, 141, 168, 223, 260, 446
Plasticizers	149, 223, 278

Practical Introduction to GC/MS Analysis with Quadrupoles

The text gives answers to questions such as: how does the mass spectrometer work, what problems can occur and how do I detect them, how must separation and detection be adapted to each other, and what pitfalls can be avoided when elucidating structures and quantifying compounds.

M. Oehme, Wiley-VCH

1999, 195pp., ISBN 3-527-29748-0

cat.# 21098



Interpretation of Mass Spectra, 4th Edition

This updated version builds on the strengths of the previous editions and presents the information required to clearly and concisely interpret mass spectra. Chapters include information on elemental composition, molecular ions, mechanisms of ion fragmentations, unimolecular ion decompositions, and mass spectra of common compound classes. It is valuable and necessary resource for every person practicing mass spectrometry.

F.W. McLafferty and F. Turecek, University Science

1993, 371pp., ISBN 0-935702-25-3

cat.# 20498



Applications Using GC Detection Systems

Purge and Trap Applications Using Tandem PID-ELCD: US EPA Methods 502.2, 601, 602, 8010, 8020, 8021B

EPA methods for GC analyses of volatile compounds require purge and trap units for concentrating the contaminants in water, soil, or wastewater. While purge and trap concentration significantly increases sensitivity, relative to other sample introduction techniques, it does have a downside: early-eluting volatile compounds typically exhibit broad peaks, due to inefficient sample transfer from the trap to the GC. This distorted peak shape decreases resolution between closely eluting compounds, placing demands on the analytical system and requiring optimized GC operating conditions. Although cryofocusing improves separations of early eluting compounds, most environmental laboratories do not use this approach because it increases costs.

EPA methods for monitoring volatiles by GC often recommend using a PID and an ELCD, connected in tandem or series. Coelutions of target compounds are allowed, as long as they are resolved by the detectors.¹³ For example, in Figure 39 bromoform and styrene elute with the same retention time, but bromoform elicits a response only from the ELCD and styrene elicits a response only from the PID. Thus, the selective detectors resolve these two compounds. Because it characteristically produces tailing peaks, the ELCD is the more problematic of the two detectors; sensitivity can be increased, but not without a sacrifice in peak shape. Optimization of an ELCD minimizes tailing and maximizes sensitivity.

Analysis Time: Several factors contribute to the total analysis time for volatiles separations, including purge and trap cycle time, sample analysis time, and GC oven cool-down time (time required for the oven to cool from the final temperature to the initial temperature for the next analysis). Long purge and trap cycles are a product of long purge times, dry purges, long desorb times, and long trap bake times. Long oven cycle times result from low initial oven temperatures (i.e., subambient to 35°C) and slow temperature program rates. A column that unnecessarily exceeds the length needed to resolve the analytes can increase analysis time and cost without significantly adding to the data obtained.

An Rtx®-VGC primary column paired with an Rtx®-VRX confirmation column make a good combination for analyzing the compounds listed in Figures 39A & B. The target list includes unregulated but commonly analyzed compounds such as methyl-*tert*-butyl ether (MTBE) and Freon® 113 (1,1,2-trichloro-1,2,2-trifluoroethane). A 35°C starting temperature is necessary to resolve Freon® 113 from 1,1-dichloroethane. Figure 39A shows there are no early-analyte coelution problems on the primary column when using PID/ELCD detectors in tandem – the gases and the trihalomethanes are separated.

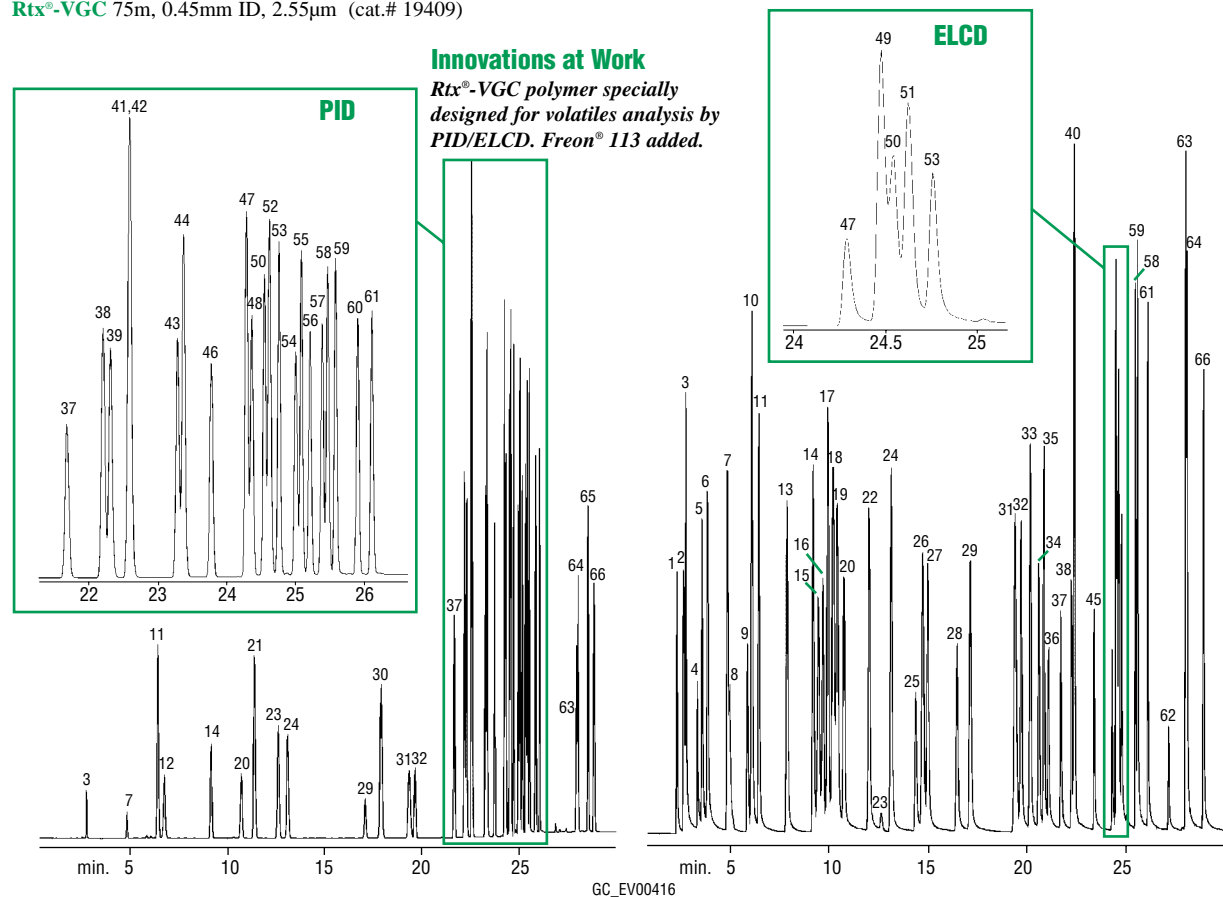
Figures 40A & B show the analysis of Method 8021A/502.2 compounds, without Freon® 113, using an Rtx®-VGC column and an Rtx®-502.2 column. A 50°C initial oven temperature can be used, which greatly reduces the time needed for the GC to complete the oven cycle and return to the starting temperature (cycle time) and, therefore, increases throughput. An Agilent 5890 GC oven will cool from 205°C to 35°C in 9 minutes; this time, added to the 28-minute analysis time in Figure 39, produces the fastest cycle time for this analysis: 37 minutes. In the analysis in Figure 40, the starting temperature is 50°C, the final temperature is 200°C, and the oven takes 4 minutes to cool. The total cycle time, less than 30 minutes, is significantly faster than for other pairs of columns. For example, an Rtx®-VRX column requires a starting temperature of 40°C; this, combined with a 28 minute analysis time, means the total cycle time cannot be faster than 35 minutes.

13. EPA Method 8000B, *Determinative Chromatographic Separations*; US EPA. U.S. Government Printing Office: Washington, DC, 1996, Rev. 2.

Figure 39A.

An Rtx®-VGC primary column and an Rtx®-VRX confirmation column separate volatile organic gases from the trihalomethanes – there are no coelutions on the primary column using the PID/ELCD detectors in tandem.

Rtx®-VGC 75m, 0.45mm ID, 2.55µm (cat.# 19409)



20ppb in 5mL of RO water.

Primary column: 75m, 0.45mm ID, 2.55 m Rtx®-VGC (cat.# 19409)

Confirmation column: 75m, 0.45mm ID, 2.55 m Rtx®-VRX (cat.# 19309)

Concentrator: Tekmar LSC-3000 Purge and Trap

Trap: Vocarb 3000

Purge: 11 min. @ 40mL/min.

Dry purge: 1 min. @ 40mL/min. (MCS by-passed with Silcosteel® tubing [cat.# 21035])

Desorb preheat: 245 C

Desorb: 250 C for 2 min.

Bake: 260 C for 8 min.

Interface: direct

Transfer line: 0.32mm ID Siltek tubing (Cat. #10027)

GC: Finnigan 9001

Oven temp.: 35 C (hold 4 min.) to 75 C @ 3 C/min. (hold 2 min.)

to 175 C @ 21 C/min. to 205 C @ 35 C/min. (hold 5 min.)

Carrier gas: helium 11mL/min., constant pressure

Adjust dichlorodifluoromethane to a retention time of 2.28 min. @ 35 C on the Rtx®-VGC column.

Detectors: Gold Tandem PID/HALL 2000 ELCD

PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV, base temp 200 C.

Hall 2000 ELCD: RxnGas 25mL/min., RxnTemp. 940 C, propanol flow 470 L/min.

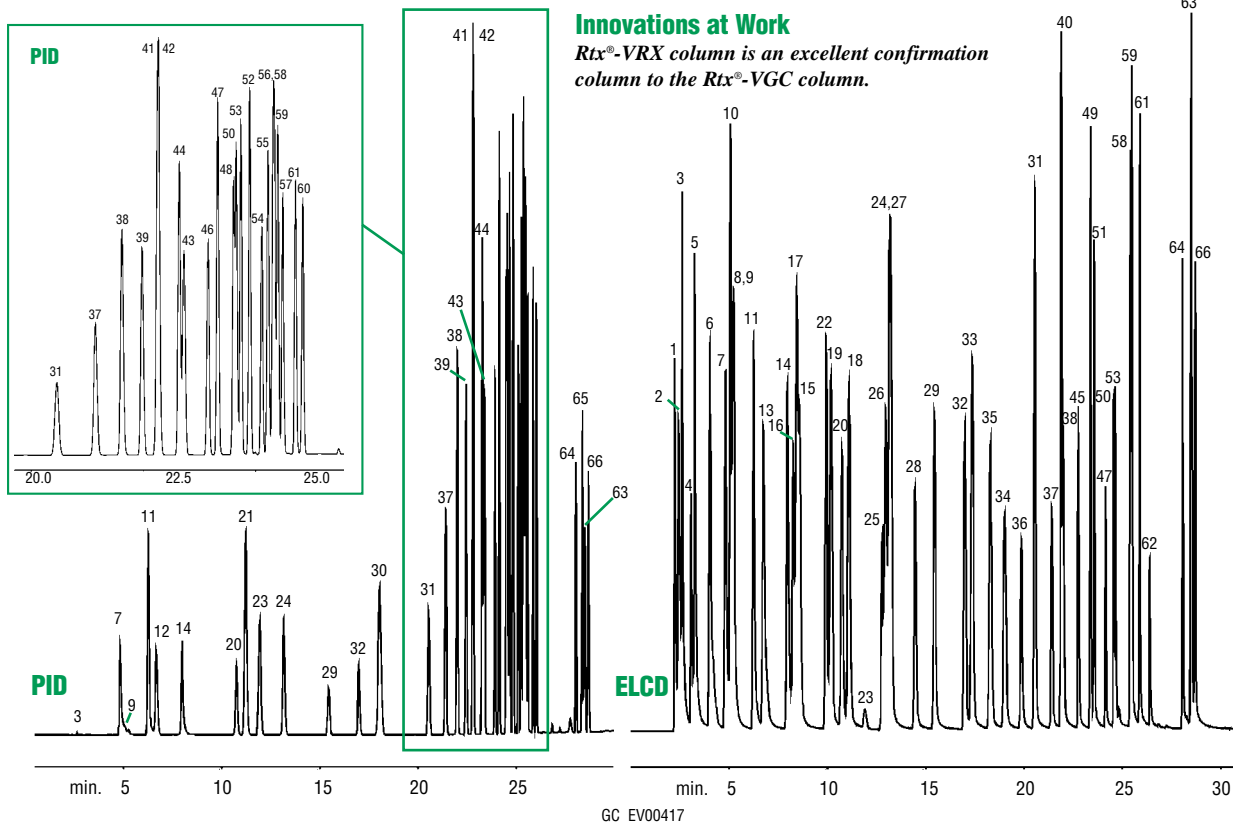
- | | | | |
|--------------------------------------|---------------------------------------|-----------------------------------|---------------------------------|
| 1. dichlorodifluoromethane | 18. carbon tetrachloride | 35. 1,3-dichloropropane | 52. 1,3,5-trimethylbenzene |
| 2. chloromethane | 19. 1,1,1-trichloroethane | 36. 1,2-dibromoethane | 53. 4-chlorotoluene |
| 3. vinyl chloride | 20. 1,1-dichloropropene | 37. 1-chloro-3-fluorobenzene (SS) | 54. <i>tert</i> -butylbenzene |
| 4. bromomethane | 21. benzene | 38. chlorobenzene | 55. 1,2,4-trimethylbenzene |
| 5. chloroethane | 22. 1,2-dichloroethane | 39. ethylbenzene | 56. <i>sec</i> -butylbenzene |
| 6. trichlorofluoromethane | 23. fluorobenzene (SS) | 40. 1,1,1,2-tetrachloroethane | 57. <i>p</i> -isopropyltoluene |
| 7. 1,1-dichloroethene | 24. trichloroethene | 41. <i>m</i> -xylene | 58. 1,3-dichlorobenzene |
| 8. Freon® 113 | 25. dibromomethane | 42. <i>p</i> -xylene | 59. 1,4-dichlorobenzene |
| 9. allyl chloride | 26. 1,2-dichloropropane | 43. <i>o</i> -xylene | 60. <i>n</i> -butylbenzene |
| 10. methylene chloride | 27. bromodichloromethane | 44. styrene | 61. 1,2-dichlorobenzene |
| 11. <i>trans</i> -1,2-dichloroethene | 28. 1-bromo-2-chloroethane (SS) | 45. bromoform | 62. 1,2-dibromo-3-chloropropane |
| 12. methyl <i>tert</i> -butyl ether | 29. <i>cis</i> -1,3-dichloropropene | 46. isopropylbenzene | 63. hexachlorobutadiene |
| 13. 1,1-dichloroethane | 30. toluene | 47. bromobenzene | 64. 1,2,4-trichlorobenzene |
| 14. <i>cis</i> -1,2-dichloroethene | 31. tetrachloroethene | 48. <i>n</i> -propylbenzene | 65. naphthalene |
| 15. 2,2-dichloropropane | 32. <i>trans</i> -1,3-dichloropropene | 49. 1,1,2,2-tetrachloroethane | 66. 1,2,3-trichlorobenzene |
| 16. bromochloromethane | 33. 1,1,2-trichloroethane | 50. 2-chlorotoluene | |
| 17. chloroform | 34. dibromochloromethane | 51. 1,2,3-trichloropropane | |

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL® 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

Figure 39B.

An Rtx®-VGC primary column and an Rtx®-VRX confirmation column separate volatile organic gases from the trihalomethanes – there are no coelutions on the primary column using the PID/ELCD detectors in tandem.

Rtx®-VRX 75m, 0.45mm ID, 2.55µm (cat.# 19309)



Innovations at Work

Rtx®-VRX column is an excellent confirmation column to the Rtx®-VGC column.

20ppb in 5mL of RO water.

Primary column: 75m, 0.45mm ID, 2.55µm Rtx®-VGC (cat.# 19409)

Confirmation column: 75m, 0.45mm ID, 2.55µm Rtx®-VRX (cat.# 19309)

Concentrator: Tekmar LSC-3000 Purge and Trap

Trap: Vocab® 3000

Purge: 11 min. @ 40mL/min.

Dry purge: 1 min. @ 40mL/min. (MCS by-passed with Silcosteel® tubing (cat.# 21035))

Desorb preheat: 245°C

Desorb: 250°C for 2 min.

Bake: 260°C for 8 min.

Interface: direct

Transfer line: 0.32mm ID Siltek™ tubing (Cat. #10027)

GC: Finnigan 9001

Oven temp.: 35°C (hold 4 min.) to 75°C @ 3°C/min. (hold 2 min.) to 175°C @ 21°C/min. to 205°C @ 35°C/min. (hold 5 min.)

Carrier: helium 11mL/min., constant pressure
Adjust dichlorodifluoromethane to a retention time of 2.28 min. @ 35°C on the Rtx®-VGC column.

Detectors: µGold Tandem PID/HALL 2000 ELCD

PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV, base temp 200°C.
Hall 2000 ELCD: RxnGas 25mL/min., RxnTemp. 940°C, propanol flow 470µL/min.

1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethene
8. Freon® 113
9. allyl chloride
10. methylene chloride
11. *trans*-1,2-dichloroethene
12. methyl *tert*-butyl ether
13. 1,1-dichloroethane
14. *cis*-1,2-dichloroethene
15. 2,2-dichloropropane
16. bromochloromethane
17. chloroform

18. carbon tetrachloride
19. 1,1,1-trichloroethane
20. 1,1-dichloropropene
21. benzene
22. 1,2-dichloroethane
23. fluorobenzene (SS)
24. trichloroethene
25. dibromomethane
26. 1,2-dichloropropane
27. bromodichloromethane
28. 1-bromo-2-chloroethane (SS)
29. *cis*-1,3-dichloropropene
30. toluene
31. tetrachloroethene
32. *trans*-1,3-dichloropropene
33. 1,1,2-trichloroethane
34. dibromochloromethane

35. 1,3-dichloropropane
36. 1,2-dibromoethane
37. 1-chloro-3-fluorobenzene (SS)
38. chlorobenzene
39. ethylbenzene
40. 1,1,1,2-tetrachloroethane
41. *m*-xylene
42. *p*-xylene
43. *o*-xylene
44. styrene
45. bromoform
46. isopropylbenzene
47. bromobenzene
48. *n*-propylbenzene
49. 1,1,2,2-tetrachloroethane
50. 2-chlorotoluene
51. 1,2,3-trichloropropane

52. 1,3,5-trimethylbenzene
53. 4-chlorotoluene
54. *tert*-butylbenzene
55. 1,2,4-trimethylbenzene
56. *sec*-butylbenzene
57. *p*-isopropyltoluene
58. 1,3-dichlorobenzene
59. 1,4-dichlorobenzene
60. *n*-butylbenzene
61. 1,2-dichlorobenzene
62. 1,2-dibromo-3-chloropropane
63. hexachlorobutadiene
64. 1,2,4-trichlorobenzene
65. naphthalene
66. 1,2,3-trichlorobenzene

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL® 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

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Figure 40A.

An Rtx®-VGC / Rtx®-502.2 column pair and a 50°C initial temperature reduce total cycle time to less than 30 minutes for EPA Method 8021A/502.2.

Innovations at Work

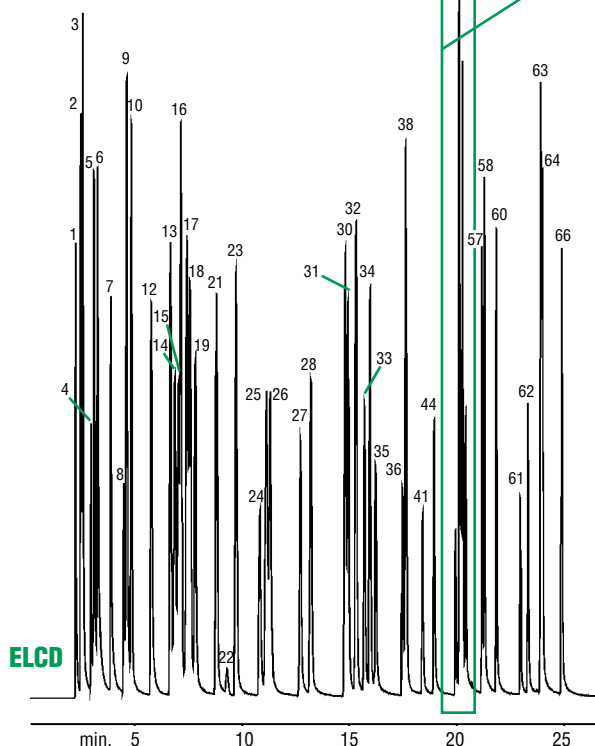
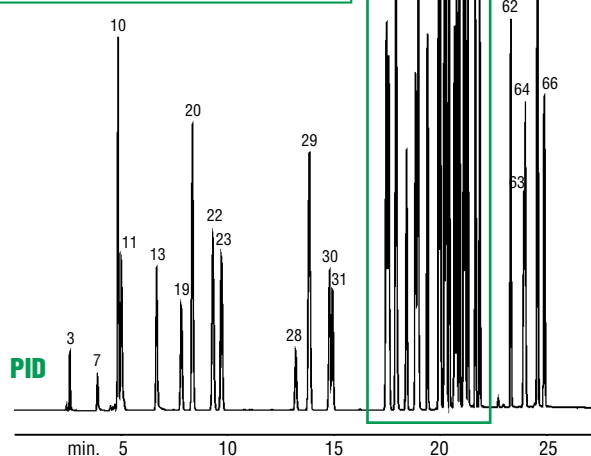
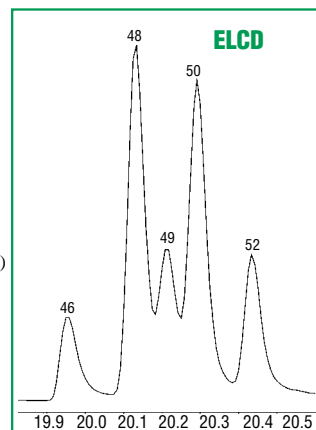
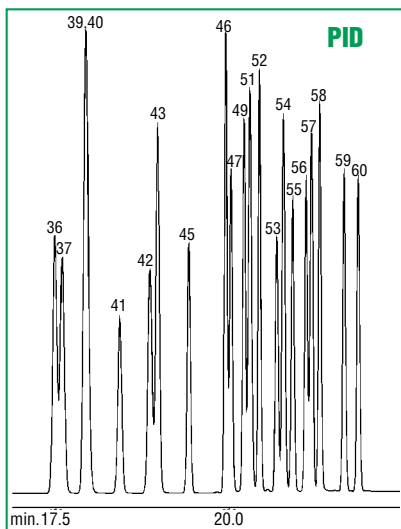
Fastest cycle time for 8021/502.2!

50°C starting temperature brings total cycle time to less than 30 min.

Rtx®-VGC 75m, 0.45mm ID, 2.55µm (cat.# 19409)

Primary column: 75m, 0.45mm ID, 2.55 m Rtx®-VGC (cat.# 19409)
 Confirmation column: 75m, 0.45mm ID, 2.55 m Rtx®-502.2 (cat.# 10986)
 Conc.: 10ppb in 5mL of RO water
 Concentrator: Tekmar LSC3100 Purge and Trap
 Trap: Vocab 3000
 Purge: 11 min. @ 40mL/min.
 Dry purge: 1 min. @ 40mL/min.
 Desorb preheat: 245 C
 Desorb: 250 C for 2 min.
 Bake: 260 C for 8 min.
 Interface: direct connection from concentrator to column
 Transfer line: Siltek 0.32mm fused silica transfer line direct to columns w/ Press-Tight Y connector (cat. # 20403)

Gas chromatograph: Finnigan 9001
 Carrier gas: helium @ ~10mL/min. constant pressure
 Adjust dichlorodifluoromethane to a retention time of 2.28 min. @ 50 C on the Rtx®-VGC column.
 50 C (hold 2 min.) to 70 C @ 2 C/min. to 130 C @ 200 C @ 40 C/min. (final hold 5 min.)
 Gold Tandem PID/Hall 2000 ELCD
 PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV base temp 200 C.
 Hall 2000 ELCD: RxnGas 25mL/min., Temp.940 C, propanol flow 470 L/min.



- | | | | | |
|--------------------------------------|-------------------------------------|---------------------------------------|--------------------------------|----------------------------------|
| 1. dichlorodifluoromethane | 15. bromochloromethane | 29. toluene | 43. styrene | 57. 1,3-dichlorobenzene |
| 2. chloromethane | 16. chloroform | 30. tetrachloroethene | 44. bromoform | 58. 1,4-dichlorobenzene |
| 3. vinyl chloride | 17. carbon tetrachloride | 31. <i>trans</i> -1,3-dichloropropene | 45. isopropylbenzene | 59. <i>n</i> -butylbenzene |
| 4. bromomethane | 18. 1,1,1-trichloroethane | 32. 1,1,2-trichloroethane | 46. bromobenzene | 60. 1,2-dichlorobenzene |
| 5. chloroethane | 19. 1,1-dichloropropene | 33. dibromochloromethane | 47. <i>n</i> -propylbenzene | 61. 1,2-dibromo-3-chloropropane |
| 6. trichlorofluoromethane | 20. benzene | 34. 1,3-dichloropropane | 48. 1,1,2,2-tetrachloroethane | 62. 2-bromo-1-chlorobenzene (SS) |
| 7. 1,1-dichloroethene | 21. 1,2-dichloroethane | 35. 1,2-dibromoethane | 49. 2-chlorotoluene | 63. hexachlorobutadiene |
| 8. allyl chloride | 22. fluorobenzene (SS) | 36. chlorobenzene | 50. 1,2,3-trichloropropane | 64. 1,2,4-trichlorobenzene |
| 9. methylene chloride | 23. trichloroethene | 37. ethylbenzene | 51. 1,3,5-trimethylbenzene | 65. naphthalene |
| 10. <i>trans</i> -1,2-dichloroethene | 24. dibromomethane | 38. 1,1,1,2-tetrachloroethane | 52. 4-chlorotoluene | 66. 1,2,3-trichlorobenzene |
| 11. methyl <i>tert</i> -butyl ether | 25. 1,2-dichloropropane | 39. <i>m</i> -xylene | 53. <i>tert</i> -butylbenzene | |
| 12. 1,1-dichloroethane | 26. bromodichloromethane | 40. <i>p</i> -xylene | 54. 1,2,4-trimethylbenzene | |
| 13. <i>cis</i> -1,2-dichloroethene | 27. 1-bromo-2-chloroethane (SS) | 41. 1-chloro-2-fluorobenzene (SS) | 55. <i>sec</i> -butylbenzene | |
| 14. 2,2-dichloropropane | 28. <i>cis</i> -1,3-dichloropropene | 42. <i>o</i> -xylene | 56. <i>p</i> -isopropyltoluene | |

GC_EV00418

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL® 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

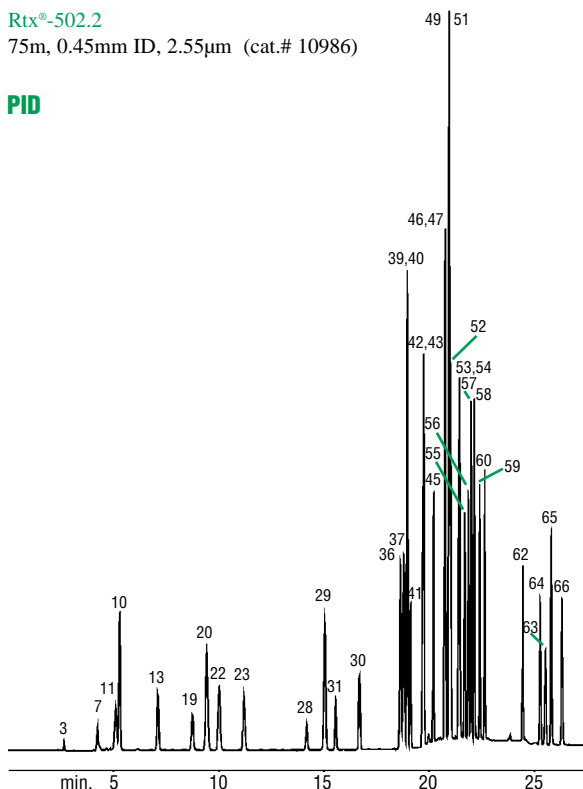
Figure 40B.

An Rtx®-VGC / Rtx®-502.2 column pair and a 50°C initial temperature reduce total cycle time to less than 30 minutes for EPA Method 8021A/502.2.

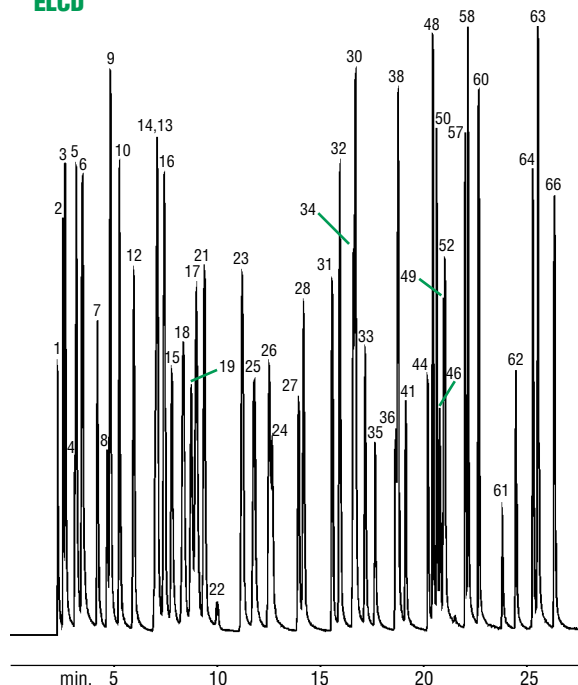
Rtx®-502.2

75m, 0.45mm ID, 2.55µm (cat.# 10986)

PID



ELCD



GC_EV00419

- | | |
|---------------------------------------|-----------------------------------|
| 1. dichlorodifluoromethane | 34. 1,3-dichloropropane |
| 2. chloromethane | 35. 1,2-dibromoethane |
| 3. vinyl chloride | 36. chlorobenzene |
| 4. bromomethane | 37. ethylbenzene |
| 5. chloroethane | 38. 1,1,1,2-tetrachloroethane |
| 6. trichlorofluoromethane | 39. <i>m</i> -xylene |
| 7. 1,1-dichloroethene | 40. <i>p</i> -xylene |
| 8. allyl chloride | 41. 1-chloro-2-fluorobenzene (SS) |
| 9. methylene chloride | 42. <i>o</i> -xylene |
| 10. <i>trans</i> -1,2-dichloroethene | 43. styrene |
| 11. methyl <i>tert</i> -butyl ether | 44. bromoform |
| 12. 1,1-dichloroethane | 45. isopropylbenzene |
| 13. <i>cis</i> -1,2-dichloroethene | 46. bromobenzene |
| 14. 2,2-dichloropropane | 47. <i>n</i> -propylbenzene |
| 15. bromochloromethane | 48. 1,1,2,2-tetrachloroethane |
| 16. chloroform | 49. 2-chlorotoluene |
| 17. carbon tetrachloride | 50. 1,2,3-trichloropropane |
| 18. 1,1,1-trichloroethane | 51. 1,3,5-trimethylbenzene |
| 19. 1,1-dichloropropene | 52. 4-chlorotoluene |
| 20. benzene | 53. <i>tert</i> -butylbenzene |
| 21. 1,2-dichloroethane | 54. 1,2,4-trimethylbenzene |
| 22. fluorobenzene (SS) | 55. <i>sec</i> -butylbenzene |
| 23. trichloroethene | 56. <i>p</i> -isopropyltoluene |
| 24. dibromomethane | 57. 1,3-dichlorobenzene |
| 25. 1,2-dichloropropane | 58. 1,4-dichlorobenzene |
| 26. bromodichloromethane | 59. <i>n</i> -butylbenzene |
| 27. 1-bromo-2-chloroethane (SS) | 60. 1,2-dichlorobenzene |
| 28. <i>cis</i> -1,3-dichloropropene | 61. 1,2-dibromo-3-chloropropane |
| 29. toluene | 62. 2-bromo-1-chlorobenzene (SS) |
| 30. tetrachloroethene | 63. hexachlorobutadiene |
| 31. <i>trans</i> -1,3-dichloropropene | 64. 1,2,4-trichlorobenzene |
| 32. 1,1,2-trichloroethane | 65. naphthalene |
| 33. dibromochloromethane | 66. 1,2,3-trichlorobenzene |

Primary column:
Confirmation column:
Conc.:
Concentrator:
Trap:
Purge:
Dry purge:
Desorb preheat:
Desorb:
Bake:
Interface:
Transfer line:

Gas chromatograph:
Carrier gas:

Oven temp.:

Detectors:

75m, 0.45mm ID, 2.55µm Rtx®-VGC (cat.# 19409)
75m, 0.45mm ID, 2.55µm Rtx®-502.2 (cat.# 10986)
10ppb in 5mL of RO water
Tekmar LSC3100 Purge and Trap
Vocarb 3000
11 min. @ 40mL/min.
1 min. @ 40mL/min.
245°C
250°C for 2 min.
260°C for 8 min.
direct connection from concentrator to column
Siltek 0.32mm fused silica transfer line direct to
columns w/ Press-Tight "Y" connector (cat. #20403)

Finnigan 9001
helium @ ~10 mL/min. constant pressure
Adjust dichlorodifluoromethane to a retention time of
2.28 min. @ 50°C on the Rtx®-VGC column.
50°C (hold 2 min.) to 70°C @ 2°C/min. to 130°C @ 9°C/min.
to 200°C @ 40°C/min. (final hold 5 min.)
µGold Tandem PID/Hall 2000 ELCD
PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV
base temp 200°C.
Hall 2000 ELCD: RxnGas 25mL/min., RxnTemp. 940°C,
propanol flow 470µL/min.

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL® 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

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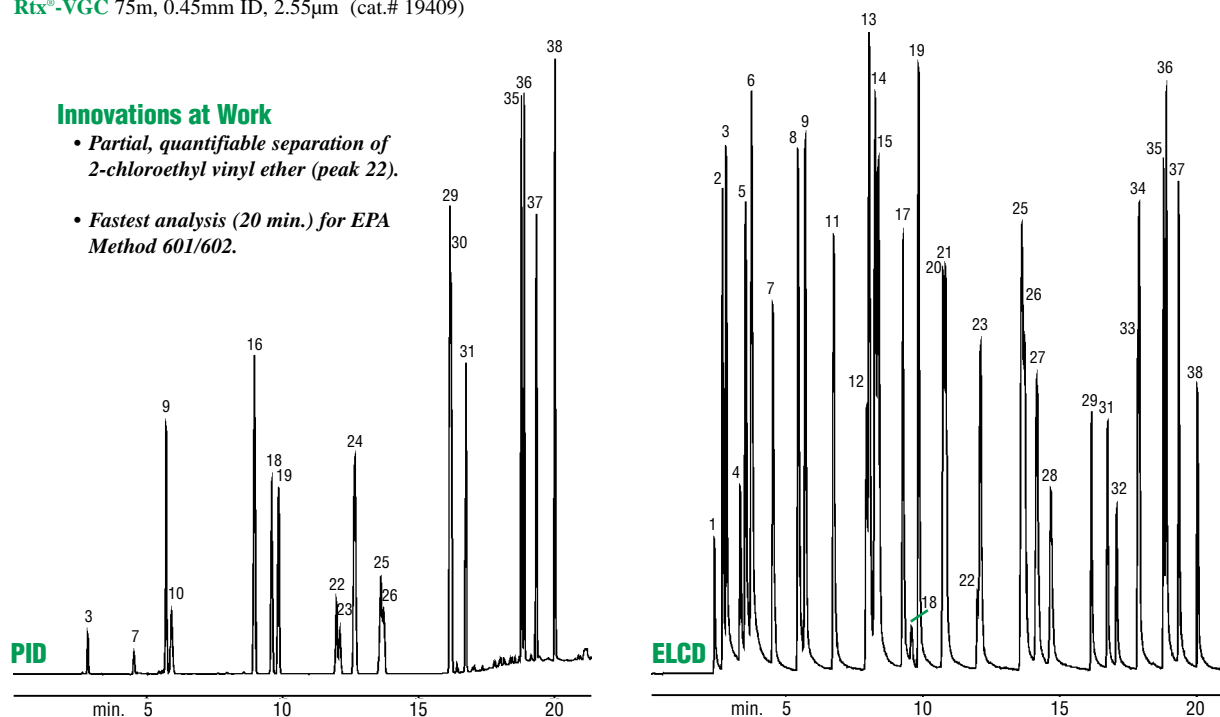
Figure 41A.

Analytes listed in EPA Method 601/602 can be separated in 20 minutes by an Rtx®-VGC / Rtx®-502.2 column pair.

Rtx®-VGC 75m, 0.45mm ID, 2.55µm (cat.# 19409)

Innovations at Work

- Partial, quantifiable separation of 2-chloroethyl vinyl ether (peak 22).
- Fastest analysis (20 min.) for EPA Method 601/602.



GC_EV00420

- | | |
|-------------------------------------|---------------------------------------|
| 1. dichlorodifluoromethane | 20. 1,2-dichloropropane |
| 2. chloromethane | 21. bromodichloromethane |
| 3. vinyl chloride | 22. 2-chloroethyl vinyl ether |
| 4. bromomethane | 23. <i>cis</i> -1,3-dichloropropene |
| 5. chloroethane | 24. toluene |
| 6. trichlorofluoromethane | 25. tetrachloroethene |
| 7. 1,1-dichloroethene | 26. <i>trans</i> -1,3-dichloropropene |
| 8. methylene chloride | 27. 1,1,2-trichloroethane |
| 9. <i>trans</i> -1,2-dichloroethene | 28. dibromochloromethane |
| 10. methyl <i>tert</i> -butyl ether | 29. chlorobenzene |
| 11. 1,1-dichloroethane | 30. ethylbenzene |
| 12. bromochloromethane (SS) | 31. 1-chloro-2-fluorobenzene (SS) |
| 13. chloroform | 32. bromoform |
| 14. carbon tetrachloride | 33. 1,4-dichlorobutane (SS) |
| 15. 1,1,1-trichloroethane | 34. 1,1,2,2-tetrachloroethane |
| 16. benzene | 35. 1,3-dichlorobenzene |
| 17. 1,2-dichloroethane | 36. 1,4-dichlorobenzene |
| 18. fluorobenzene (SS) | 37. 1,2-dichlorobenzene |
| 19. trichloroethene | 38. 4-bromo-1-chlorobenzene (SS) |

suggested surrogates: peaks 12, 18, 31, & 38

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL® 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

Primary column: 75m, 0.45mm ID, 2.55µm Rtx®-VGC (cat.# 19409)
 Confirmation column: 75m, 0.45mm ID, 2.55µm Rtx®-502.2 (cat.# 10986)
 Conc.: 10ppb in 5mL of RO water
 Concentrator: Tekmar LSC3100 Purge and Trap
 Trap: Vocab 3000
 Purge: 11 min. @ 40mL/min.
 Dry purge: 1 min. @ 40mL/min.
 Desorb preheat: 245°C
 Desorb: 250°C for 2 min.
 Bake: 260°C for 8 min.
 Interface: direct connection from concentrator to column
 Transfer line: 0.53mm ID Silcosteel® tubing (cat. #70045)
 Gas chromatograph: Finnigan 9001
 Carrier gas: helium @ ~10mL/min. constant pressure
 Adjust dichlorodifluoromethane to a retention time of 2.47 min. @ 40°C.
 Oven temp.: 40°C (hold 2 min.) to 58°C @ 4°C/min. to 90°C @ 10°C/min. (hold 5 min.) to 220°C @ 40°C/min. (hold 5 min.)
 Detectors: µGold Tandem PID/Hall 2000 ELCD
 PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV base temp: 200 C.
 Hall 2000 ELCD: RxnGas 25mL/min., Rxn Temp. 940 C, propanol flow 470 L/min.

Standard
 502.2 mix#1
 624 cal mix #2
 624 cal mix #3
 MTBE
 1,4-dichlorobutane

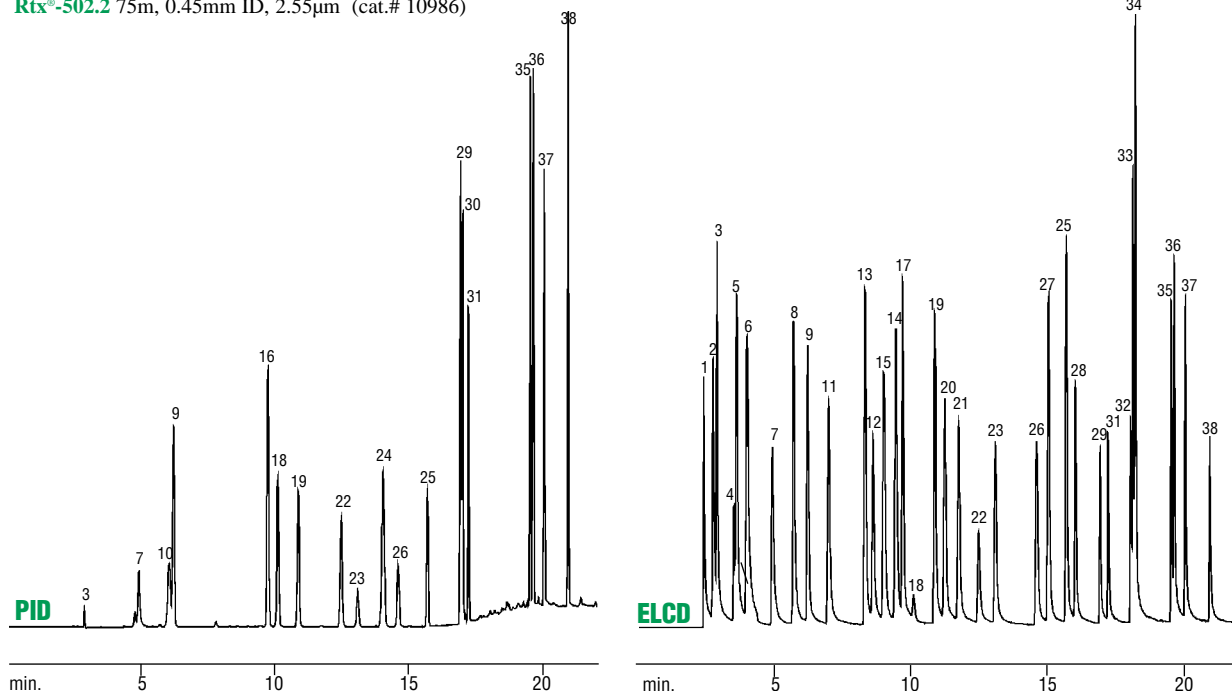
cat.#	Standard	cat.#
30042	fluorobenzene	30030
30021	1-chloro-2-fluorobenzene	30040
30022	4-bromo-1-chlorobenzene	30230
30402	bromochloromethane	30225
30227		

Client compound lists might match the compound list in Method 601/602, but the calibration criteria and low detection limits of Method 8021 are enforced. Figures 41A & B illustrate common compounds analyzed by GC/PID-ELCD, along with 2-chloroethyl vinyl ether. The starting temperature is 40°C, and the analysis time is 20 minutes. An Rtx®-502.2 column is a good choice for this analysis because it exhibits good resolution for 2-chloroethyl vinyl ether.

Figure 41B.

Analytes listed in EPA Method 601/602 can be separated in 20 minutes by an Rtx®-VGC / Rtx®-502.2 column pair.

Rtx®-502.2 75m, 0.45mm ID, 2.55µm (cat.# 10986)



GC_EV00540

- | | |
|-------------------------------------|---------------------------------------|
| 1. dichlorodifluoromethane | 21. bromodichloromethane |
| 2. chloromethane | 22. 2-chloroethyl vinyl ether |
| 3. vinyl chloride | 23. <i>cis</i> -1,3-dichloropropene |
| 4. bromomethane | 24. toluene |
| 5. chloroethane | 25. tetrachloroethene |
| 6. trichlorofluoromethane | 26. <i>trans</i> -1,3-dichloropropene |
| 7. 1,1-dichloroethene | 27. 1,1,2-trichloroethane |
| 8. methylene chloride | 28. dibromochloromethane |
| 9. <i>trans</i> -1,2-dichloroethene | 29. chlorobenzene |
| 10. methyl <i>tert</i> -butyl ether | 30. ethylbenzene |
| 11. 1,1-dichloroethane | 31. 1-chloro-2-fluorobenzene (SS) |
| 12. bromochloromethane (SS) | 32. bromoform |
| 13. chloroform | 33. 1,4-dichlorobutane |
| 14. carbon tetrachloride | 34. 1,1,2,2-tetrachloroethane |
| 15. 1,1,1-trichloroethane | 35. 1,3-dichlorobenzene |
| 16. benzene | 36. 1,4-dichlorobenzene |
| 17. 1,2-dichloroethane | 37. 1,2-dichlorobenzene |
| 18. fluorobenzene (SS) | 38. 4-bromo-1-chlorobenzene (SS) |
| 19. trichloroethene | |
| 20. 1,2-dichloropropane | |

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL[®] 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

Primary column: 75m, 0.45mm ID, 2.55 m Rtx®-VGC (cat.# 19409)
 Confirmation column: 75m, 0.45mm ID, 2.55 m Rtx®-502.2 (cat.# 10986)
 Conc.: 10ppb in 5mL of RO water
 Concentrator: Tekmar LSC3100 Purge and Trap
 Trap: Vocarb 3000
 Purge: 11 min. @ 40mL/min.
 Dry purge: 1 min. @ 40mL/min.
 Desorb preheat: 245 C
 Desorb: 250 C for 2 min.
 Bake: 260 C for 8 min.
 Interface: direct connection from concentrator to column
 Transfer line: 0.53mm ID Silcosteel® tubing (cat. #70045)
 Gas chromatograph: Finnigan 9001
 Carrier gas: helium @ ~10mL/min. constant pressure
 Adjust dichlorodifluoromethane to a retention time of 2.47 min. @ 40 C.

Dead time: 2.04 min.
 Oven temp.: 40 C (hold 2 min.) to 58 C @ 4 C/min. to 90 C @ 10 C/min. (hold 5 min.) to 220 C @ 40 C/min. (hold 5 min.)

Detectors: Gold Tandem PID/Hall 2000 ELCD
 PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV base temp: 200 C.
 Hall 2000 ELCD: RxnGas 25mL/min., Rxn Temp. 940 C, propanol flow 470 L/min.

Standard	cat.#	Standard	cat.#
502.2 mix#1	30042	fluorobenzene	30030
624 cal mix #2	30021	1-chloro-2-fluorobenzene	30040
624 cal mix #3	30022	4-bromo-1-chlorobenzene	30230
MTBE	30402	bromochloromethane	30225
1,4-dichlorobutane	30227		

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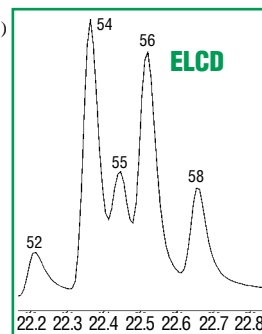
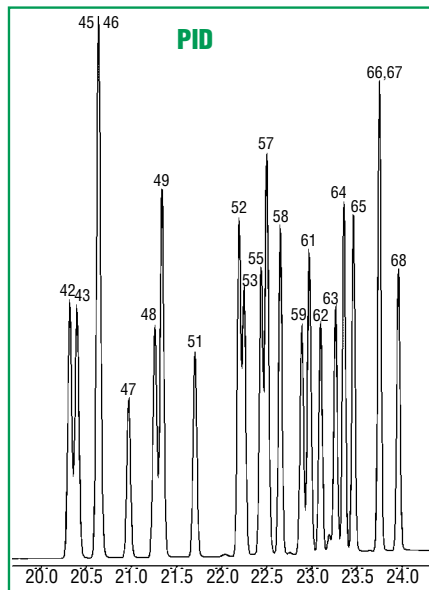
Figure 42A.

An Rtx®-VGC / Rtx®-502.2 column pair separates the expanded list of compounds in EPA Method 8021B in less than 30 minutes.

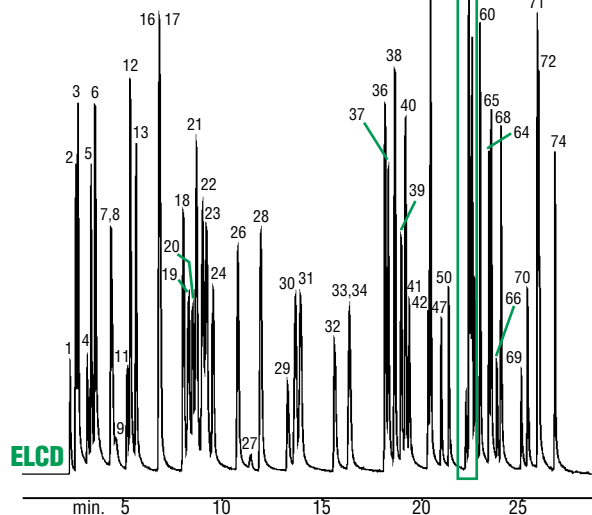
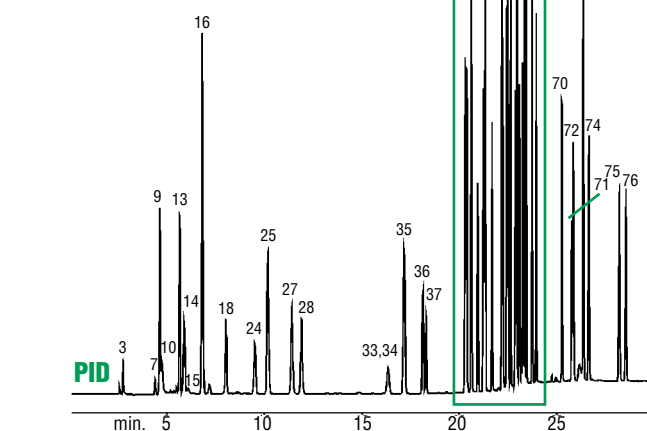
Rtx®-VGC

75m, 0.45mm ID, 2.55µm (cat.# 19409)

Primary column: 75m, 0.45mm ID, 2.55 µm Rtx®-VGC (cat.# 19409)
 Confirmation column: 75m, 0.45mm ID, 2.55 µm Rtx®-502.2 (cat.# 10986)
 Conc.: 10ppb in 5mL of RO Water
 Concentrator: Tekmar LSC-3000 Purge and Trap
 Trap: Vocarb 3000
 Purge: 11 min. @ 40 mL/min.
 Dry purge: 1 min. @ 40mL/min. (MCS bypassed using Silcosteel® tubing)
 Desorb preheat: 245 C, Flow 10mL/min.
 Desorb: 250 C for 2 min.
 Bake: 260 C for 8 min.
 Interface: direct using 0.32mm ID Siltek transfer line (cat. #10027)
 GC: Finnigan 9001
 Carrier gas: helium @ ~10mL/min. constant pressure
 Adjust dichlorodifluoromethane to a retention time of 2.40 min. @ 45 C on the Rtx®-VGC column.
 Oven temp.: 45 C (hold 4 min.) to 70 C @ 2 C/min. to 210 C @ 20 C/min. (hold 10 min.)
 Detectors: Gold Tandem PID/HALL 2000 ELCD
 PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV base temp. 200 C.
 Hall 2000 ELCD: RxnGas 25mL/min., RxnTemp. 940 C, propanol flow 470 L/min.



Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL® 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728



- | | | | | |
|--|------------------------------------|---------------------------------------|--------------------------------|----------------------------------|
| 1. dichlorodifluoromethane | 17. 1,1-dichloroethane | 33. 2-chloroethyl vinyl ether | 49. styrene | 65. 1,4-dichlorobenzene |
| 2. chloromethane | 18. <i>cis</i> -1,2-dichloroethene | 34. <i>cis</i> -1,3-dichloropropene | 50. bromoform | 66. benzyl chloride |
| 3. vinyl chloride | 19. 2,2-dichloropropane | 35. toluene | 51. isopropylbenzene | 67. <i>n</i> -butylbenzene |
| 4. bromomethane | 20. bromochloromethane | 36. tetrachloroethene | 52. bromobenzene | 68. 1,2-dichlorobenzene |
| 5. chloroethane | 21. chloroform | 37. <i>trans</i> -1,3-dichloropropene | 53. <i>n</i> -propylbenzene | 69. 1,2-dibromo-3-chloropropane |
| 6. trichlorofluoromethane | 22. carbon tetrachloride | 38. 1,1,2-trichloroethane | 54. 1,1,2,2-tetrachloroethane | 70. 2-bromo-1-chlorobenzene (SS) |
| 7. 1,1-dichloroethene | 23. 1,1,1-trichloroethane | 39. dibromochloromethane | 55. 2-chlorotoluene | 71. hexachlorobutadiene |
| 8. Freon® 113 | 24. 1,1-dichloropropene | 40. 1,3-dichloropropane | 56. 1,2,3-trichloropropane | 72. 1,2,4-trichlorobenzene |
| 9. chloromethyl methyl ether | 25. benzene | 41. 1,2-dibromoethane | 57. 1,3,5-trimethylbenzene | 73. naphthalene |
| 10. iodomethane (40ppb) | 26. 1,2-dichloroethane | 42. chlorobenzene | 58. 4-chlorotoluene | 74. 1,2,3-trichlorobenzene |
| 11. allyl chloride | 27. fluorobenzene (SS) | 43. ethylbenzene | 59. <i>tert</i> -butylbenzene | 75. 2-methylnaphthalene (40ppb) |
| 12. methylene chloride | 28. tetrachloroethene | 44. 1,1,1,2-tetrachloroethane | 60. pentachloroethane | 76. 1-methylnaphthalene (40ppb) |
| 13. <i>trans</i> -1,2-dichloroethene | 29. dibromomethane | 45. <i>m</i> -xylene | 61. 1,2,4-trimethylbenzene | |
| 14. methyl <i>tert</i> -butyl ether | 30. 1,2-dichloropropane | 46. <i>p</i> -xylene | 62. <i>sec</i> -butylbenzene | |
| 15. <i>tert</i> -butyl alcohol (40ppb) | 31. bromodichloromethane | 47. 1-chloro-2-fluorobenzene (SS) | 63. <i>p</i> -isopropyltoluene | |
| 16. chloroprene | 32. 1-bromo-2-chloroethane (SS) | 48. <i>o</i> -xylene | 64. 1,3-dichlorobenzene | |

GC_EV00421

The chromatograms in Figures 42A & B incorporate a broader range of analytes, many of which are listed in EPA Method 8021B, along with other requested compounds, such as 1-methylnaphthalene and 2-methylnaphthalene. Of these 72 target compounds, those that coelute on the Rtx®-VGC column are resolved by the Rtx®-502.2 column. Even with the addition of the semivolatile methylnaphthalenes, the analysis time is less than 30 minutes.

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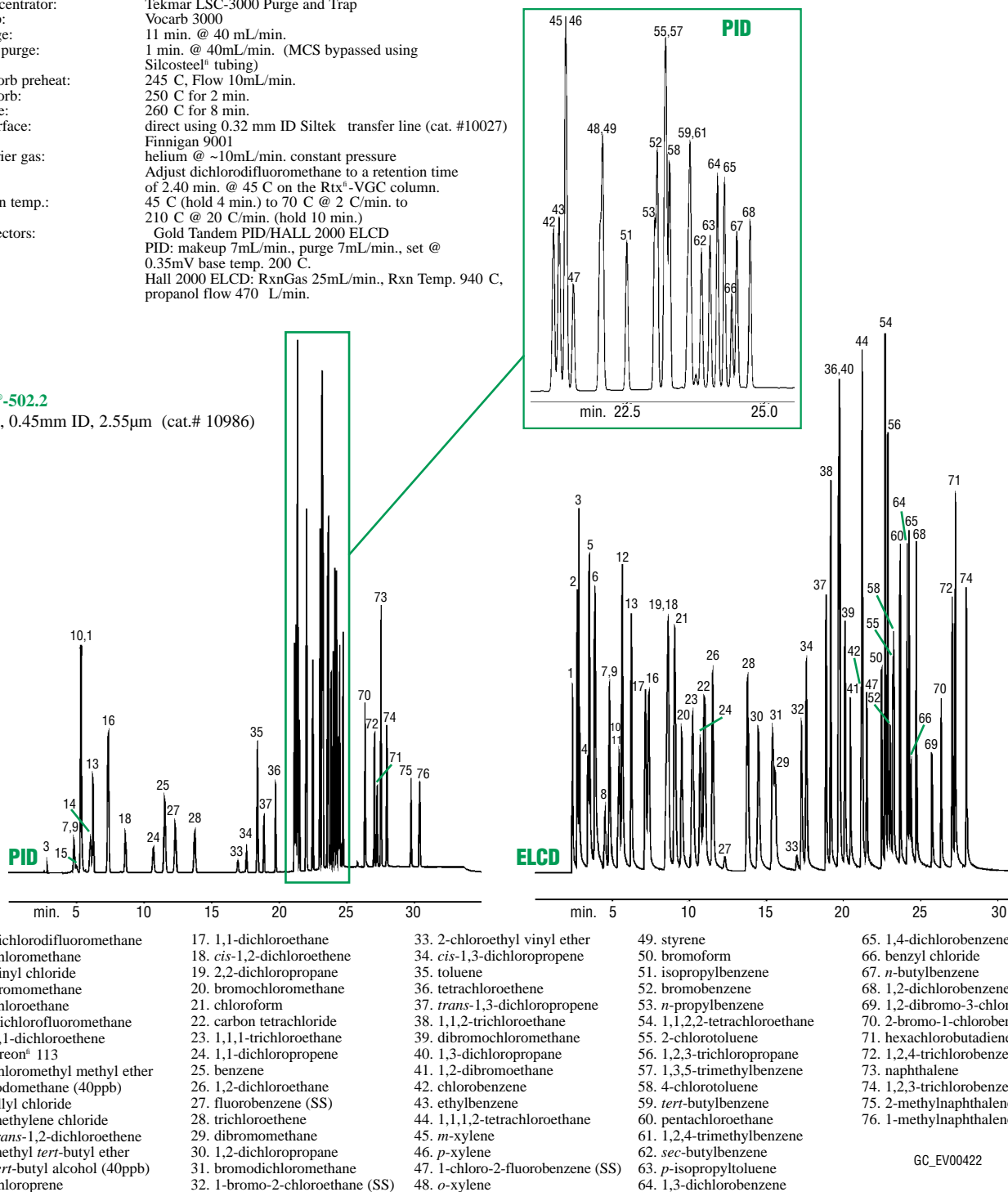
Figure 42B.

An Rtx®-VGC / Rtx®-502.2 column pair separates the expanded list of compounds in EPA Method 8021B in less than 30 minutes.

Primary column: 75m, 0.45mm ID, 2.55 m Rtx®-VGC (cat.# 19409)
 Confirmation column: 75m, 0.45mm ID, 2.55 m Rtx®-502.2 (cat.# 10986)
 Conc.: 10 ppb in 5 mL of RO Water
 Concentrator: Tekmar LSC-3000 Purge and Trap
 Trap: Vocab 3000
 Purge: 11 min. @ 40 mL/min.
 Dry purge: 1 min. @ 40 mL/min. (MCS bypassed using Silcosteel® tubing)
 Desorb preheat: 245 C, Flow 10 mL/min.
 Desorb: 250 C for 2 min.
 Bake: 260 C for 8 min.
 Interface: direct using 0.32 mm ID Siltek transfer line (cat. #10027)
 GC: Finnigan 9001
 Carrier gas: helium @ ~10 mL/min. constant pressure
 Adjust dichlorodifluoromethane to a retention time of 2.40 min. @ 45 C on the Rtx®-VGC column.
 Oven temp.: 45 C (hold 4 min.) to 70 C @ 2 C/min. to 210 C @ 20 C/min. (hold 10 min.)
 Detectors: Gold Tandem PID/HALL 2000 ELCD
 PID: makeup 7 mL/min., purge 7 mL/min., set @ 0.35 mV base temp. 200 C.
 Hall 2000 ELCD: Rxn Gas 25 mL/min., Rxn Temp. 940 C, propanol flow 470 L/min.

Rtx®-502.2

75m, 0.45mm ID, 2.55µm (cat.# 10986)



Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL® 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

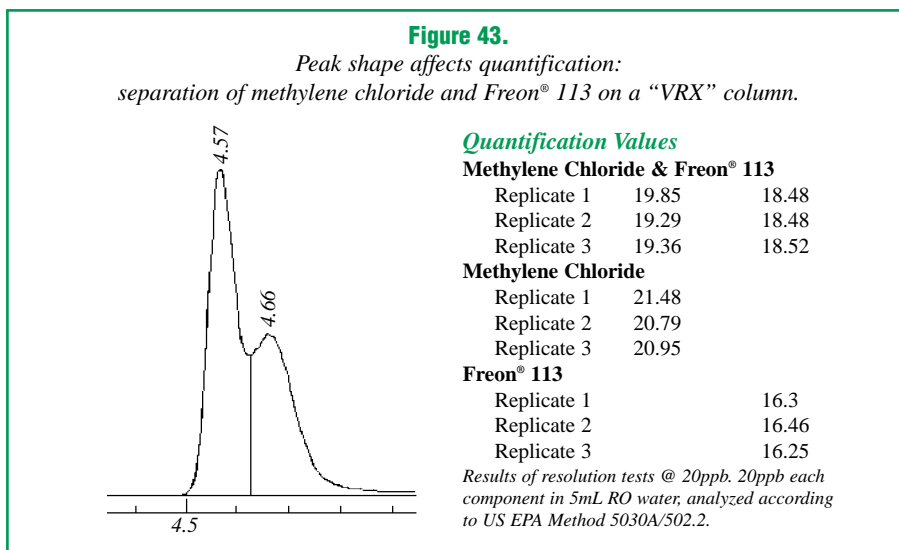
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Importance of Resolution in GC Analysis

Figure 43 shows the effects of resolution on quantification for methylene chloride and Freon® 113 (1,1,2-trichloro-1,2,2-trifluoroethane) when separated on an Rtx®-VRX column. The first three replicates are check standards analyzed on a six-point curve. These values are taken from integrating the areas for the two closely eluting peaks. Based on peak height (the methylene chloride peak is about twice the height of the Freon® 113 peak), the contribution of the first peak, methylene chloride, to the Freon® 113 peak is more than twice the contribution of Freon® 113 to the methylene chloride peak. Both compounds are quantified from the ELCD, creating another quantification issue with tailing peaks: the tail of the methylene chloride peak contributes to the area of the Freon® 113 peak, thereby increasing the quantification error.

Next, we analyzed methylene chloride and Freon® 113 separately, to determine if there would be quantification differences off the six-point curve. With the addition of the peak tail, the peak area for methylene chloride increased slightly, and the peak area for Freon® 113 decreased. Without the contribution from methylene chloride, Freon® 113 exhibited a noticeably lower quantification value of 16ppb.

This experiment illustrates the importance of resolution for accurate quantification of analytes in environmental samples taken from the field. Obviously, no column can provide baseline separation of all Method 502.2/8021 compounds. The key to accurate quantification is to recognize which compounds are most commonly present in your samples and choose a column that best resolves these analytes. As shown in the example described above, both the Rtx®-VRX column and the Rtx®-VGC column have difficulty resolving methylene chloride from Freon® 113.

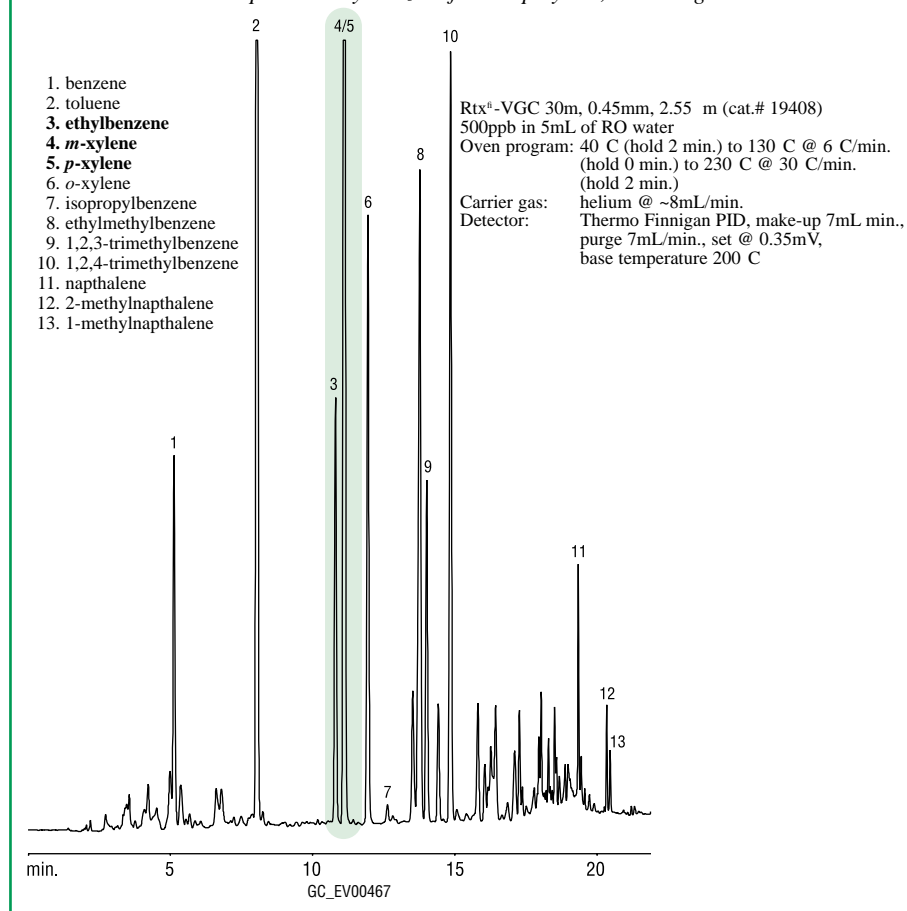


**Purge and Trap Applications Using PID/FID in Tandem:
EPA Method 602 and Others; State Gasoline Methods**

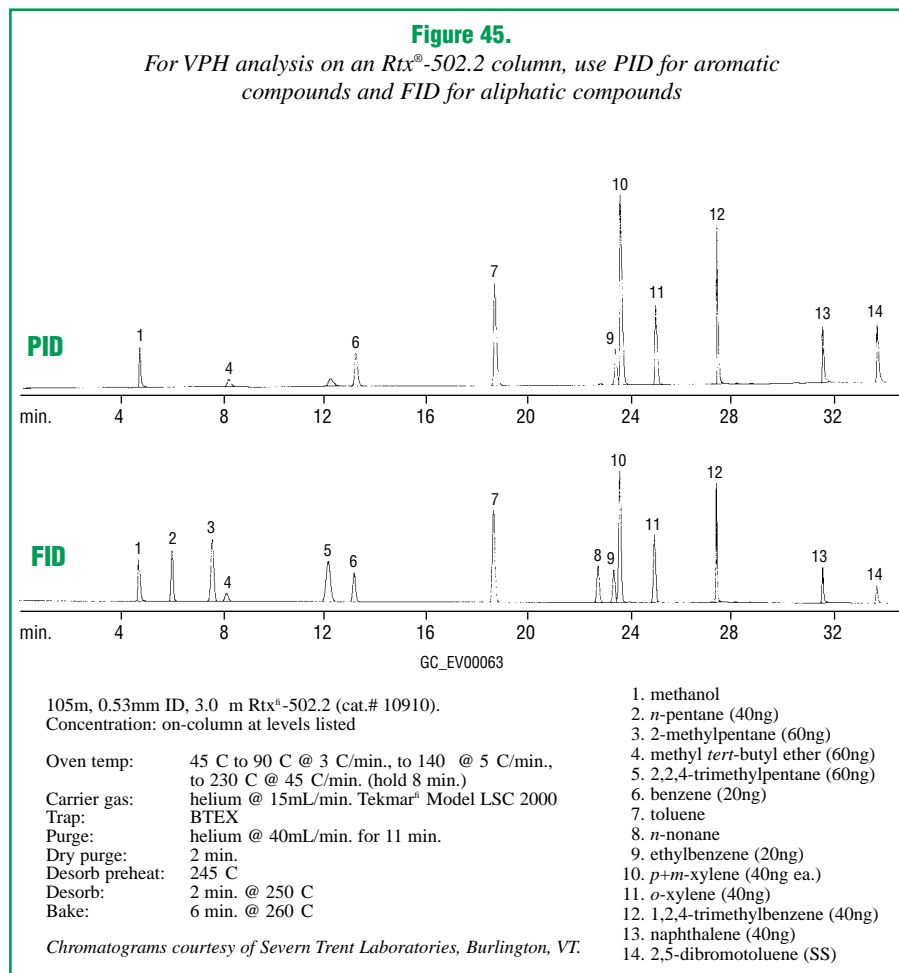
EPA Methods 602, 8015, 8020, and 8260 describe analyses of gasoline and oxygenates. Most environmental laboratories have relied on one of these methods to report gasoline and oxygenate concentrations in difficult sample matrixes. Success with these methods is based on the ability of the capillary column to resolve close-eluting pairs. An analysis according to Method 8015, for example, involves separating ethylbenzene from m/p-xylene (Figure 44).

Figure 44.

An Rtx®-VGC column separates ethylbenzene from m/p-xylene, according to EPA method 8015.



Many state methods, such as the Massachusetts Volatile Petroleum Hydrocarbon (VPH) method, require resolution of the oxygenates and the early eluting alkanes, such as 2-methylpentane and 3-methylpentane (Figure 45). The most common column used for GRO analysis is a 30m, 0.53mm ID column with a 1.5µm df film of 5% diphenylpolysiloxane phase, such as Rtx®-5. These columns resolve the difficult compounds chlorobenzene and ethylbenzene from the xylenes. An Rtx®-502.2 column performs this separation equally well, but a longer column is needed.

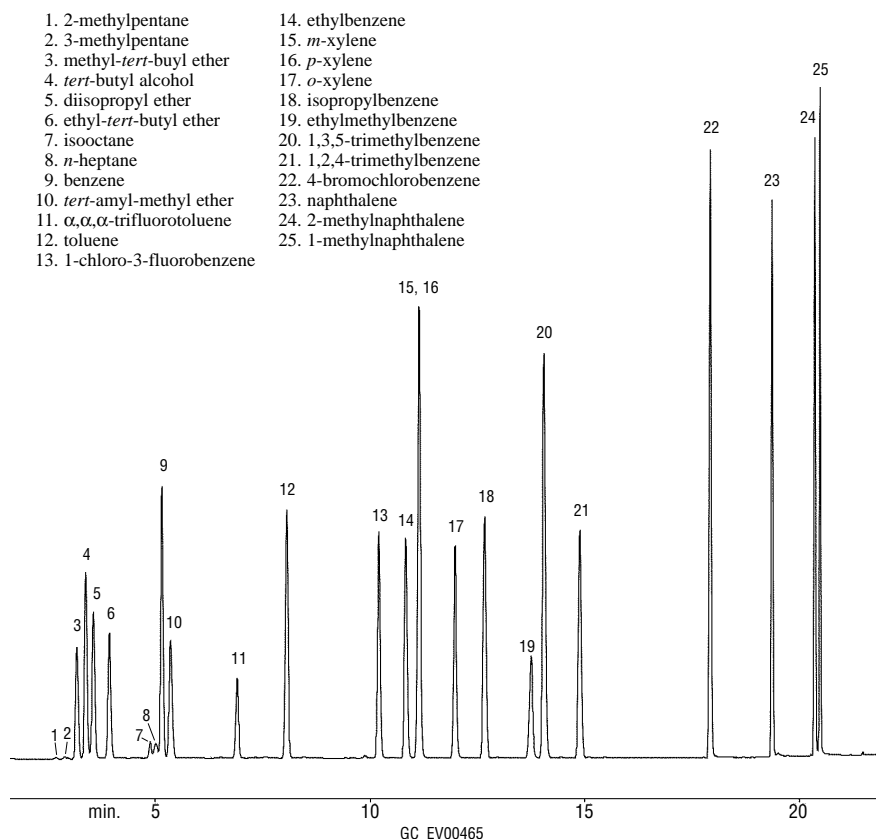


Gasoline Analysis with Oxygenates and Alcohols

Success of gasoline range organics (GRO) analyses are based on the ability of the analytical column to resolve oxygenates from the alkanes, alkenes, and, to a lesser extent, alkynes. To minimize false positive results for methyl-*tert*-butyl ether (MTBE) it is important to separate this analyte from 2-methylpentane and 3-methylpentane. Another potential interference is *tert*-butyl alcohol (TBA). Both MTBE and TBA elicit response on a PID, and they share ions used in MS detection, so they must be resolved regardless of which detector is used. Figure 46 is an example of GRO target compounds analyzed using PID detection, with the oxygen-containing gasoline additives resolved from the other analytes.

Figure 46.

An Rtx®-VGC column resolves oxygen-containing gasoline derivatives from other GRO.



30m, 0.45mm, 2.55 m Rtx®-VGC (cat.# 19408)

each component 100ppb in 5mL of RO water, except *tert*-butyl alcohol 5000ppb; 2/1-methylnaphthalene 150ppb; ethylmethylbenzene 50ppb.

Concentrator: Tekmar LSC-3100 Purge and Trap

Trap: Vocab 3000

Purge: 11 min. @ 40mL/min. @ 35 C

Dry purge: 1 min. (MCS bypassed)

GC: Finnigan 9001

Oven temp.: 40 C (hold 2 min.) to 130 C @ 6 C/min. (hold 0 min.) to 230 C @ 30 C/min. (hold 2 min.).

Carrier gas: helium @ ~8mL/min.

Detector: Thermo Finnigan PID, make-up 7mL min., purge 7mL/min., set @ 0.35mV, base temperature 200 C.

Advances in Sample Throughput

The demand for increased productivity in volatiles analysis by GC/MS has resulted in the creation of automated water and soil autosamplers that reduce the amount of manual sample preparation required. Autosamplers enable environmental laboratories to run purge and trap systems around the clock. Even though prices for analyses of samples by Methods 8260 and 524.2 have stabilized, laboratories still push for faster turn-around-time, to get a better return on capital equipment investments. This has resulted in a need for columns that can drastically reduce separation time and for instruments that can accommodate short cycle times. Currently, the limiting factor in VOA is the purge and trap cycle time, because it includes an 11-minute purge time followed by a 6-12 minute bake-out time. A modern GC, on the other hand, can acquire a sample in 10 minutes or less. To overcome the time limitations of the purge and trap, connect two purge and traps, each with its own autosampler unit, to one GC/MS operating system. Use the dual-concentrator configuration to synchronize the steps so while the first system is desorbing the sample and starting the GC/MS analysis, the second system is completing the bake cycle and starting to purge the next sample to be desorbed onto the column. The Duet® system, designed and sold by Tekmar-Dohrmann, allows communication between the two concentrators for configuration to one GC/MS. The Duet® interface gates the signals between the concentrators to prevent a faster system from catching up to a slightly slower one and allowing a double injection. Calibration curves and quality control samples (QC, MS, MSD) must be run for each concentrator.

A tracer compound must be added to one of the concentrators, to eliminate any potential question as to which purge and trap system purged/desorbed the sample. With this system it is possible to run 80 samples in 24 hours, thereby increasing output from a single GC/MS instrument. Figure 47 (page 51) shows an analysis on an Rtx®-VMS column according to US EPA Method 8260B, using the correct internal standards and surrogates. For more information see the literature cited.¹⁴

Applications Using GC/MS Detection Systems

EPA Methods 8260, 524.2, 624, 8240 and OLM 04.2

Method 8260: Client target lists may remain the same as the Method 8240 compound list, but the calibration criteria and low detection limits set by Method 8260 are enforced (Figure 48, page 52).¹⁵ Chromatograms for the 8240 compound list can be produced from different GC oven conditions, different compound concentrations, and altered MS scan windows. Alcohols analyses require scanning below 35amu because many of the fragments used to identify the spectra for these compounds are between 25 and 35amu. A good example is 2-chloroethanol – this target analyte purges poorly and does not respond well by MS detection. The best way to increase sensitivity by MS detection is by changing the scan rate to include ion 31, the base peak. This also improves the ability of the software and the analyst to identify alcohols because it gives more spectral data. The disadvantage of this approach is an increase in noise, producing an overall decrease in sensitivity for all compounds. In Figure 48, the second chromatogram shows an increase in baseline noise as a result of the lower scan window. A comparison of peak 38 (2-chloroethanol) on the two chromatograms clearly shows a significantly higher response on the second chromatogram, despite a lower concentration.

Method 8260 contains many mid-range volatile compounds that are the most common non-petroleum contaminants in the environment. Unfortunately, these compounds tend to exhibit broad peak shapes due to poor sample transfer from the purge and trap, making them difficult to resolve. Rtx®-VMS columns were designed using computer-assisted stationary phase design (CASPD) software to improve solubility of these analytes in the stationary phase, and thus provide greater separation for these compounds.¹⁶ This tuned selectivity ensures separation of tetrahydrofuran/2-butanone, carbon tetrachloride/1,1,1-trichloroethane, and methyl acrylate/propionitrile. Although these compounds share common ions and have very similar spectra, they are resolved by retention time difference on an Rtx®-VMS column (Figure 49, page 53). Analytes that share ions and coelute on an Rtx®-624 column, but are resolved by an Rtx®-VMS column, include: ether/ethanol, vinyl acetate/ethyl-tert-butyl ether, and tert-butyl alcohol/methyl-tert-butyl ether. Several of these compounds require a lower initial oven temperature (35°C), which is not shown in these applications.

Higher-boiling volatile compounds, typically branched or substituted aromatic compounds, provide analytical challenges of their own. Isomers of the branched aromatic compounds share the same parent ions and cannot be identified accurately by MS alone. The Rtx®-VMS phase also was modeled for maximum separation of the substituted aromatic isomers, such as 2- and 4-chlorotoluene. The comparison in Table VII shows isomer resolution on four other stationary phases, modeled under the same conditions, compared to resolution on the Rtx®-VMS phase. The tuned selectivity of the Rtx®-VMS phase allows a rapid final GC oven ramp rate of 40°C/min., or faster, thereby promoting fast analysis times. Also, initial temperatures of up to 60°C are possible (Figure 49, page 53). This higher initial temperature provides the required separation and allows faster oven cycle times, although some laboratories prefer to start at 50°C, to better enhance the resolution of chloromethane from vinyl chloride (peaks 2 and 3).

Figure 47 (page 51) shows an analysis of the Method 8260B compound list, using an Rtx®-VMS column (20m, 0.18mm ID, 1.0µm film) without cryogenic cooling. Resolution is greatly enhanced, due to the higher efficiency of the 0.18mm ID column. The desorb flow rate is set at 40mL/min. for 1 minute. Many laboratories desorb under these conditions for 2 minutes, but the Rtx®-VMS column makes this unnecessary, because the higher flow rate will desorb the volatiles from the trap in less than a minute.

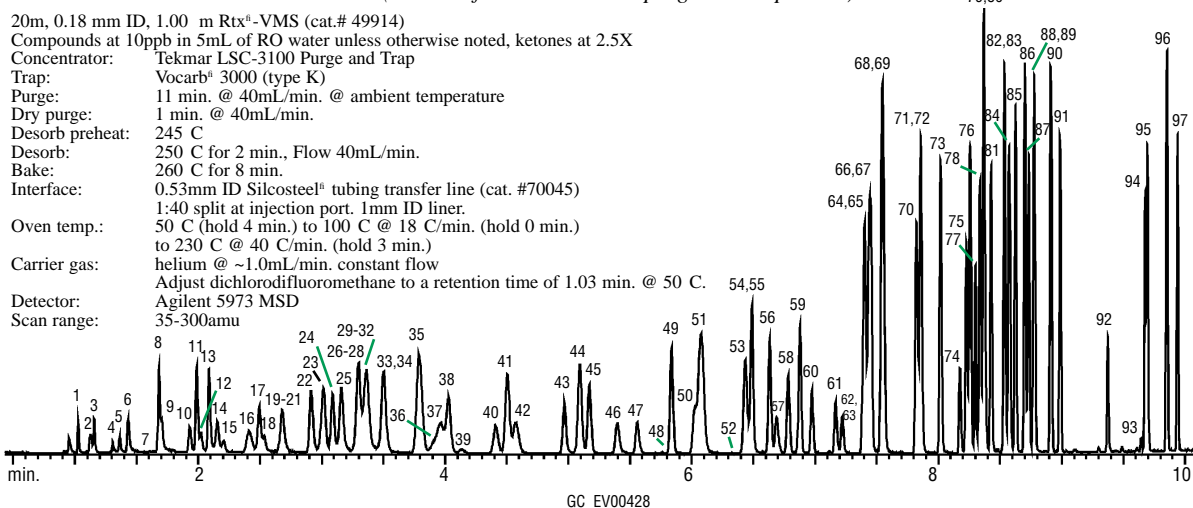
Table VII.

An Rtx®-VMS column best separates 2- and 4-chlorotoluene.

Retention Time (min.)	Rtx®-VMS	Rtx®-624	Rtx®-502.2	Rtx®-VRX	Rtx®-1
2-Chlorotoluene	8.35	8.63	8.80	8.49	8.38
4-Chlorotoluene	8.44	8.69	8.84	8.53	8.41
RT diff.	0.09	0.06	0.04	0.04	0.03

Figure 47.

Volatile organics by US EPA Method 8260B on an Rtx®-VMS column; conditions optimized for fast Method 8260 analysis.
(Suitable for use with dual purge and trap units.)



- | | | | |
|---|-------------------------------------|---------------------------------------|---------------------------------|
| 1. dichlorodifluoromethane | 26. ethyl acetate | 51. toluene | 76. <i>n</i> -propylbenzene |
| 2. chloromethane | 27. carbon tetrachloride | 52. pyridine (250ppb) | 77. 1,1,2,2-tetrachloroethane |
| 3. vinyl chloride | 28. methyl acrylate | 53. tetrachloroethene | 78. 2-chlorotoluene |
| 4. bromomethane | 29. propargyl alcohol (500ppb) | 54. 4-methyl-2-pentanone | 79. 1,3,5-trimethylbenzene |
| 5. chloroethane | 30. dibromofluoromethane (SMC) | 55. <i>trans</i> -1,3-dichloropropene | 80. 1,2,3-trichloropropane |
| 6. trichlorofluoromethane | 31. tetrahydrofuran | 56. 1,1,2-trichloroethane | 81. 4-chlorotoluene |
| 7. ethanol (2500ppb) | 32. 1,1,1-trichloroethane | 57. ethyl methacrylate | 82. <i>tert</i> -butylbenzene |
| 8. 1,1-dichloroethene | 33. 2-butanone | 58. dibromochloromethane | 83. pentachloroethane |
| 9. carbon disulfide (40ppb) | 34. 1,1-dichloropropene | 59. 1,3-dichloropropane | 84. 1,2,4-trimethylbenzene |
| 10. allyl chloride | 35. benzene | 60. 1,2-dibromoethane | 85. <i>sec</i> -butylbenzene |
| 11. methylene chloride | 36. pentafluorobenzene (IS) | 61. <i>n</i> -butyl acetate | 86. <i>p</i> -isopropyltoluene |
| 12. acetone | 37. <i>tert</i> -amyl-methyl ether | 62. 2-hexanone | 87. 1,3-dichlorobenzene |
| 13. <i>trans</i> -1,2-dichloroethene | 38. 1,2-dichloroethane | 63. 2-picoline (250ppb) | 88. 1,4-dichlorobenzene-d4 (IS) |
| 14. methyl <i>tert</i> -butyl ether | 39. isobutyl alcohol (500ppb) | 64. chlorobenzene-d5 (IS) | 89. 1,4-dichlorobenzene |
| 15. <i>tert</i> -butyl alcohol (100ppb) | 40. isopropyl acetate | 65. chlorobenzene | 90. <i>n</i> -butylbenzene |
| 16. diisopropyl ether | 41. trichloroethene | 66. ethylbenzene | 91. 1,2-dichlorobenzene |
| 17. 1,1-dichloroethane | 42. 1,4-difluorobenzene (SMC) | 67. 1,1,1,2-tetrachloroethane | 92. 1,2-dibromo-3-chloropropane |
| 18. acrylonitrile | 43. dibromomethane | 68. <i>m</i> -xylene | 93. nitrobenzene (250ppb) |
| 19. vinyl acetate | 44. 1,2-dichloropropane | 69. <i>p</i> -xylene | 94. hexachlorobutadiene |
| 20. allyl alcohol (250ppb) | 45. bromodichloromethane | 70. <i>o</i> -xylene | 95. 1,2,4-trichlorobenzene |
| 21. ethyl <i>tert</i> -butyl ether | 46. methyl methacrylate | 71. styrene | 96. naphthalene |
| 22. <i>cis</i> -1,2-dichloroethene | 47. <i>n</i> -propyl acetate | 72. bromoform | 97. 1,2,3-trichlorobenzene |
| 23. 2,2-dichloropropane | 48. 2-chloroethanol (2500ppb) | 73. isopropylbenzene | |
| 24. bromochloromethane | 49. <i>cis</i> -1,3-dichloropropene | 74. 4-bromo-1-fluorobenzene (SMC) | |
| 25. chloroform | 50. toluene-d8(SMC) | 75. bromobenzene | |

EPA-recommended SMC/IS used.

One of the most important factors in optimizing conditions for using the narrow-bore column is adjusting the flow. Most MS systems are designed for optimum sensitivity at 1mL/min.; flow rates higher or lower will greatly compromise the method detection limit (MDL). For Figure 47 (page 51) the retention time for the first gas, dichlorodifluoromethane, is 1.03 minutes at 50°C, which dictates a column flow of 1mL/min. Figure 49 (page 53) also lists specific information for setting the correct flow rate.

Electronic pressure control (EPC) makes it possible to maintain a constant flow over the course of the oven temperature program, which can cut several minutes from the analysis time, compared to a system set up for constant pressure. When setting up a system for constant pressure, always adjust the flow at the initial oven temperature to be approximately 1mL/min. It is true that, under constant pressure, higher flows at the beginning of the analysis will equate to normal flows (closer to 1mL/min.) as the temperature, and carrier gas viscosity, increases, but maximum sensitivity is needed for the more volatile analytes because they exhibit broader peaks. Also, higher flows at the start of the analysis, while methanol and water are entering the MS, could cause excessive source pressure and automatically shut the filament off. Figure 49 (page 53) shows an analysis on a 60m, 0.25mm ID, 1.4µm film Rtx®-VMS column, using an initial temperature of 60°C. The injection port is set for a 1:20 split and constant flow is adjusted to 1.3mL/min. Again, the best way to set the flow for these columns is to use the retention time for dichlorodifluoromethane or for an unretained compound. Carbon dioxide is a good choice for an unretained compound.

14. A.L. Hilling and G. Smith, *Environmental Testing & Analysis*, 10(3),15-19, 2001.

15. *Method 8260B Volatile Organic Compounds in Water by Gas Chromatography/Mass Spectrometry*, Revision 2.0, 1996, SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

16. F.L. Dorman, P.D. Schettler, C.M. English and D.V. Patwardhan. "Predicting Gas Chromatographic Separation and Stationary-Phase Selectivity Using Computer Modeling." *Anal. Chem.* 2002, 74, 2133-2138.

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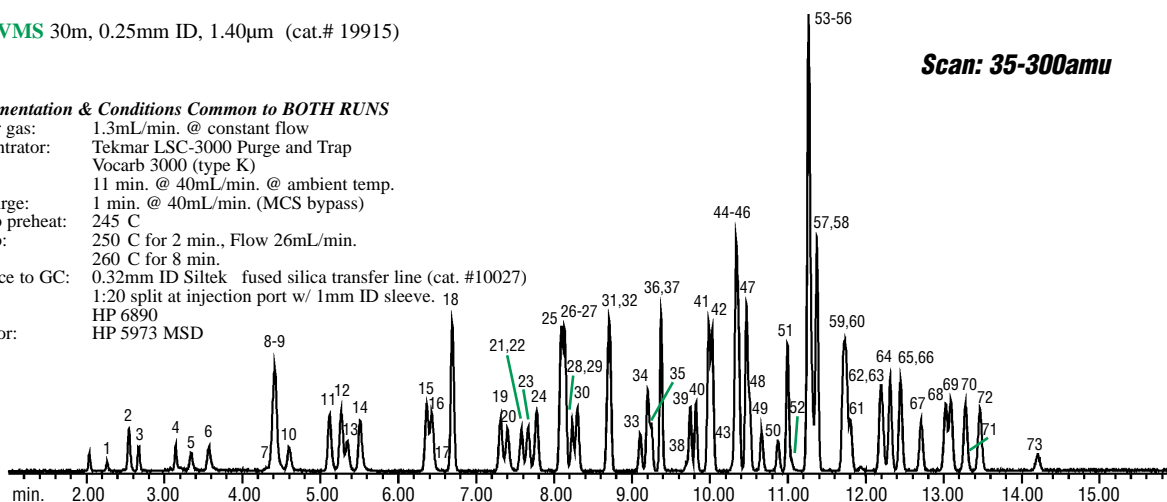
Figure 48.

EPA Method 8240 analysis using an Rtx®-VMS column. Alcohols require scans below 35amu.

Rtx®-VMS 30m, 0.25mm ID, 1.40µm (cat.# 19915)

Scan: 35-300amu**Instrumentation & Conditions Common to BOTH RUNS**

Carrier gas: 1.3mL/min. @ constant flow
 Concentrator: Tekmar LSC-3000 Purge and Trap
 Trap: Vocab 3000 (type K)
 Purge: 11 min. @ 40mL/min. @ ambient temp.
 Dry purge: 1 min. @ 40mL/min. (MCS bypass)
 Desorb preheat: 245 C
 Desorb: 250 C for 2 min., Flow 26mL/min.
 Bake: 260 C for 8 min.
 Interface to GC: 0.32mm ID Siltek fused silica transfer line (cat. #10027)
 1:20 split at injection port w/ 1mm ID sleeve. 18
 GC: HP 6890
 Detector: HP 5973 MSD

**Top chromatogram:**

Oven temp.: 40 C (hold 4 min.) to 90 C @ 16 C/min. (no hold)
 to 210 C @ 32 C/min. (hold 5 min.)
 Adjust dichlorodifluoromethane to a retention time of
 2.27 min. @ 40 C.

MS Scan Range: 35-300amu

Compound Concentrations, by mix: (in 5mL of RO water)

Compounds at 100ppb (cat.# 30213, 30004, 30006, 30011, 30042)

Alcohols at 1ppm (cat.# 30214); 2-chloroethanol at 10ppm.

vinyl acetate at 500ppb (cat.#30216)

8240 Nitrile Mix at 200ppb (cat.# 30215)

8240 Mix 1A at 300ppb (cat.# 30217)

8240 Mix 2A at 500ppb (cat.# 30218)

Bottom chromatogram:

Oven temp.: 45 C (hold 4 min.) to 110 C @ 19 C/min. (hold 5 min.) to
 220 C @ 32 C/min. (hold 5 min.)
 Adjust dichlorodifluoromethane to 2.23 min. @ 45 C.

MS Scan Range: 29-260amu, for 2-chloroethanol response

Compound Concentrations, by mix: (in 5mL of RO water)

Compounds at 100ppb (cat.# 30213, 30004, 30006, 30011, 30042)

Alcohols at 1ppm (cat.# 30214) (see MS scan)

vinyl acetate at 100ppb (cat.# 30216)

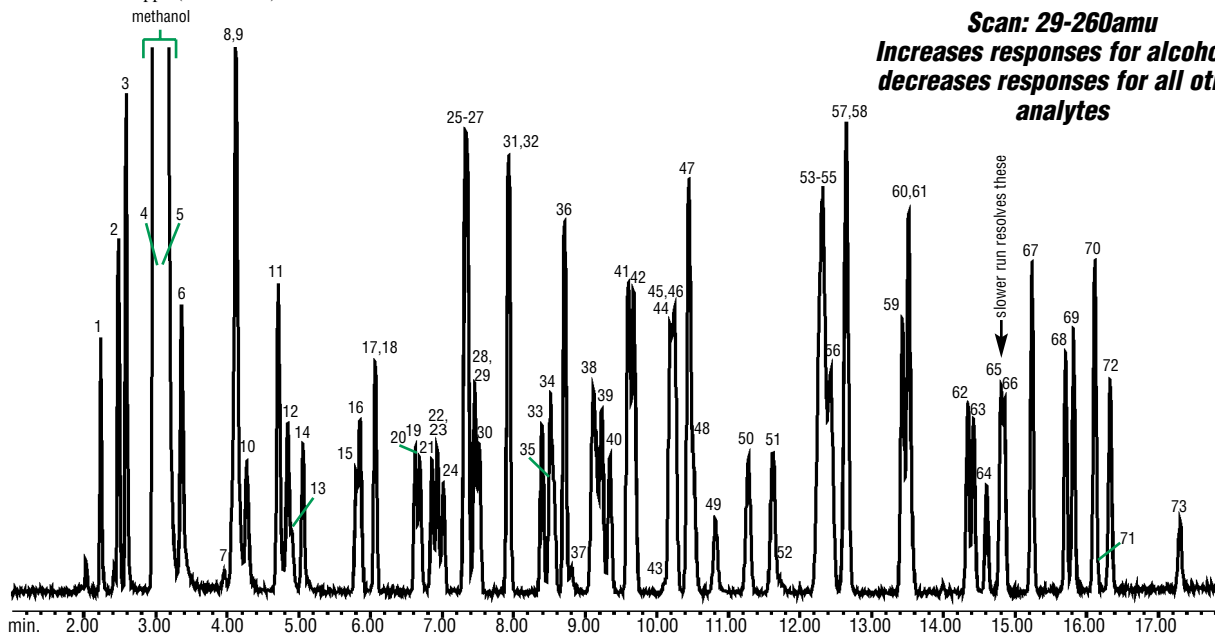
8240 Nitrile Mix at 400ppb (cat.# 30215)

8240 Mix 1A at 300ppb (cat.# 30217)

8240 Mix 2A at 500ppb (cat.# 30218)

Scan: 29-260amu

**Increases responses for alcohols,
 decreases responses for all other
 analytes**



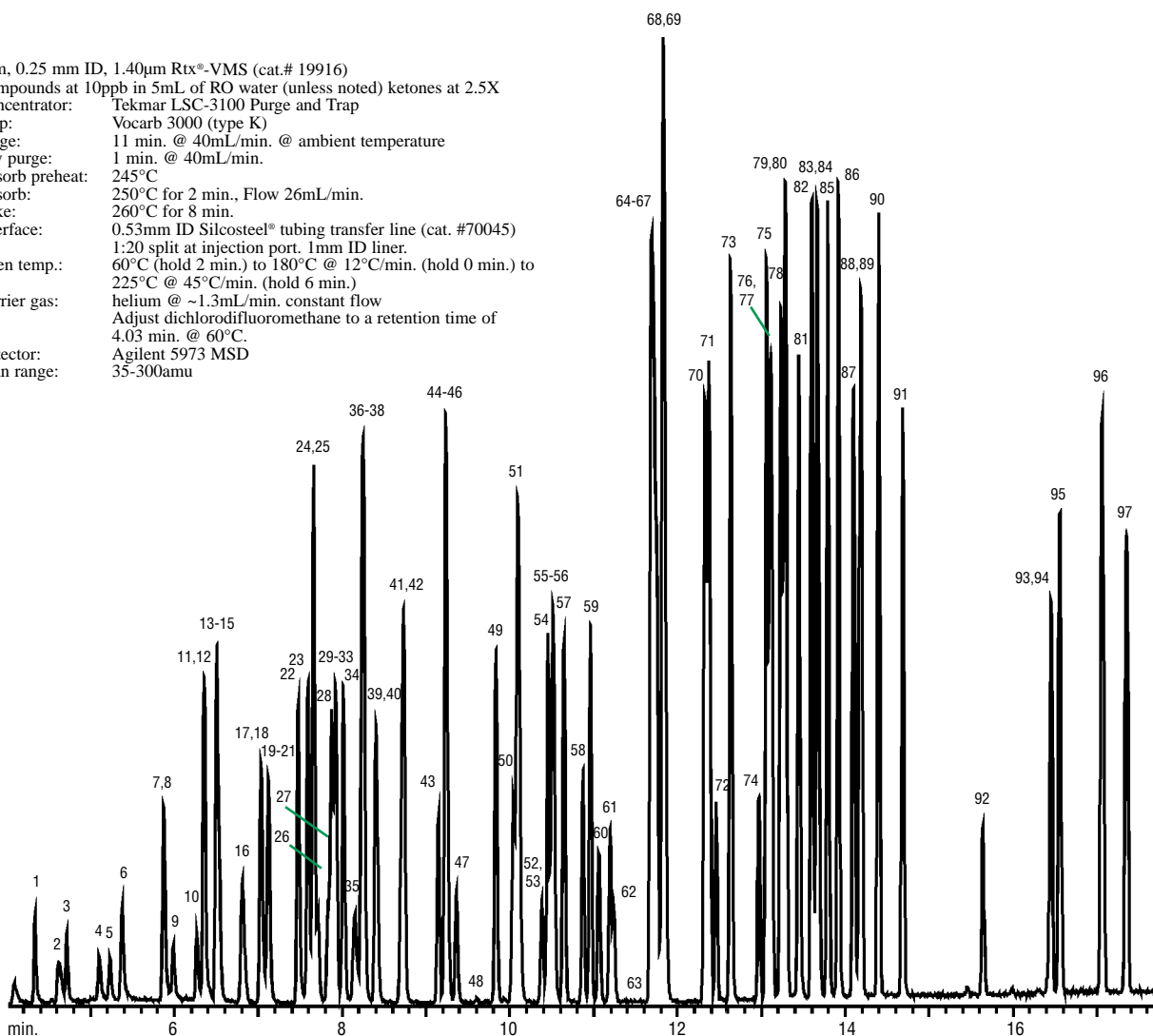
- | | | | | | |
|----------------------------|--------------------------------------|-------------------------------|---------------------------------------|---------------------------------------|---|
| 1. dichlorodifluoromethane | 14. <i>trans</i> -1,2-dichloroethene | 27. methacrylonitrile | 40. <i>cis</i> -1,3-dichloropropene | 53. chlorobenzene-D5 | 66. <i>trans</i> -1,4-dichloro-2-butene |
| 2. chloromethane | 15. 1,1-dichloroethane | 28. 1,2-dichloroethane-d4 | 41. toluene-d8 | 54. ethylbenzene | 67. pentachloroethane |
| 3. vinyl chloride | 16. acrylonitrile | 29. isobutyl alcohol | 42. toluene | 55. chlorobenzene | 68. 1,3-dichlorobenzene |
| 4. bromomethane | 17. allyl alcohol | 30. 1,2-dichloroethane | 43. pyridine | 56. 1,1,1,2-tetrachloroethane | 69. 1,4-dichlorobenzene |
| 5. chloroethane | 18. vinyl acetate | 31. trichloroethene | 44. 4-methyl-2-pentanone | 57. <i>m</i> -xylene | 70. benzyl chloride |
| 6. trichlorofluoromethane | 19. bromochloromethane | 32. 1,4-difluorobenzene | 45. tetrachloroethene | 58. <i>p</i> -xylene | 71. malononitrile |
| 7. ethanol | 20. chloroform | 33. dibromomethane | 46. <i>trans</i> -1,3-dichloropropene | 59. <i>o</i> -xylene | 72. 1,2-dichlorobenzene |
| 8. 1,1-dichloroethene | 21. carbon tetrachloride | 34. 1,2-dichloropropane | 47. ethyl methacrylate | 60. styrene | 73. 1,2-dibromo-3-chloropropane |
| 9. carbon disulfide | 22. propargyl alcohol | 35. bromodichloromethane | 48. 1,1,2-trichloroethane | 61. bromoform | |
| 10. iodomethane | 23. 1,1,1-trichloroethane | 36. methyl methacrylate | 49. dibromochloromethane | 62. 4-bromo-1-fluorobenzene | |
| 11. allyl chloride | 24. 2-butanone | 37. 1,4-dioxane | 50. 1,2-dibromoethane | 63. <i>cis</i> -1,4-dichloro-2-butene | |
| 12. methylene chloride | 25. benzene | 38. 2-chloroethanol | 51. 2-hexanone | 64. 1,1,2,2-tetrachloroethane | |
| 13. acetone | 26. propionitrile | 39. 2-chloroethyl vinyl ether | 52. 2-picoline | 65. 1,2,3-trichloropropane | |

GC_EV00426

Figure 49.

An Rtx®-VMS column allows an initial temperature of 60°C, for fast oven cycle times by EPA Method 8260.

60m, 0.25 mm ID, 1.40µm Rtx®-VMS (cat.# 19916)
 Compounds at 10ppb in 5mL of RO water (unless noted) ketones at 2.5X
 Concentrator: Tekmar LSC-3100 Purge and Trap
 Trap: Vocab 3000 (type K)
 Purge: 11 min. @ 40mL/min. @ ambient temperature
 Dry purge: 1 min. @ 40mL/min.
 Desorb preheat: 245°C
 Desorb: 250°C for 2 min., Flow 26mL/min.
 Bake: 260°C for 8 min.
 Interface: 0.53mm ID Silcosteel® tubing transfer line (cat. #70045)
 1:20 split at injection port. 1mm ID liner.
 Oven temp.: 60°C (hold 2 min.) to 180°C @ 12°C/min. (hold 0 min.) to
 225°C @ 45°C/min. (hold 6 min.)
 Carrier gas: helium @ ~1.3mL/min. constant flow
 Adjust dichlorodifluoromethane to a retention time of
 4.03 min. @ 60°C.
 Detector: Agilent 5973 MSD
 Scan range: 35-300amu



1. dichlorodifluoromethane	21. ethyl- <i>tert</i> -butyl ether*	41. 1,4-difluorobenzene (SMC)	61. 1,2-dibromoethane	81. 4-chlorotoluene
2. chloromethane	22. <i>cis</i> -1,2-dichloroethene	42. trichloroethene	62. 2-hexanone	82. <i>tert</i> -butylbenzene
3. vinyl chloride	23. 2,2-dichloropropane	43. dibromomethane	63. 2-picoline (250ppb)	83. 1,2,4-trimethylbenzene
4. bromomethane	24. bromochloromethane	44. bromodichloromethane	64. ethylbenzene	84. pentachloroethane
5. chloroethane	25. chloroform	45. 1,2-dichloropropane	65. chlorobenzene-D5 (IS)	85. <i>sec</i> -butylbenzene
6. trichlorofluoromethane	26. ethyl acetate	46. methyl methacrylate	66. chlorobenzene	86. <i>p</i> -isopropyltoluene
7. ethanol (2500ppb)	27. methyl acrylate	47. <i>n</i> -propyl acetate	67. 1,1,1,2-tetrachloroethane	87. 1,3-dichlorobenzene
8. 1,1-dichloroethene	28. propargyl alcohol (500ppb)	48. 2-chloroethanol (2500ppb)	68. <i>m</i> -xylene	88. 1,4-dichlorobenzene-d4 (IS)
9. carbon disulfide (40ppb)	29. dibromofluoromethane (SMC)	49. <i>cis</i> -1,3-dichloropropene	69. <i>p</i> -xylene	89. 1,4-dichlorobenzene
10. allyl chloride	30. tetrahydrofuran	50. toluene-d8 (SMC)	70. <i>o</i> -xylene	90. <i>n</i> -butylbenzene
11. methylene chloride	31. carbon tetrachloride	51. toluene	71. styrene	91. 1,2-dichlorobenzene
12. acetone	32. 2-butanone	52. 4-methyl-2-pentanone	72. bromoform	92. 1,2-dibromo-3-chloropropane
13. <i>trans</i> -1,2-dichloroethene	33. 1,1,1-trichloroethane	53. pyridine (250ppb)	73. isopropylbenzene	93. nitrobenzene (250ppb)
14. <i>tert</i> -butyl alcohol (100ppb)	34. 1,1-dichloropropene	54. <i>trans</i> -1,3-dichloropropene	74. 4-bromo-1-fluorobenzene (SMC)	94. hexachlorobutadiene
15. methyl <i>tert</i> -butyl ether	35. pentafluorobenzene (IS)	55. ethyl methacrylate	75. <i>n</i> -propylbenzene	95. 1,2,4-trichlorobenzene
16. diisopropyl ether	36. <i>tert</i> -amyl methyl ether	56. tetrachloroethene	76. 1,1,2,2-tetrachloroethane	96. naphthalene
17. 1,1-dichloroethane	37. benzene	57. 1,1,2-trichloroethane	77. bromobenzene	97. 1,2,3-trichlorobenzene
18. acrylonitrile	38. isobutyl alcohol (500ppb)	58. dibromochloromethane	78. 1,3,5-trimethylbenzene	
19. vinyl acetate*	39. 1,2-dichloroethane	59. 1,3-dichloropropane	79. 2-chlorotoluene	
20. allyl alcohol (250ppb)	40. isopropyl acetate	60. <i>n</i> -butyl acetate	80. 1,2,3-trichloropropane	

GC_EV00427

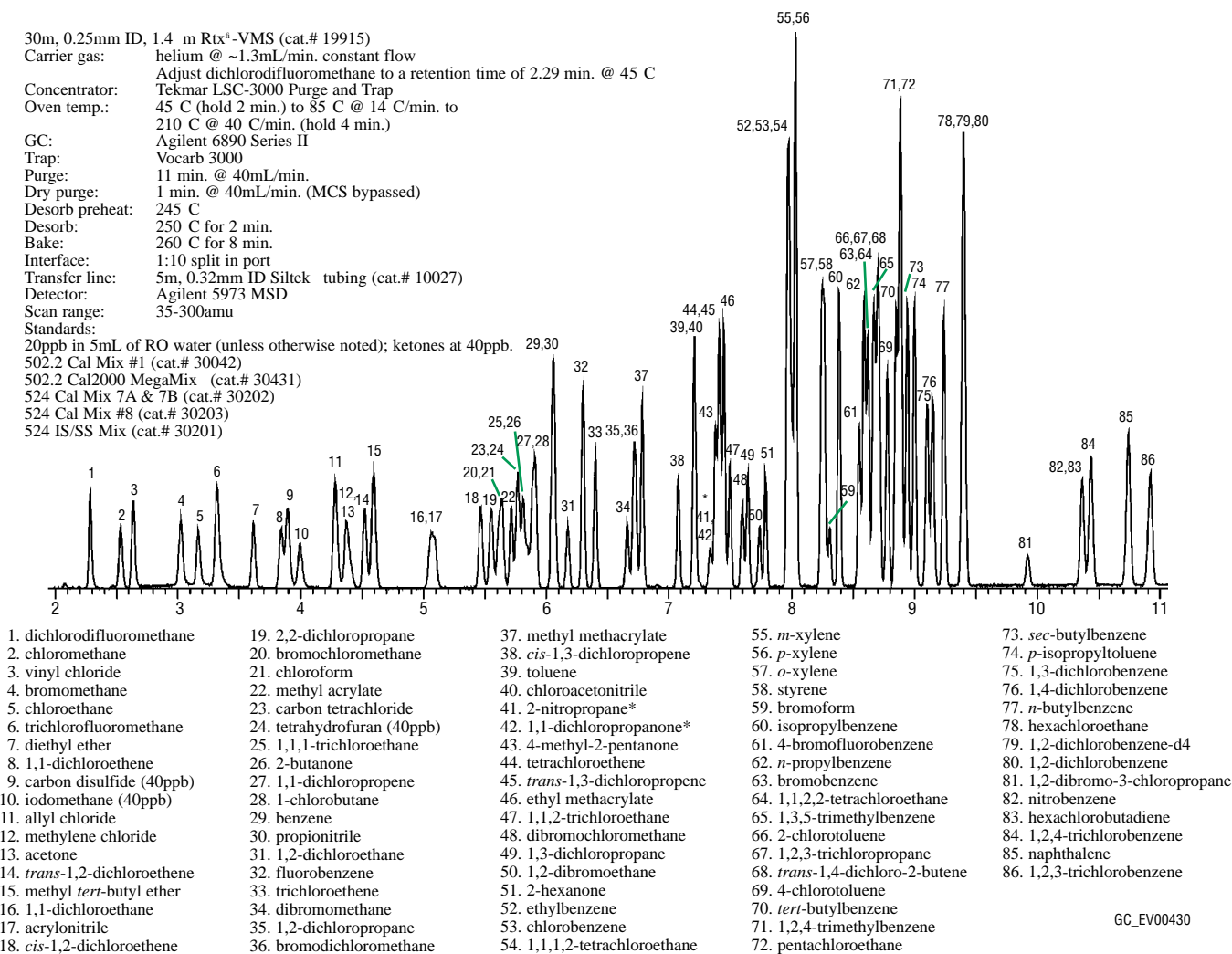
*These compounds share ions, and coelute on "624"-type columns.

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Figure 50.

Volatile Organics by US EPA Method 524.2, rev. IV. A 30m x 0.25mm ID column and 11 minute analysis provide excellent resolution of gaseous VOAs.

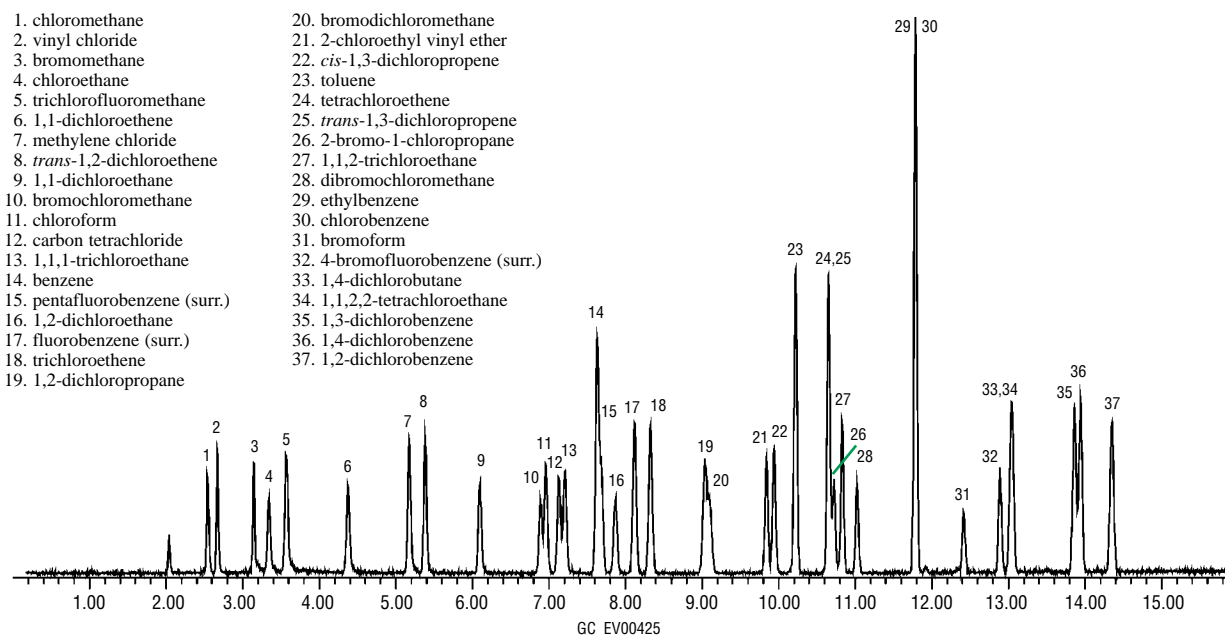
30m, 0.25mm ID, 1.4 m Rtx®-VMS (cat.# 19915)
 Carrier gas: helium @ ~1.3mL/min. constant flow
 Adjust dichlorodifluoromethane to a retention time of 2.29 min. @ 45 C
 Concentrator: Tekmar LSC-3000 Purge and Trap
 Oven temp.: 45 C (hold 2 min.) to 85 C @ 14 C/min. to 210 C @ 40 C/min. (hold 4 min.)
 GC: Agilent 6890 Series II
 Trap: Vocab 3000
 Purge: 11 min. @ 40mL/min.
 Dry purge: 1 min. @ 40mL/min. (MCS bypassed)
 Desorb preheat: 245 C
 Desorb: 250 C for 2 min.
 Bake: 260 C for 8 min.
 Interface: 1:10 split in port
 Transfer line: 5m, 0.32mm ID Siltek tubing (cat.# 10027)
 Detector: Agilent 5973 MSD
 Scan range: 35-300amu
 Standards:
 20ppb in 5mL of RO water (unless otherwise noted); ketones at 40ppb.
 502.2 Cal Mix #1 (cat.# 30042)
 502.2 Cal2000 MegaMix (cat.# 30431)
 524 Cal Mix 7A & 7B (cat.# 30202)
 524 Cal Mix #8 (cat.# 30203)
 524 IS/SS Mix (cat.# 30201)



*These compounds share a quantitation ion (43)

Method 524.2: In EPA method updates, such as Method 524.2, rev. IV, minor ions from newly listed target compounds interfere with the quantification ions from other target compounds.¹⁷ An example of this problem occurs when a 75m, 0.53mm ID “624/1301” column is used to resolve methyl acrylate and propionitrile. The quantification ion for methyl acrylate is mass 55, and propionitrile has a minor ion of mass 55, which thus can interfere with determining concentrations of methyl acrylate in “real world” samples. 1,1-dichloro-2-propanone and 4-methyl-2-pentanone are another difficult pair to resolve on a “624/1301” column, because they share ion 43. These compounds can be resolved (in more than 30 minutes), however, by using a 60m, 0.32mm ID column. Because Rtx®-VMS columns were designed to resolve compounds by primary quantification ion, using extracted ion chromatography (Figure 50), the only compounds from Method 524.2, rev. IV, that are difficult for an Rtx®-VMS column to resolve are 2-nitropropane and 1,1-dichloro-2-propanone, (peaks 41 and 42) which share ion 43.

17. *Methods for the Determination of Organic Compounds in Drinking Water, Supplement II Method 524.2: U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring Systems Laboratory, Cincinnati, OH. 1992.*

Figure 51.*Gaseous analytes resolved to baseline on an Rtx®-VMS column.*

30m, 0.25mm ID, 1.40 m Rtx®-VMS (cat#19915)

Conc.: 20 ppb in 5mL of RO water

Concentrator: Tekmar LSC-3000 Purge and Trap

Trap: Vocab 3000 (type K)

Purge: 11 min. @ 40mL/min. @ ambient temperature.

Dry purge: 1 min. @ 40mL/min. (MCS bypassed using Silcosteel® tubing)

Desorb preheat: 245 C

Desorb: 250 C for 2 min., Flow 10mL/min.

Bake: 260 C for 8 min.

GC Interface: 1:10 split at injection port. 1mm ID sleeve.

GC: HP 6890

Oven temp.: 40 C (hold 4 min.) to 95 C @ 24 C/min. (hold 3 min.), to

210 C @ 40 C/min. (hold 6 min.)

Carrier gas: helium @ ~1mL/min. constant flow

Adjust dichlorodifluoromethane to a retention time of 2.54 min. @ 40 C

Detector: HP 5973 MSD

Scan range: 25-300amu

Method 624: Previously, analysts used packed columns to perform Method 624, but now they generally use capillary chromatographic techniques. The compound list in Method 624 includes commonly analyzed aromatic and halogenated compounds in wastewater. This analysis can be achieved on many capillary columns; Figure 51 shows a 30m, 0.25mm ID Rtx®-VMS column resolves the gaseous analytes to the baseline.

Table VIII

Volatile organic compounds: retention time indexes

Data collected using 105m, 0.53mm ID, 3.0µm Rtx®-502.2, Rtx®-1, and Rtx®-624 columns.

Oven temp.: Rtx®-502.2: 35°C (hold 10 min.) to 220°C @ 4°C/min. (hold 2 min.), Rtx®-1: 35°C (hold 10 min.) to 220°C @ 4°C/min. (hold 2 min.), Rtx®-624: 35°C (hold 12 min.) to 220°C @ 7°C/min.; Carrier gas: helium; Regulation: constant flow; Flow rate: 10.0mL/min.

Retention Time				Retention Time				Retention Time			
Component Name	Rtx-502.2	Rtx-1	Rtx-624	Component Name	Rtx-502.2	Rtx-1	Rtx-624	Component Name	Rtx-502.2	Rtx-1	Rtx-624
dichlorodifluoromethane	4.173	4.24	4.255	1,1-dichloropropene	20.163	17.808	19.448	m-xylene	33.351	30.898	28.37
C3	4.181	4.241	4.211	carbon tetrachloride	20.502	18.493	19.333	p-xylene	33.354	30.961	28.358
chloromethane	4.838	4.513	4.857	1,2-dichloroethane-d4	20.726	16.133	20.04	1-chloro-2-fluorobenzene	33.68	30.232	28.605
C4	5.136	5.319	6.321	benzene-d6	20.91	17.998	19.888	malononitrile	34.623	26.078	31.796
vinyl chloride	5.159	4.872	5.243	C7	20.935	21.532	20.582	o-xylene	34.877	32.149	29.238
ethylene oxide	6.289	5.467	6.389	1,2-dichloroethane	21.047	16.411	20.263	styrene	34.975	31.843	29.294
bromomethane	6.527	5.604	6.412	benzene	21.097	18.124	19.999	1,3-dichloro-2-propanol	36.032	31.018	31.41
chloroethane	6.812	5.938	6.798	fluorobenzene	21.897	18.917	20.802	bromoforn	36.123	30.983	29.746
ethanol	7.109	6.261	9.056	2-chloroethanol	21.951	17.171	23.115	isopropylbenzene	36.139	33.718	30.014
C5	7.692	8.133	8.069	trichloroacetonitrile	22.125	33.558	30.181	cis-1,4-dichloro-2-butene	36.312	31.594	30.302
trichlorofluoromethane	7.715	7.426	7.672	1,4-difluorobenzene	22.209	19.061	21.179	1,4-dichlorobutane	36.471	31.864	30.377
diethyl ether	8.857	8.232	8.977	trichloroethene	23.326	20.674	21.713	1,1,2,2-tetrachloroethane	36.735	32.053	30.779
1,1,2-trichloro-1,2,2-trifluoroethane	9.142	9.745	10.044	trifluorotoluene	23.607	21.311	22.527	4-bromofluorobenzene	37.004	33.318	30.426
acrolein	9.285	6.831	9.817	1,2-dichloropropane	23.924	20.036	22.378	1,2,3-trichloropropane	37.233	32.402	30.902
acetone	9.558	7.042	10.6	methyl methacrylate	24.233	21.258	22.632	C10	37.305	37.539	31.095
1,1-dichloroethene	10.117	8.74	9.953	chloroacetonitrile	24.239	17.035	23.993	trans-1,4-dichloro-2-butene	37.516	32.626	30.929
tert-butyl alcohol	10.516	8.864	13.546	bromodichloromethane	24.743	20.545	23.045	n-propylbenzene	37.566	35.151	30.895
acetonitrile	10.661	6.571	12.909	1,4-dioxane	24.808	20.645	22.644	bromobenzene	37.652	33.875	30.772
iodomethane	11.311	8.567	10.634	dibromomethane	24.881	19.975	22.668	dibromoacetonitrile	37.812	30.956	31.86
methyl iodide	11.311	8.567	10.634	2-nitropropane	24.999	19.356	23.762	1,3,5-trimethylbenzene	38.133	35.817	31.268
allyl chloride	11.717	9.323	11.9	2-chloroethyl vinyl ether	25.888	22.108	23.796	2-chlorotoluene	38.166	34.986	31.145
n-propylamine	11.815	9.36	11.69	dichloroacetonitrile	25.894	19.998	25.478	4-chlorotoluene	38.33	35.214	31.4
carbon disulfide	12.069	9.658	10.747	4-methyl-2-pentanone (MIBK)	26.001	22.887	24.458	tert-butylbenzene	39.393	37.008	31.959
dichloromethane	12.169	9.001	13.034	epichlorohydrin	26.109	20.907	23.971	1,2,4-trimethylbenzene	39.521	37.033	32.089
methylene chloride	12.169	9.001	13.034	1,1-dichloropropanone	26.444	22.124	25.469	pentachloroethane	39.576	35.98	32.088
allyl alcohol	12.659	10.215	16.115	cis-1,3-dichloropropene	26.674	22.744	24.104	thiophenol	39.606	35.186	31.919
methyl tert-butyl ether	12.785	12.074	13.734	C8	27.186	27.642	24.868	sec-butylbenzene	40.151	37.886	32.428
acrylonitrile	13.429	7.947	14.158	toluene-d8	27.502	24.835	24.612	p-isopropyltoluene	40.657	38.413	32.731
trans-1,2-dichloroethene	13.438	11.346	13.725	pyridine	27.525	22.8	24.598	1,3-dichlorobenzene	40.938	37.404	32.765
C6	13.828	14.557	14.668	toluene	27.78	25.081	24.768	1,4-dichlorobenzene-d4	41.228	37.544	32.92
propargyl alcohol	14.458	11.32	18.532	trans-1,3-dichloropropene	28.428	24.014	25.417	1,4-dichlorobenzene	41.317	37.624	32.972
di-isopropyl ether	14.874	14.602	15.745	ethyl methacrylate	28.455	25.852	25.489	C11	41.585	41.76	33.638
1,1-dichloroethane	15.151	11.849	15.616	1,1,2-trichloroethane	28.925	24.384	25.829	benzyl chloride	41.739	37.378	33.279
vinyl acetate	15.256	12.283	15.883	2-bromo-1-chloropropane	28.965	25.018	25.569	n-butylbenzene	42.12	39.764	33.612
2-butanone	16.82	12.834	17.766	2-hexanone	29.03	25.778	26.326	bis(2-chloroisopropyl)ether	42.498	39.009	34.229
methyl ethyl ketone	16.82	12.834	17.766	1,3-dichloropropane	29.826	25.124	26.205	1,2-dichlorobenzene-d4	42.536	38.589	33.74
propionitrile	17.053	10.733	18.167	tetrachloroethene	30.036	27.8	25.928	1,2-dichlorobenzene	42.62	38.685	33.8
2,2-dichloropropane	17.337	14.659	17.506	dibromochloromethane	30.685	26.024	26.639	hexachloroethane	43.898	40.901	34.244
cis-1,2-dichloroethene	17.478	13.924	17.628	2-picoline	31.242	27.462	26.977	3-bromochlorobenzene	45.335	41.342	35.231
methacrylonitrile	17.837	12.732	18.505	1,2-dibromoethane (EDB)	31.375	26.613	26.924	1,2-dibromo-3-chloropropane	45.501	40.095	35.485
methylacrylate	17.947	14.54	18.351	1-chlorohexane	32.066	29.717	27.831	4-bromochlorobenzene	45.567	41.476	35.377
isobutyl alcohol	18.015	15.723	20.048	bromochloroacetonitrile	32.079	25.757	28.837	C12	45.586	45.659	35.96
chloroform	18.104	14.659	18.625	C9	32.546	32.893	28.243	nitrobenzene	45.742	40.282	35.992
pentafluorobenzene	18.628	16.371	19.273	1,1,1-trichloro-2-propanone	32.633	28.967	28.104	2-bromochlorobenzene	46.885	42.47	36.162
bromochloromethane	18.667	14.289	18.355	chlorobenzene-d5	32.749	29.372	27.887	bis(2-chloroethoxy)methane	47.514	42.935	36.98
tetrahydrofuran	18.777	15.554	18.341	ethylbenzene-d10	32.756	30.191	27.934	1,2,4-trichlorobenzene	48.643	44.704	37.165
dibromofluoromethane	18.893	14.966	19.086	chlorobenzene	32.864	29.473	27.949	hexachlorobutadiene	49.219	46.558	37.418
1,1,1-trichloroethane	19.575	17.042	18.972	ethylbenzene-d5	32.976	30.378	28.089	C13	49.276	49.295	38.078
cyclohexane	19.619	18.779	16.854	3-chloropropionitrile	33.001	24.824	29.176	naphthalene	49.544	45.073	37.729
1-chlorobutane	19.724	17.221	19.314	1,1,1,2-tetrachloroethane	33.03	29.429	28.133	1,2,3-trichlorobenzene	50.481	46.076	38.23
				ethylbenzene	33.087	30.476	28.118	C14	52.771	52.704	40.173

Table IX

Volatile organic compounds: retention time indexes

Data collected using a 60m, 0.25mm ID, 1.4µm Rtx®-VMS column; Oven: 40°C (hold 6 min.) to 230°C @ 14°C/min. (hold 11 min); Carrier gas: helium; Regulation: constant pressure; Flow rate: 1mL/min.; Linear velocity: 21cm/sec.; Dead time: 4.90 min.

Component Name	Rtx®-VMS Ret. Time	Component Name	Rtx®-VMS Ret. Time	Component Name	Rtx®-VMS Ret. Time	Component Name	Rtx®-VMS Ret. Time
dichlorodifluoromethane	5.52	methyl acrylate	13.87	4-methyl-2-pentanone	17.76	<i>trans</i> -1,4-dichloro-2-butene	21.40
chloromethane	6.26	carbon tetrachloride	13.94	2-bromo-1-chloropropane	17.81	2-chlorotoluene	21.40
vinyl chloride	6.54	tetrahydrofuran	14.03	2-nitropropane	17.83	4-chlorotoluene	21.61
water	6.70	1,1,1-trichloroethane	14.06	pyridine	17.86	cyclohexane	21.78
bromomethane	7.61	ethyl acetate	14.13	1,1-dichloropropanone	17.88	<i>tert</i> -butylbenzene	21.81
methanol	7.93	2-butanone	14.18	<i>trans</i> -1,3-dichloropropene	17.88	1-ethyl-2-methylbenzene	21.82
2-methylbutane	7.96	dibromofluoromethane	14.18	tetrachloroethene	17.89	1,2,4-trimethylbenzene	21.88
chloroethane	8.00	1,1-dichloropropene	14.20	ethyl methacrylate	17.92	pentachloroethane	21.92
trichlorofluoromethane	8.41	propargyl alcohol	14.35	1,1,2-trichloroethane	18.11	1,3-dichloro-2-propanol	22.05
<i>n</i> -pentane	8.61	1-chlorobutane	14.51	dibromochloromethane	18.40	<i>sec</i> -butylbenzene	22.06
diethylether	9.59	2,2,4-trimethylpentane	14.53	1,3-dichloropropane	18.49	isocaproic acid	22.09
1,1-dichloroethene	9.64	propionitrile	14.59	isobutyric acid	18.55	<i>p</i> -isopropyltoluene	22.22
carbon disulfide	9.65	benzene	14.60	1,2-dibromoethane	18.78	1,3-dichlorobenzene	22.53
Freon® 113	9.70	<i>n</i> -heptane (C7)	14.62	<i>n</i> -butyl acetate	18.80	caproic acid	22.55
ethanol	9.74	methacrylonitrile	14.64	2-hexanone	18.82	1,4-dichlorobenzene	22.64
iodomethane	9.99	benzene-d6	14.72	butyric acid	19.17	<i>n</i> -butylbenzene	22.88
3-chlorotrifluoropropane	10.45	pentafluorobenzene	14.75	1-chloro-3-fluorobenzene	19.17	malononitrile	22.89
chloro-methyl-methylether	10.54	1,2-dichloroethane-d4	14.79	ethylbenzene	19.36	benzyl chloride	23.23
acrolein	10.57	1,2-dichloroethane	14.90	chlorobenzene	19.39	1,2-dichlorobenzene-d4	23.36
2-methylpentane	10.59	<i>tert</i> -amyl-methyl ether	15.00	1-chloro-4-fluorobenzene	19.39	1,2-dichlorobenzene	23.38
allyl chloride	10.72	isobutyl alcohol	15.06	ethylbenzene-d10	19.40	hexachloroethane	23.63
methylene chloride	10.98	fluorobenzene	15.16	1-chlorohexane	19.41	1-octanol	23.70
3-methylpentane	11.09	isopropyl acetate	15.34	1,1,1,2-tetrachloroethane	19.44	<i>bis</i> (2-chloroisopropyl) ether	24.06
acetone	11.24	formic acid	15.37	<i>m</i> -xylene	19.53	4-bromo-1-chlorobenzene	24.09
<i>trans</i> -1,2-dichloroethene	11.24	trichloroethene	15.39	<i>p</i> -xylene	19.54	benzyl alcohol	24.23
methyl <i>tert</i> -butyl ether	11.42	1,4-difluorobenzene	15.58	chlorobenzene-d5	19.55	heptanoic acid	24.29
2-propanol	11.52	<i>n</i> -butanol	15.60	1-chloro-2-fluorobenzene	19.67	<i>n</i> -dodecane	24.54
<i>tert</i> -butyl alcohol	11.56	methyl cyclohexane	15.78	<i>o</i> -xylene	20.13	3-bromochlorobenzene	24.61
methyl acetate	11.63	acetic acid	15.93	styrene	20.17	1,2-dibromo-3-chloropropane	24.78
hexane	11.64	dibromomethane	16.05	isovaleric acid	20.18	2-bromochlorobenzene	25.54
acetonitrile	12.22	1,2-dichloropropane	16.17	bromoform	20.30	hexachlorobutadiene	25.99
chloroprene	12.30	bromodichloromethane	16.23	isopropylbenzene	20.51	nitrobenzene	26.02
1,1-dichloroethane	12.42	methyl methacrylate	16.28	1,2-butanediol	20.82	1,2,4-trichlorobenzene	26.19
acrylonitrile	12.60	α,α,α -trifluorotoluene	16.45	valeric acid	20.89	benzyl acetate	26.29
diisopropyl ether	12.62	1,4-dioxane	16.49	1,4-dichlorobutane	20.90	<i>n</i> -tridecane	26.51
2,4-dimethylpentane	12.68	<i>n</i> -propyl acetate	16.70	bromobenzene	20.91	naphthalene	27.01
vinyl acetate	13.02	2-chloroethyl vinyl ether	16.92	4-bromo-1-fluorobenzene	20.95	1,2,3-trichlorobenzene	27.46
ethyl- <i>tert</i> -butyl ether	13.08	2-chloroethanol	16.93	<i>cis</i> -1,4-dichloro-2-butene	20.97	<i>n</i> -tetradecane	28.83
1-propanol	13.18	<i>cis</i> -1,3-dichloropropene	17.04	<i>n</i> -decane	21.04	2-methylnaphthalene	30.36
<i>cis</i> -1,2-dichloroethene	13.32	1-bromo-2-chloroethane	17.05	<i>n</i> -propylbenzene	21.07	1-methyl-naphthalene	30.96
allyl alcohol	13.35	<i>n</i> -octane	17.17	1,1,2,2-tetrachloroethane	21.10	<i>n</i> -pentadecane	31.65
2,2-dichloropropane	13.48	toluene-d8	17.28	1,3,5-trimethylbenzene	21.30	2-chloronaphthalene	33.36
bromochloromethane	13.62	toluene	17.36	1-ethyl-4-methylbenzene	21.33		
chloroform	13.75	propionic acid	17.61	1-ethyl-3-methylbenzene	21.34		
cyclohexane	13.84	chloroacetonitrile	17.64	1,2,3-trichloropropane	21.39		

Choosing Columns for Subsets of Method 502.2/8021 Compound Lists

Table X shows the elution times for compounds listed in EPA Method 502.2 under the GC conditions given. Analysts monitoring subsets of Method 502.2 compounds may find one of these columns to be more suitable for the analysis than a commonly used volatile phase.

Table X.

Elution times for Method 502.2 compounds on various Rtx® GC columns.

Column: 60m x 0.53mm x 3.00 mdf
Flow: ~10mL/min., dead time 1.80 min.
Oven: 35 C (hold 9 min.) to 220 C @ 11 min. (hold 10 min.)

Elution Time in Minutes	Rtx-VGC	Rtx-502.2	Rtx-VRX	Rtx-624	Rtx-1	Rtx-1701	Rtx-200	Rtx-35	Rtx-50
dichlorodifluoromethane	2.31	2.18	2.54	2.59	2.16	1.99	1.95	2.04	1.96
chloromethane	2.64	2.51	2.75	2.94	2.30	2.36	2.18	2.39	2.39
vinyl chloride	2.78	2.67	2.99	3.16	2.51	2.51	2.23	2.51	2.47
bromomethane	3.35	3.35	3.46	3.86	2.89	3.16	2.62	3.21	3.27
chloroethane	3.58	3.44	3.66	4.07	3.04	3.32	2.85	3.21	3.27
trichlorofluoromethane	3.83	3.91	4.50	4.56	3.80	3.45	2.85	3.46	3.25
1,1-dichloroethene	4.83	4.93	5.32	5.75	4.74	4.57	3.34	4.58	4.46
Freon® 113	4.91	4.61	5.77	5.68	4.93	4.17	3.56	3.78	3.38
methylene chloride	6.10	6.02	5.68	7.39	4.62	6.64	3.97	6.07	6.40
trans-1,2-dichloroethene	6.49	6.78	7.07	8.07	5.85	6.68	4.20	6.40	6.59
methyl-tert-butyl-ether	6.80	6.67	7.68	8.21	6.28	6.34	4.88	5.76	5.52
tert-butyl alcohol	7.19	5.76	5.99	8.40	4.62	7.61	4.86	5.08	5.48
1,1-dichloroethane	8.10	8.08	7.74	9.74	6.16	8.68	5.54	8.00	8.30
cis-1,2-dichloroethene	9.80	10.14	9.47	11.51	7.59	10.75	6.12	10.27	10.50
2,2-dichloropropane	10.10	10.01	10.24	11.37	8.22	10.15	7.41	9.60	9.41
bromochloromethane	10.33	11.05	9.88	12.13	7.91	11.66	6.12	11.46	12.06
chloroform	10.61	10.65	10.11	12.38	8.21	11.79	5.69	10.66	11.02
carbon tetrachloride	10.87	12.31	12.60	12.87	11.39	11.26	6.44	11.80	11.72
1,1,1-trichloroethane	11.07	11.68	11.85	12.59	10.28	11.31	7.59	11.40	11.25
1,1-dichloropropene	11.39	12.09	12.31	12.98	10.89	11.67	7.79	11.74	11.62
benzene	11.97	12.72	12.70	13.43	11.13	12.44	8.66	12.86	12.95
1,2-dichloroethane	12.44	12.73	11.67	13.70	8.78	13.38	9.52	12.90	13.60
fluorobenzene	12.90	13.22	13.24	14.11	11.70	13.27	10.55	13.25	13.38
trichloroethene	13.24	14.08	14.09	14.83	12.89	13.79	9.52	13.93	13.98
dibromomethane	14.03	14.97	13.89	15.63	12.47	15.23	10.03	15.51	16.02
1,2-dichloropropane	14.25	14.44	14.00	15.39	12.51	14.83	12.04	14.54	14.77
bromodichloromethane	14.39	14.92	14.20	15.94	12.83	15.48	9.68	15.22	15.60
2-chloroethyl vinyl ether	15.48	15.57	14.92	16.52	13.80	16.08	13.32	15.81	16.01
cis-1,3-dichloropropene	15.54	15.96	15.46	16.76	14.18	16.21	13.19	16.20	16.36
toluene	15.95	16.53	16.74	17.23	15.48	16.30	13.55	16.58	16.57
tetrachloroethene	16.58	17.67	17.92	18.13	16.94	16.88	13.77	17.51	17.35
trans-1,3-dichloropropene	16.61	16.87	16.23	17.78	14.91	17.39	14.47	17.16	17.45
2-bromo-1-chloropropane	16.71	17.13	16.48	17.88	15.46	17.28	14.43	17.32	17.52
1,1,2-trichloroethane	16.91	17.13	16.45	18.10	15.13	17.76	14.23	17.49	17.74
dibromochloromethane	17.18	18.01	17.20	18.71	16.03	18.28	13.30	18.50	18.86
1,3-dichloropropane	17.36	17.57	16.86	18.38	15.33	17.99	15.64	17.91	18.10
1,2-dibromoethane	17.56	18.35	17.58	18.92	16.34	18.46	14.49	18.77	19.16
chlorobenzene	18.42	19.05	18.97	19.69	17.79	18.91	16.31	19.27	19.38
ethyl benzene	18.47	19.15	19.31	19.79	18.26	18.83	16.14	19.25	19.09
1,1,1,2-tetrachloroethane	18.53	19.13	18.87	19.83	17.78	19.14	16.02	19.29	19.39
m-xylene	18.69	19.28	19.62	19.98	18.48	19.02	16.42	19.27	19.17
p-xylene	18.71	19.28	19.62	19.99	18.48	19.02	16.45	19.27	19.17
1-chloro-2-fluorobenzene	19.08	19.44	19.14	20.20	18.16	19.50	17.28	19.61	19.67
o-xylene	19.37	20.01	20.22	20.66	19.07	19.74	17.26	20.07	20.02
styrene	19.45	20.04	20.10	20.69	18.93	19.92	17.44	20.25	20.33
bromoform	19.49	20.56	19.96	21.08	18.58	20.64	15.88	21.19	21.63
isopropyl benzene	19.86	20.60	20.77	21.23	19.80	20.20	17.54	20.54	20.35
bromobenzene	20.47	21.30	21.09	21.83	19.92	21.08	18.32	21.65	21.78
n-propyl benzene	20.51	21.22	21.43	21.89	20.46	20.87	18.20	21.19	20.99
1,4-dichlorobutane	20.56	20.71	19.95	21.54	18.94	21.14	19.02	21.01	21.16
1,1,2,2-tetrachloroethane	20.62	20.83	20.21	21.84	19.06	21.49	17.72	21.19	21.41
2-chlorotoluene	20.76	21.47	21.55	22.10	20.42	21.22	18.66	21.68	21.66
1,3,5-trimethylbenzene	20.81	21.52	21.88	22.17	20.75	21.15	18.50	21.42	21.31
1,2,3-trichloropropane	20.84	21.08	20.42	21.93	19.22	21.61	18.81	21.45	21.65
4-chlorotoluene	21.03	21.59	21.67	22.28	20.52	21.46	19.04	21.68	21.76
tert-butylbenzene	21.32	22.04	22.26	22.69	21.31	21.62	19.06	21.98	21.72
1,2,4-trimethylbenzene	21.42	22.11	22.44	22.78	21.32	21.79	19.15	22.09	22.02
sec-butylbenzene	21.60	22.38	22.59	23.04	21.69	21.95	19.16	22.29	22.04
p-isopropyl toluene	21.81	22.60	22.87	23.26	21.91	22.17	19.30	22.49	22.24
1,3-dichlorobenzene	21.95	22.77	22.66	23.33	21.54	22.49	19.87	22.95	23.03
1,4-dichlorobenzene	22.09	22.93	22.77	23.48	21.64	22.68	20.00	23.15	23.30
n-butylbenzene	22.47	23.25	23.46	23.92	22.53	22.85	20.12	23.18	22.89
1,2-dichlorobenzene	22.76	23.52	23.30	24.10	22.13	23.36	20.73	23.84	23.96
4-bromo-1-chlorobenzene	23.77	24.82	22.18	25.28	23.37	24.50	21.55	25.15	25.33
1,2-bromo-3-chloropropane	24.02	24.75	23.96	25.38	22.78	24.98	21.70	25.26	25.54
hexachlorobutadiene	25.03	26.37	26.42	26.82	25.55	25.53	22.42	26.41	26.15
1,2,4-trichlorobenzene	25.13	26.16	25.95	26.66	24.79	25.77	22.96	26.37	26.55
naphthalene	25.64	26.61	26.30	27.13	24.98	26.40	23.70	27.18	27.45
1,2,3-trichlorobenzene	25.94	27.04	26.63	27.57	25.41	26.72	23.81	27.46	27.65

Table XI.
Choosing a Volatiles GC Column for PID/ELCD

Restek Rtx® Phase	Coelutions by Peak #'s (Coelutions by PID/ELCD are indicated in BOLD)	Close Pairs by PID/ELCD	Suggested Confirmation Column	Poor Choice for Confirmation Column	Thick Phase Stable Temp. (°C)	Recommended High Temp. for VOA Work (°C)	Advantages
Rtx-VGC	28/29 ¹ , 53/54	7/8,32/33	Rtx-502.2, Rtx-VRX, Rtx-1	Rtx-VMS, Rtx-DX1	260	230	fast runtime
Rtx-502.2	14/15, 33/34, 39/40	4/5, 44/45, 56/57, 52/55, 64/65	Rtx-VGC, Rtx-VRX, Rtx-1	Rtx-Volatiles, Vocol, Rtx-35, Rtx-20	270	240	low bleed
Rtx-Volatiles	14/15, 21/22, 38/40, 44/45, 53/55	56/57, 68/69	Rtx-VGC, Rtx-1, Rtx-624	Rtx-502.2, Vocol, Rtx-20, Rtx-35	270	240	low bleed
Rtx-624	7/8, 10/11, 52/53, 31/34, 53/55, 59/60	32/33, 44/45, 51/54	Rtx-VGC, Rtx-502.2	Rtx-1701	280	240	
Rtx-VRX	11/13, 39/43, 46/50, 40/44	8/9,15/17, 24/27, 58/60	Rtx- VGC, Rtx-502.2	Rtx-1	260	230	
Rtx-1	9/12, 15/17, 25/26, 24/27, 33/36, 38/40, 40/44, 45/50, 56/57	7/12, 49/55	Rtx-502	Rtx-VRX	320	260	
Rtx-1701	9/10, 18/19, 16/20, 50/53, 51/55, 54/56	5/6,29/30,32/33	Rtx-502.2	Rtx-624	270	240	
Rtx-200	2/3, 5/6, 11/12, 14/16, 22/24, 28/35, 32/33/37, 43/44, 50/55/56, 57/58	13/17,36/37	Rtx-VGC		320	240	m/p xylene separation
Rtx-35	4/5, 16/19, 18/20, 21/22, 34/31, 39/38/41/42/40, 46/51/49, 53/54, 48/52/55, 61/62, 66/67	2/3	Rtx-VGC, Rtx-624	Rtx-502.2, Rtx-Volatiles, Rtx-20	270	240	
Rtx-50	4/5/6, 8/7/12, 25/28, 32/33, 37/41/42, 38/40, 45/47, 46/54/52, 56/55/48, 57/58	2/3, 20/18, 31/32, 39/41/42	Rtx-VGC, Rtx-624	Rtx-35	280	240	
Rtx-DX1 (custom)	4/5, 9/10, 25/27, 38/39, 47/50, 49/46/48, 52/54, 53/55	27/28, 32/36/31, 65/67	Rtx-502.2	Rtx-VGC	220	200	

¹ 2-chloroethyl vinyl ether can be resolved under different conditions. See the applications section of our current catalog.

Volatile Analytes:

- | | | | |
|--------------------------------------|---------------------------------------|-------------------------------|---------------------------------|
| 1. dichlorodifluoromethane | 19. 1,1,1-trichloroethane | 37. 1,2-dibromoethane | 55. 4-chlorotoluene |
| 2. chloromethane | 20. 1,1-dichloropropene | 38. chlorobenzene | 56. <i>tert</i> -butylbenzene |
| 3. vinyl chloride | 21. benzene | 39. ethyl benzene | 57. 1,2,4-trimethylbenzene |
| 4. bromomethane | 22. 1,2-dichloroethane | 40. 1,1,1,2-tetrachloroethane | 58. <i>sec</i> -butylbenzene |
| 5. chloroethane | 23. fluorobenzene | 41. <i>m</i> -xylene | 59. <i>p</i> -isopropyl toluene |
| 6. trichlorofluoromethane | 24. trichloroethene | 42. <i>p</i> -xylene | 60. 1,3-dichlorobenzene |
| 7. 1,1-dichloroethene | 25. dibromomethane | 43. 1-chloro-2-fluorobenzene | 61. 1,4-dichlorobenzene |
| 8. Freon ®113 | 26. 1,2-dichloropropane | 44. <i>o</i> -xylene | 62. <i>n</i> -butylbenzene |
| 9. methylene chloride | 27. bromodichloromethane | 45. styrene | 63. 1,2-dichlorobenzene |
| 10. <i>trans</i> -1,2-dichloroethene | 28. 2-chloroethyl vinyl ether | 46. bromoform | 64. 4-bromo-1-chlorobenzene |
| 11. methyl <i>tert</i> -butyl ether | 29. <i>cis</i> -1,3-dichloropropene | 47. isopropyl benzene | 65. 1,2-bromo-3-chloropropane |
| 12. <i>tert</i> -butyl alcohol | 30. toluene | 48. bromobenzene | 66. hexachlorobutadiene |
| 13. 1,1-dichloroethane | 31. tetrachloroethene | 49. <i>n</i> -propylbenzene | 67. 1,2,4-trichlorobenzene |
| 14. <i>cis</i> -1,2-dichloroethene | 32. <i>trans</i> -1,3-dichloropropene | 50. 1,4-dichlorobutane | 68. naphthalene |
| 15. 2,2-dichloropropane | 33. 2-bromo-1-chloropropane | 51. 1,1,2,2-tetrachloroethane | 69. 1,2,3-trichlorobenzene |
| 16. bromochloromethane | 34. 1,1,2-trichloroethane | 52. 2-chlorotoluene | |
| 17. chloroform | 35. dibromochloromethane | 53. 1,3,5-trimethylbenzene | |
| 18. carbon tetrachloride | 36. 1,3-dichloropropane | 54. 1,2,3-trichloropropane | |

Compounds listed in US EPA Methods 502.2, 8021, 8010, 8020, 601 & 602, plus commonly added compounds.

m/p xylene coelute on all phases except Rtx®-200 in 60m, 0.25mm ID, 1.0µm under optimized conditions. See the applications section of our current catalog.

Conditions for Rtx®-VGC, Rtx®-502.2, Rtx®-Volatiles, Rtx®-VRX and Rtx®-1: optimum conditions on 75m, 0.45mm ID, 2.55µm columns. For more details please see chromatograms in the applications section of our current catalog or call technical service (1-800-356-1688, ext. 4) or your Restek representative.

Conditions for all other columns:

Column: 60m, 0.53mm ID, 3.0µm

Column Flow: 10mL/min.

GC Program: 35°C (hold 9 min.) to 220°C @ 11 min. (hold 10 min.)

Analytes identified using Thermo Finnigan PID/ELCD or HP5971a mass selective detector with splitless injection.

Conclusion

Analyses of volatile organic compounds – VOCs – generally require concentration of the sample using dynamic headspace (purge and trap), which reduces matrix effects and increases sensitivity, relative to other extraction techniques. The concentrated sample is transferred to the capillary GC column. Because GC detectors operate well with higher flows (10mL/min.), wide-bore columns, 0.45mm ID to 0.53mm ID, are appropriate. GC/MS instruments have the greatest sensitivity, but flow into the MS source cannot exceed 1mL/min. This makes narrow-bore columns, usually 0.18mm ID to 0.25mm ID, but up to 0.32mm ID, the preferred choice for GC/MS analysis. The column is plumbed through the injection port to allow high desorb flows (>10mL/min.) and sample splitting at the injection port (>10:1). Determining the type of stationary phase in the capillary column is a matter of preference and requires an examination of conditions specific to the analysis to be performed.

Information in this guide explains many of the factors to be considered in analyses of VOCs, but cannot anticipate every situation. If you have any questions regarding this guide, or your particular application, please contact our Technical Service Team via email at support@restekcorp.com or via phone at 800-356-1688 or 814-353-1300, ext. 4.

Recommended Products

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Note: Many additional reference materials, including custom mixes, are available from Restek. Please call us at 800-356-1688, or contact your Restek distributor; we'll help you find what you're looking for.

Rtx®-VMS

- Special polymer formulation designed specifically for volatiles analysis by GC/MS.
- Complete separation of all US EPA Method 8260 compounds in less than 18 minutes.
- Excellent thermal stability resulting in low bleed.
- Wide variety of column dimensions.

Ordering Information | Rtx®-VMS (Fused Silica)

ID	df (µm)	temp. limits	30-Meter	60-Meter	75-Meter
0.25mm	1.40	-40 to 240/260°C	19915	19916	
0.45mm	2.55	-40 to 240/260°C	19908	19909	
0.53mm	3.00	-40 to 240/260°C	19985	19988	19974
ID	df (µm)	temp. limits	20-Meter	40-Meter	
0.18mm	1.00	-40 to 240/260°C	49914	49915	

Rtx®-VGC

- Special polymer formulation designed for volatiles analysis using PID/ELCD.
- Performs US EPA Method 8021A analysis in less than 28 minutes.
- Excellent separation of the trihalomethanes.
- Excellent inertness and thermally stable to 260°C.

Ordering Information | Rtx®-VGC (Fused Silica)

ID	df (µm)	temp. limits	30-Meter**	60-Meter**	75-Meter	105-Meter
0.25mm	1.40	-40 to 240/260°C	19415	19416		
0.45mm	2.55	-40 to 240/260°C	19408		19409	
0.53mm	3.00	-40 to 240/260°C	19485	19488	19474	19489
ID	df (µm)	temp. limits	20-Meter	40-Meter		
0.18mm	1.00	-40 to 240/260°C	49414	49415		

Rtx®-VRX

- Excellent selectivity for volatile compound analysis.
- Equivalent performance to DB-VRX column.
- Excellent for US EPA Method 8021 analyses.

Ordering Information | Rtx®-VRX (Fused Silica)

ID	df (µm)	temp. limits	30-Meter**	60-Meter**	75-Meter	105-Meter
0.25mm	1.40	-40 to 240/260°C	19315	19316		
0.45mm	2.55	-40 to 240/260°C	19308		19309	
0.53mm	3.00	-40 to 240/260°C	19385	19388	19374	19389
ID	df (µm)	temp. limits	20-Meter	40-Meter		
0.18mm	1.00	-40 to 240/260°C	49314	49315		

Rtx®-624

- Recommended for analyses of volatile organic compounds (VOCs) in EPA Methods.
- Crossbond® technology.
- 280°C thermal stability.
- Similar to DB-624 and HP-624 columns.

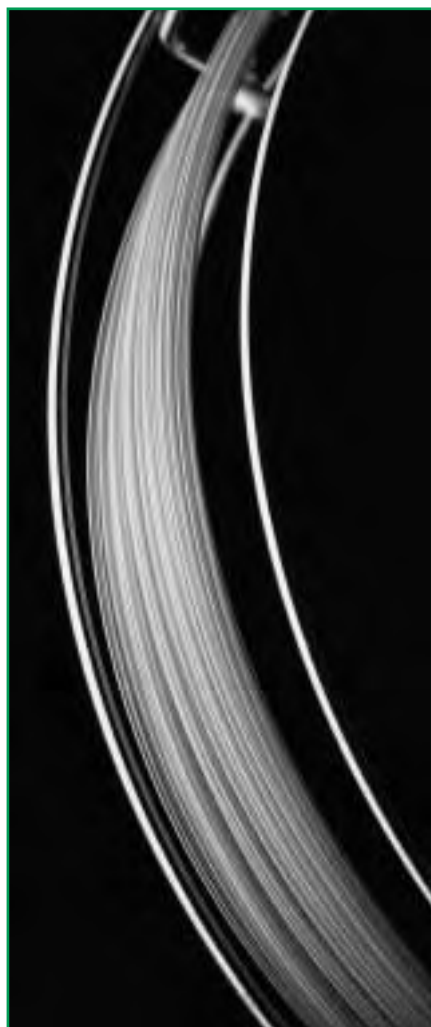
Ordering Information | Rtx®-624 (Fused Silica)

(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	temp. limits	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	1.40	-20 to 240°C	10968	10969		
0.45mm	2.55	-20 to 240°C			10982	
0.53mm	3.00	-20 to 240°C	10971	10973	10974	10975
ID	df (µm)	temp. limits	10-Meter	20-Meter	40-Meter	
0.18mm	1.00	-20 to 240°C		40924	40925	



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Rtx®-502.2

- Recommended for the analysis of volatile organic compounds (VOCs) in EPA Methods.
- Specified in many GRO methods for monitoring leaking underground storage tanks.
- Crossbond® technology. **Reduced bleed, Increased column lifetime, Solvent rinsability.**
- Similar to DB-502.2 columns.

Ordering Information | Rtx®-502.2 (Fused Silica)

(EPA Volatiles in Methods 502.2, 524.2)

ID	df (µm)	temp. limits	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	1.40	-20 to 250/270°C	10915	10916		
0.45mm	2.55	-20 to 250/270°C			10986	
0.53mm	3.00	-20 to 250/270°C	10908	10909		10910
ID	df (µm)	temp. limits	20-Meter	40-Meter		
0.18mm	1.00	-20 to 250/270°C	40914	40915		

Rtx®-Volatiles

- Analyze volatile organic compounds (VOCs) in EPA methods.
- Crossbond® technology.
- 280°C thermal stability.
- Similar to VOCOL® columns.

Ordering Information | Rtx®-Volatiles (Fused Silica)

(EPA Volatile Organic Methods)

ID	df (µm)	temp. limits*	30-Meter	60-Meter	105-Meter
0.25mm	1.00	-20 to 270/280°C	10900	10903	
0.53mm	2.00	-20 to 270/280°C	10902	10905	10906

*The maximum temperatures listed are for 15- and 30-meter lengths.

Longer lengths may have a slightly reduced maximum temperature.

Rtx®-1

- Ideal for analysis of solvents and petrochemicals.
- Available in unbreakable Silcosteel® (MXT®) tubing.
- Thermally stable to 350°C (MXT® stable to 400°C).
- Similar to DB-1, SPB-1, HP-1, and Ultra-1 phases.

Ordering Information | Rtx®-1 (Fused Silica)

(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	75-Meter	105-Meter
0.45mm	2.55	-60 to 270/290°C	10992	
0.53mm	3.00	-60 to 270/290°C		10189

The maximum temperatures listed are for 15- and 30-meter lengths.

Longer lengths may have a slightly reduced maximum temperature.

Intermediate-Polarity Deactivated Guard Columns & Transfer Lines

- Useful for a wide range of applications.
- Use with most common solvents.
- Maximum temperature: 325°C
- Reduce effects of dirty samples on column performance.
- Reduce downtime and maintenance.

Ordering Information | Fused Silica

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.
0.32mm	0.45 ± 0.04mm	10044	10044-600
0.53mm	0.69 ± 0.05mm	10045	10045-600

Ordering Information | MXT® (Silcosteel®)

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.	10-Meter
0.53mm	0.74 ± 0.025mm	70045	70045-600	70047

* Not tested with the Grob test mix because of a large pressure drop.

** 30- and 60-meter lengths are banded in 5-meter sections.

Siltek™-Deactivated Guard Columns/Transfer Lines

- Revolutionary deactivation process lowers endrin breakdown to less than 1%.
- Minimize bleed.
- Ideal for chlorinated pesticide analysis.
- Analyze tough samples quickly and accurately.
- Maximum temperature of 380°C.
- Reduces effects of dirty samples on column performance.
- Reduces downtime and maintenance.

Ordering Information | Siltek™-Deactivated Guard Columns

Nominal ID	Nominal OD	5-Meter
0.32mm	0.45 ± 0.04mm	10027
0.53mm	0.69 ± 0.05mm	10028

Low-Volume Injector for Agilent 5890 Septum Packed Purge Port

- Allows syringe injections onto the column for purge & trap troubleshooting or calibration.
- Silcosteel® treatment eliminates adsorption of active compounds.
- Attaches to GC inlet without cutting injection port lines.

Everything you need for installation is provided, including a 1/16-inch nut, a 1/16-inch ferrule, a base nut and 1/4-inch Vespel®/graphite ferrule, a 1/16-inch capillary nut, a 5-pack of low-bleed plug septa, and a special low-mass septum nut. Order appropriate capillary ferrules separately—see our catalog.

Description	qty.	cat.#
Low-Volume Injector for Agilent 5890 Septum Packed Purge Port	kit	21698

Low-Volume Injector for Agilent GCs

- Fits Agilent split/splitless injectors.
- Attaches to the GC inlet without cutting existing injection port lines.
- Allows injections from a syringe onto the column for purge & trap troubleshooting or calibration.
- Silcosteel® treatment eliminates adsorption of active compounds.

Our low-volume injector can be installed in a matter of minutes. Remove the septum nut or splitless weldment and insert the Restek low-volume injector through the split injector. Tighten the base nut and you're ready! Includes a 1/16-inch nut, a 1/16-inch ferrule, a base nut and 1/4-inch Vespel®/graphite ferrule, a 1/16-inch capillary nut, a 5-pack of low-bleed plug septa, and a low-mass septum nut. Order appropriate capillary ferrules separately—see our catalog.

Description	qty.	cat.#
Low-Volume Injector for Agilent Split/Splitless GC Inlets	kit	21692

Low-Volume Injector for Varian Split/Splitless Inlets

- Attaches to the GC inlet without cutting existing injection port lines.
- Allows injections from a syringe onto the column for purge & trap troubleshooting or calibration.
- Silcosteel® treatment eliminates adsorption of active compounds.
- Order capillary ferrules separately—see our catalog.

Description	qty.	cat.#
Low-Volume Injector for Varian Split/Splitless GC Inlets	kit	21693



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MXT®-Union Connector Kits—For Fused Silica Columns

- Low-dead-volume, leak-free connection.
- Reusable.
- Silcosteel® treatment ensures maximum inertness.
- Ideal for connecting a guard column or transfer line to an analytical column.
- Use to oven temperatures of 350°C.
- Available in union and “Y” configurations.

Each kit contains the MXT® union, two 1/32-inch nuts and two one-piece fused silica adaptors.

Description	qty.	cat.#
For 0.25mm ID Fused Silica Columns	kit	21386
For 0.32mm ID Fused Silica Columns	kit	21385
For 0.53mm ID Fused Silica Columns	kit	21384



Valco® Connectors—One-Piece Fused Silica Adaptor Ferrule

For connecting fused silica or 1/16- or 1/8-inch metal tubing.

- Use with Mxt®-Union Connectors
- Use to oven temperature of 350°C
- Made from graphite-reinforced polyimide

1/32-Inch Adaptor Ferrule

Tubing OD	Tubing ID	Valco® #	Valcon Polyimide	
			qty.	cat.#
0.25–0.4mm	0.25mm	FS.4-5	5-pk.	20137
0.4–0.5mm	0.32mm	FS.5-5	5-pk.	20140
0.5–0.8mm	0.53mm	ZF.5V-5	5-pk.	20141
1/32" Replacement Nut			5-pk.	20389



Heavy Duty Purge & Trap Syringe (Dynatech Precision Sampling)

- Heavy-duty glass barrel with metal front and rear flanges.
- Teflon® Luer-Lock® tip.
- Can fill and empty sparge tubes.
- Accepts standard Luer-Lock® needles.

Syringe	2mL cat.#	5mL cat.#	10mL cat.#
without Sample-Lok	21205	21206	21209
with Sample-Lok	—	21208	21207



Teflon® Tip, Gas-Tight Syringe Replacement Needles for Luer-Lock Syringes

Hub Material	Needle Gauge	Needle Length	Point Style	SGE cat.#	Restek qty.	Restek cat.#
metal	23	50mm	2	039802	5-pk.	24763
metal	22	2"/51mm	3	039895**	2-pk.	24765
metal	18	50mm	2	039842	5-pk.	24764

**For Rheodyne®/Valco® valves.

Leak Detective™ II Leak Detector

- Affordable thermal conductivity leak detector—every analyst should have one.
- Compact, ergonomic design is easy to hold and operate.
- Detects helium, hydrogen, and nitrogen at 1×10^{-4} cc/sec. or at an absolute concentration as low as 100ppm.*
- Fast results—responds in less than 2 seconds to trace leaks of gases with thermal conductivities different than air.
- Microchip design improves sensitivity and response time over previous models.
- Auto zeroing with the touch of a button.
- Battery-operated for increased portability (one 9-volt).

The compact, affordable tool that every analyst should have!

Description	qty.	cat.#
Leak Detective™ II Leak Detector	ea.	20413

**Never use liquid leak detectors on a capillary system because liquids can be drawn into the column.*

Caution: NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possible hazardous conditions.



Pre-Cleaned Volatile Organic Analyte Sampling Vials

- Container, liner and closure cleaned and assembled.
- Clear or amber.
- Open top caps.
- Teflon® faced 0.125" silicone septa.
- Each case lot numbered.

Description	qty.	cat.#
20mL Clear Pre-Cleaned VOA Vials	72-pk.	21798
20mL Amber Pre-Cleaned VOA Vials	72-pk.	21799
40mL Clear Pre-Cleaned VOA Vials	72-pk.	21796
40mL Amber Pre-Cleaned VOA Vials	72-pk.	21797




1mm ID Liners

for Agilent/Finnigan GCs	ID**/OD & Length (mm)	cat.# ea.	cat.# 5-pk.
	1.0 ID 6.3 OD x 78.5	20972	20973
1mm Split			

for Varian GCs	ID**/OD & Length (mm)	cat.# ea.	cat.# 5-pk.
	1.0 ID 6.3 OD x 72	20970	20971
1mm Split			

for Shimadzu GCs	ID**/OD & Length (mm)	cat.# ea.	cat.# 5-pk.	cat.# 25-pk.
	1.0 ID 5.0 OD x 95	20976	20977	20978
17A 1mm Split				

PSS Liners for PerkinElmer GCs	ID*/OD & Length (mm)	Similar to PE part #	cat.# ea.	cat.# 5-pk.	cat.# 25-pk.
	1.0 ID 4.0 OD x 86.2	N612-1006	20738	20741	—
PSS Split/Splitless (1mm ID)					

for Thermo Finnigan 8000 & TRACE Series GCs	ID**/OD & Length (mm)	Similar to TF Part #	cat.# ea.	cat.# 5-pk.
	1.0 ID 8.0 OD x 105	453 20075	20916	20917
1mm Split				

**all liners are
100%
deactivated**

We deactivate Restek liners using our unique polymeric deactivation process to ensure accurate chromatographic data. We evaluate them with an endrin breakdown test for complete inertness, and each liner is dimensionally checked for a perfect fit. Siltek™ and base deactivation are available for specialized analyses

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Method 8260B

8260A/B Internal Standard Mix

chlorobenzene-d5 fluorobenzene
1,4-dichlorobenzene-d4
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30241	30241-510	—
w/data pack		
30241-500	30241-520	30341

8260A/B Surrogate Mix

4-bromofluorobenzene 1,2-dichloroethane-d4
dibromofluoromethane toluene-d8
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30240	30240-510	—
w/data pack		
30240-500	30240-520	30340

4-Bromofluorobenzene

4-bromofluorobenzene
2,500µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30067	30067-510	—
w/data pack		
30067-500	30067-520	30167

10,000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30082	30082-510	—
w/data pack		
30082-500	30082-520	30182

8260B Matrix Spike Mix

benzene toluene
chlorobenzene trichloroethylene
1,1-dichloroethene
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30479	30479-510	—
w/data pack		
30479-500	30479-520	30579

PFTBA (MS Tuning Compound)

perfluorotributylamine (PFTBA)
1mL/ampul, neat

Each
30482

VOA Calibration Mix #1 (ketones)

acetone 2-hexanone
2-butanone 4-methyl-2-pentanone
5,000µg/mL each in P&T methanol:water (90:10),
1mL/ampul

Each	5-pk.	10-pk.
30006	30006-510	—
w/data pack		
30006-500	30006-520	30106

8260B Calibration Mix #1

(76 + 1 components)

Note: This product is provided as a two ampul set:
acetonitrile *trans*-1,3-dichloropropene
acrylonitrile diethyl ether
allyl chloride 1,4-dioxane
benzene ethylbenzene
bromobenzene ethyl methacrylate
bromochloromethane hexachlorobutadiene
bromodichloromethane iodomethane
bromoform isobutyl alcohol
n-butylbenzene isopropylbenzene
sec-butylbenzene *p*-isopropyltoluene
tert-butylbenzene methacrylonitrile
carbon disulfide methyl acrylate
carbon tetrachloride methyl methacrylate
chlorobenzene methylene chloride
2-chloroethanol naphthalene
chloroform nitrobenzene
chloroprene 2-nitropropane
2-chlorotoluene pentachloroethane
4-chlorotoluene propionitrile
dibromochloromethane *n*-propylbenzene
1,2-dibromo-3-chloro- styrene
propane 1,1,1,2-tetrachloroethane
1,2-dibromoethane 1,1,2,2-tetrachloroethane
dibromomethane tetrachloroethene
1,2-dichlorobenzene tetrahydrofuran
1,3-dichlorobenzene toluene
1,4-dichlorobenzene 1,2,3-trichlorobenzene
cis-1,4-dichloro-2-butene 1,2,4-trichlorobenzene
trans-1,4-dichloro-2-butene 1,1,1-trichloroethane
1,1-dichloroethane 1,1,2-trichloroethane
1,2-dichloroethane trichloroethene
1,1-dichloroethene 1,2,3-trichloropropane
cis-1,2-dichloroethene 1,1,2-trichlorotrifluoroethane
trans-1,2-dichloroethene (Freon® 113)
1,2-dichloropropane 1,2,4-trimethylbenzene
1,3-dichloropropane 1,3,5-trimethylbenzene
2,2-dichloropropane *m*-xylene
1,1-dichloropropene *o*-xylene
cis-1,3-dichloropropene *p*-xylene

2,000µg/mL each in P&T methanol, 1mL/ampul

2-chloroethyl vinyl ether

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30475	30475-510	—
w/data pack		
30475-500	30475-520	30575

1,2-Dichlorotetrafluoroethane

1,2-dichlorotetrafluoroethane (Freon® 114)

2,000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30476	30476-510	—
w/data pack		
30476-500	30476-520	30576

California Oxygenates Mix

diisopropyl ether 2,000µg/mL *tert*-butyl alcohol 10,000
ethyl-*tert*-butyl ether 2,000 methyl *tert*-butyl ether 2,000
tert-amyl methyl ether 2,000

In P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30465	30465-510	—
w/data pack		
30465-500	30465-520	30565

Ethanol Mix

ethanol
10,000µg/mL in DI water; 1mL/ampul

Each	5-pk.	10-pk.
30466	30466-510	—
w/data pack		
30466-500	30466-520	30566

Acrolein Mix

acrolein
10,000µg/mL in water, 1mL/ampul

Each	5-pk.	10-pk.
30478	30478-510	—
w/data pack		
30478-500	30478-520	30578

8260B Acetate Mix

vinyl acetate *n*-propyl acetate
ethyl acetate *n*-butyl acetate
isopropyl acetate

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30477	30477-510	—
w/data pack		
30477-500	30477-520	30577

8260B Acetate Mix (Revised)

n-amyl acetate methyl acetate
butyl acetate propyl acetate
ethyl acetate vinyl acetate
isopropyl acetate

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30489	30489-510	—
w/data pack		
30489-500	30489-520	30589

Method 8260A

8260A Volatile Organics Kit

30005: VOA Matrix Spike Mix 30067: 4-bromofluorobenzene
30042: 502.2 Calib. Mix #1 30240: 8260A Surrogate Mix
30043: 502.2 Calib. Mix #2 30241: 8260A Internal
30044: 502.2 Calib. Mix #3 Standard Mix
30045: 502.2 Calib. Mix #4 30075: 8240/8260 System
30046: 502.2 Calib. Mix #5 Performance Check Mix
30047: 502.2 Calib. Mix #6

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30242	30242-500

Method 8260

8260 Internal Standard Mix

chlorobenzene-d5 1,4-difluorobenzene
1,4-dichlorobenzene-d4 pentafluorobenzene
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30074	30074-510	—
w/data pack		
30074-500	30074-520	30174

8260 Surrogate Mix

4-bromofluorobenzene toluene-d8
dibromofluoromethane
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30073	30073-510	—
w/data pack		
30073-500	30073-520	30173

8240/8260 System Performance Check Mix

bromoform 1,1-dichloroethane
chlorobenzene 1,1,2,2-tetrachloroethane
chloromethane

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30075	30075-510	—
w/data pack		
30075-500	30075-520	30175

8240/8260 Calibration Check Mix

chloroform ethylbenzene
1,1-dichloroethene toluene
1,2-dichloropropane vinyl chloride

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30427	30427-510	—
w/data pack		
30427-500	30427-520	30527

8260 Volatile Organics Kit

30005: VOA Matrix Spike Mix 30067: 4-bromofluorobenzene
30042: 502.2 Calib. Mix #1 30073: 8260 Surrogate Mix
30043: 502.2 Calib. Mix #2 30074: 8260 Internal
30044: 502.2 Calib. Mix #3 Standard Mix
30045: 502.2 Calib. Mix #4 30075: 8240/8260 System
30046: 502.2 Calib. Mix #5 Performance Check Mix
30047: 502.2 Calib. Mix #6

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30076	30076-500

PVOC, GRO, & BTEX

PVOC Mix (California)

benzene *m*-xylene
ethylbenzene *o*-xylene
methyl *tert*-butyl ether *p*-xylene
toluene

1,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30231	30231-510	—
w/data pack		
30231-500	30231-520	30331

PVOC/GRO Mix (Wisconsin)

benzene 1,2,4-trimethylbenzene
ethylbenzene 1,3,5-trimethylbenzene
methyl *tert*-butyl ether *m*-xylene
naphthalene *o*-xylene
toluene *p*-xylene

1,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30095	30095-510	—
w/data pack		
30095-500	30095-520	30195

GRO Mix

benzene 2,2,4-trimethylpentane
ethylbenzene (isooctane)
3-methylpentane toluene
naphthalene *m*-xylene
1,2,4-trimethylbenzene *o*-xylene

1,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30069	30069-510	—
w/data pack		
30069-500	30069-520	30169

Gasoline Component Standard

Component	Conc., (µg/mL)	Component	Conc., (µg/mL)
benzene	500	1,2,4-trimethylbenzene	1000
ethylbenzene	500	2,2,4-trimethylpentane	1500
heptane	500	<i>m</i> -xylene	1000
2-methylpentane	1500	<i>o</i> -xylene	1000
toluene	1500	<i>p</i> -xylene	1000

10,000µg/mL total in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30486	30486-510	—
w/data pack		
30486-500	30486-520	30586

GRO Mix (EPA)

benzene	500µg/mL	2,2,4-trimethylpentane	1,500
ethylbenzene	500	toluene	1,500
heptane	500	<i>m</i> -xylene	1,000
2-methylpentane	1,500	<i>o</i> -xylene	1,000
1,2,4-trimethylbenzene	1,000		

In P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30065	30065-510	—
w/data pack		
30065-500	30065-520	30165

BTEX Standard

benzene *m*-xylene
ethylbenzene *o*-xylene
toluene *p*-xylene

200µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30051	30051-510	—
w/data pack		
30051-500	30051-520	30151

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30213	30213-510	—
w/data pack		
30213-500	30213-520	30313

2,000µg/mL each in P&T methanol, except *m*-xylene and *p*-xylene at 1,000µg/mL, 1mL/ampul

Each	5-pk.	10-pk.
30488	30488-510	—
w/data pack		
30488-500	30488-520	30588

Certified BTEX in Unleaded Gas Composite Standard

Certified for:

benzene	toluene
ethylbenzene	<i>m</i> -xylene
isopropyl benzene	<i>o</i> -xylene
methyl <i>tert</i> -butyl ether	<i>p</i> -xylene
naphthalene	

5,500ppm gasoline in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30237	30237-510	—
w/data pack		
30237-500	30237-520	30337

BTEX Gas Mix

benzene *m*-xylene
ethylbenzene *o*-xylene
toluene *p*-xylene

In nitrogen, 104 liters @ 1800psig

1ppm	100ppb
34414 (ea.)	34428 (ea.)

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Method 624

624 Internal Standard Mix

bromochloromethane 1,4-dichlorobutane
2-bromo-1-chloropropane
1,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30023	30023-510	—
w/data pack		
30023-500	30023-520	30123

624 Surrogate Standard Mix

4-bromofluorobenzene pentafluorobenzene
fluorobenzene
2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30243	30243-510	—
w/data pack		
30243-500	30243-520	30343

Surrogate Standard

1,4-bromofluorobenzene α,α,α-trifluorotoluene
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30484	30484-510	—
w/data pack		
30484-500	30484-520	30584

624 Calibration Mix #1

bromomethane trichlorofluoromethane
chloroethane vinyl chloride
chloromethane
2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30020	30020-510	—
w/data pack		
30020-500	30020-520	30120

624 Calibration Mix #2

benzene 1,1-dichloroethene
carbon tetrachloride 1,2-dichloropropane
chlorobenzene methylene chloride
2-chloroethyl vinyl ether tetrachloroethene
dibromochloromethane 1,1,2-trichloroethane
1,1-dichloroethane trichloroethene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30021	30021-510	—
w/data pack		
30021-500	30021-520	30121

624 Calibration Mix #3

bromodichloromethane trans-1,2-dichloroethene
bromoform cis-1,3-dichloropropene
chloroform trans-1,3-dichloropropene
1,2-dichlorobenzene ethylbenzene
1,3-dichlorobenzene 1,1,2,2-tetrachloroethane
1,4-dichlorobenzene toluene
1,2-dichloroethane 1,1,1-trichloroethane

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30022	30022-510	—
w/data pack		
30022-500	30022-520	30122

624 Kit

30020: 624 Calib. Mix #1 30022: 624 Calib. Mix #3
30021: 624 Calib. Mix #2 30023: 624 Int. Standard Mix
Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30055	30055-500

624 Complete Kit

30020: 624 Calib. Mix #1 30023: 624 Int. Standard Mix
30021: 624 Calib. Mix #2 30024: 624 Surrogate
30022: 624 Calib. Mix #3 Standard Mix

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30244	30244-500

Method 601 & 602

601/602 Purgeable Halocarbons Kit

30042: 502.2 Calib. Mix #1 30022: 624 Calib. Mix #3
30021: 624 Calib. Mix #2

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30061	30061-500

602 Purgeable Aromatics Calibration Mix

benzene 1,4-dichlorobenzene
chlorobenzene ethylbenzene
1,2-dichlorobenzene toluene
1,3-dichlorobenzene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30035	30035-510	—
w/data pack		
30035-500	30035-520	30135

Method 502.2

502.2 CAL2000 MegaMix™ Mixture

benzene 2,2-dichloropropane
bromobenzene 1,1-dichloropropene
bromochloromethane cis-1,3-dichloropropene
bromodichloromethane trans-1,3-dichloropropene
bromoform ethylbenzene
n-butylbenzene hexachlorobutadiene
sec-butylbenzene isopropylbenzene
tert-butylbenzene p-isopropyltoluene
carbon tetrachloride methylene chloride
chlorobenzene naphthalene
chloroform n-propylbenzene
2-chlorotoluene styrene
4-chlorotoluene 1,1,1,2-tetrachloroethane
dibromochloromethane 1,1,2,2-tetrachloroethane
1,2-dibromo-3-chloropropane tetrachloroethene
1,2-dibromoethane toluene
dibromomethane 1,2,3-trichlorobenzene
1,2-dichlorobenzene 1,2,4-trichlorobenzene
1,3-dichlorobenzene 1,1,1-trichloroethane
1,4-dichlorobenzene 1,1,2-trichloroethane
1,1-dichloroethane trichloroethene
1,2-dichloroethane 1,2,3-trichloropropane
1,1-dichloroethene 1,2,4-trimethylbenzene
cis-1,2-dichloroethene 1,3,5-trimethylbenzene
trans-1,2-dichloroethene m-xylene
1,2-dichloropropane o-xylene
1,3-dichloropropane p-xylene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30431	30431-510	—
w/data pack		
30431-500	30431-520	30531

200µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30432	30432-510	—
w/data pack		
30432-500	30432-520	30532

1-Chloro-2-fluorobenzene

1-chloro-2-fluorobenzene
2,000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30040	30040-510	—
w/data pack		
30040-500	30040-520	30140

502.2 Internal Standard Mix #2

2-bromo-1-chloropropane fluorobenzene
2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30041	30041-510	—
w/data pack		
30041-500	30041-520	30141

8021/502.2 Surrogate Mix #1

1-bromo-2-chloroethane fluorobenzene
1-chloro-3-fluorobenzene
2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30463	30463-510	—
w/data pack		
30463-500	30463-520	30563

8021/502.2 Surrogate Mix #2

1-bromo-2-chloroethane 1-chloro-3-fluorobenzene
4-bromochlorobenzene fluorobenzene
2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30464	30464-510	—
w/data pack		
30464-500	30464-520	30564

502.2 VOA Calib. Kit #2 (2000ppm)

30432: 502.2 Calib. Mix #1
30431: 502.2 CAL2000 MegaMix™
Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30445	30445-500

502.2 VOA Calib. Kit #3 (200ppm)

30439: 502.2 Calib. Mix #1A
30432: 502.2 CAL200 MegaMix™
Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30446	30446-500

502.2 Calibration Mix #1 (gases)

bromomethane dichlorodifluoromethane
chloroethane trichlorofluoromethane
chloromethane vinyl chloride
2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30042	30042-510	—
w/data pack		
30042-500	30042-520	30142

502.2 Calibration Mix #1A

bromomethane dichlorodifluoromethane
chloroethane trichlorofluoromethane
chloromethane vinyl chloride
200µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30439	30439-510	—
w/data pack		
30439-500	30439-520	30539

04.2, 04.1, & 3/90 SOW

CLP 04.1 VOA Internal Standard/SMC Spike Mix

bromochloromethane 1,2-dichloroethane-d4
4-bromofluorobenzene 1,4-difluorobenzene
chlorobenzene-d5 toluene-d8
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30457	30457-510	—
w/data pack		
30457-500	30457-520	30557

VOA Internal Standard Mix

bromochloromethane chlorobenzene-d5
1,4-difluorobenzene
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30011	30011-510	—
w/data pack		
30011-500	30011-520	30111

VOA Surrogate Spike Mix

4-bromofluorobenzene toluene-d8
1,2-dichloroethane-d4
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30004	30004-510	—
w/data pack		
30004-500	30004-520	30104

VOA Matrix Spike Mix

benzene toluene
chlorobenzene trichloroethene
1,1-dichloroethene
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30005	30005-510	—
w/data pack		
30005-500	30005-520	30105

VOA Screening Mix #1

benzene o-xylene
ethylbenzene p-xylene
toluene
1,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30001	30001-510	—
w/data pack		
30001-500	30001-520	30101

VOA Screening Mix #2

n-dodecane n-nonane
1,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30002	30002-510	—
w/data pack		
30002-500	30002-520	30102

VOA Tuning Compound

4-bromofluorobenzene
5,000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30003	30003-510	—
w/data pack		
30003-500	30003-520	30103

PFTBA (MS Tuning Compound)

perfluorotributylamine (PFTBA)
1mL/ampul, neat

Each
30482

DID U KNOW?

Restek reference materials
include a silanized vial for
sample transfer.

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3/90 SOW

CLP VOA CAL2000

MegaMix™ Mixture (29 components)

Note: This product is provided as a two-ampul set:

benzene	<i>cis</i> -1,3-dichloropropene
bromodichloromethane	<i>trans</i> -1,3-dichloropropene
bromoform	ethylbenzene
carbon disulfide	methylene chloride
carbon tetrachloride	styrene
chlorobenzene	1,1,2,2-tetrachloroethane
chloroform	tetrachloroethene
dibromochloromethane	toluene
1,1-dichloroethane	1,1,1-trichloroethane
1,2-dichloroethane	1,1,2-trichloroethane
1,1-dichloroethene	trichloroethene
<i>cis</i> -1,2-dichloroethene	<i>m</i> -xylene
<i>trans</i> -1,2-dichloroethene	<i>o</i> -xylene
1,2-dichloropropane	<i>p</i> -xylene

2,000µg/mL each in P&T methanol, 1mL/ampul

vinyl acetate

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30438	30438-510	—
w/data pack		
30438-500	30438-520	30538

CLP VOA Calibration Kit #2

30006: VOA Calibration Mix #1 (ketones)

30010: VOA Calibration Mix #5 (gases)

30438: CLP VOA CAL2000 MegaMix™

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30442	30442-500

VOA Calibration Mix #5 (gases)

bromomethane chloromethane
chloroethane vinyl chloride

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30010	30010-510	—
w/data pack		
30010-500	30010-520	30110

VOA Calibration Mix #2

benzene vinyl acetate
carbon disulfide *o*-xylene
ethylbenzene *p*-xylene
toluene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30007	30007-510	—
w/data pack		
30007-500	30007-520	30107

VOA Calibration Mix #3

carbon tetrachloride 1,2-dichloropropane
chlorobenzene methylene chloride
chloroform 1,1,2-trichloroethane
1,1-dichloroethane trichloroethene
1,1-dichloroethene *m*-xylene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30008	30008-510	—
w/data pack		
30008-500	30008-520	30108

VOA Calibration Mix #4

bromodichloromethane *cis*-1,3-dichloropropene
bromoform *trans*-1,3-dichloropropene
dibromochloromethane styrene
1,2-dichloroethane 1,1,2,2-tetrachloroethane
cis-1,2-dichloroethene tetrachloroethene
trans-1,2-dichloroethene 1,1,1-trichloroethane

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30009	30009-510	—
w/data pack		
30009-500	30009-520	30109

CLP Volatile Organics Kit #2

30001: VOA Screening Mix #1 30010: VOA Calib. Mix #5
30002: VOA Screening Mix #2 (gases)
30003: VOA Tuning Comp. 30011: VOA Int'l Standard Mix
30004: VOA Surr. Spike Mix 30438: CLP VOA CAL2000
30005: VOA Matrix Spike Mix MegaMix™
30006: VOA Calib. Mix #1
(ketones)

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30440	30440-500

Volatile Organics Kit

30001: VOA Screening Mix #1 30007: VOA Calib. Mix #2
30002: VOA Screening Mix #2 30008: VOA Calib. Mix #3
30003: VOA Tuning Comp. 30009: VOA Calib. Mix #4
30004: VOA Surr. Spike Mix 30010: VOA Calib. Mix #5
30005: VOA Matrix Spike Mix (gases)
30006: VOA Calib. Mix #1 30011: VOA Internal
(ketones) Standard Mix

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30050	30150

04.2 & 04.1 SOW

CLP 04.1 VOA CAL2000

MegaMix™ Mixture (40 components)

benzene	<i>trans</i> -1,3-dichloropropene
bromodichloromethane	ethylbenzene
bromoform	isopropylbenzene
carbon disulfide	methyl acetate
carbon tetrachloride	methylcyclohexane
chlorobenzene	methylene chloride
chloroform	styrene
1,2-dibromo-3-chloropropane	methyl <i>tert</i> -butyl ether (MTBE)
cyclohexane	1,1,2,2-tetrachloroethane
dibromochloromethane	tetrachloroethene
1,2-dibromoethane	toluene
1,2-dichlorobenzene	1,2,4-trichlorobenzene
1,3-dichlorobenzene	1,1,1-trichloroethane
1,4-dichlorobenzene	1,1,2-trichloroethane
1,1-dichloroethane	trichloroethylene
1,2-dichloroethane	1,1,2-trichlorotrifluoroethane
1,1-dichloroethene	(Freon® 113)
<i>cis</i> -1,2-dichloroethene	<i>m</i> -xylene
<i>trans</i> -1,2-dichloroethene	<i>o</i> -xylene
1,2-dichloropropane	<i>p</i> -xylene
<i>cis</i> -1,3-dichloropropene	

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30456	30456-510	—
w/data pack		
30456-500	30456-520	30556

502.2 Calibration Mix #1 (gases)

bromomethane	dichlorodifluoromethane
chloroethane	trichlorofluoromethane
chloromethane	vinyl chloride

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30042	30042-510	—
w/data pack		
30042-500	30042-520	30142

502.2 Calibration Mix #1A

bromomethane	dichlorodifluoromethane
chloroethane	trichlorofluoromethane
chloromethane	vinyl chloride

200µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30439	30439-510	—
w/data pack		
30439-500	30439-520	30539

CLP 04.1 VOA Kit #3

30006: VOA Calibration Mix #1 (ketones)
30042: 502.2 Calibration Mix #1 (gases)
30456: CLP 04.1 VOA CAL2000 MegaMix™

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30460	30460-500

OLC 03.2 VOA MegaMix™ Mixture

(42 components)

benzene	<i>trans</i> -1,3-dichloropropene (E)
bromochloromethane	ethylbenzene
bromodichloromethane	isopropylbenzene (cumene)
bromoform	methyl acetate
carbon disulfide	methylcyclohexane
carbon tetrachloride	methyl <i>tert</i> -butyl ether (MTBE)
chlorobenzene	methylene chloride
chloroform	(dichloromethane)
cyclohexane	styrene
dibromochloromethane	1,1,2,2-tetrachloroethane
(chlorodibromomethane)	tetrachloroethylene
1,2-dibromo-3-chloropropane	toluene
1,2-dibromoethane (EDB)	1,2,3-trichlorobenzene
1,2-dichlorobenzene	1,2,4-trichlorobenzene
1,3-dichlorobenzene	1,1,1-trichloroethane
1,4-dichlorobenzene	1,1,2-trichloroethane
1,1-dichloroethane	trichloroethylene
1,2-dichloroethane	1,1,2-trichlorotrifluoroethane
1,1-dichloroethylene	(Freon® 113)
<i>cis</i> -1,2-dichloroethylene	<i>m</i> -xylene
<i>trans</i> -1,2-dichloroethylene	<i>o</i> -xylene
1,2-dichloropropane	<i>p</i> -xylene
<i>cis</i> -1,3-dichloropropene(Z)	

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30492	30492-510	—
w/data pack		
30492-500	30492-520	30592

OLC 03.2 VOA Deuterated Monitoring Compounds (DMC)

(14 components)

Note: This product is provided as a two-ampule set:

Non-Ketones:

benzene-d6	1,1-dichloroethene-d2
bromoform-d	1,2-dichloropropane-d6
chloroethane-d5	<i>trans</i> -1,3-dichloropropene-d4
chloroform-d	1,1,2,2-tetrachloroethane-d2
1,2-dichlorobenzene-d4	toluene-d8
1,2-dichloroethane-d4	vinyl chloride-d3

100µg/mL each in P&T methanol, 1mL/ampul

Ketones:

2-butanone-d5	2-hexanone-d5
---------------	---------------

200µg/mL each in P&T methanol, 0.5mL/ampul

Each	5-pk.
30493	30493-510

EPA Contract Lab Program (CLP)

Statement of Work (SOW) OLC03.2 is an analytical method for the detection of low concentration volatile, semivolatile, and pesticide/Arclor® organics in aqueous samples. For VOA and SVOA GC/MS analysis OLC03.2 introduces Deuterated Monitoring Compounds (DMCs) as a sample-by-sample accuracy indicator.

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TECHNICAL GUIDE

A Guide to Passive Air Sampling

Equipment Needed and Practical Techniques
for Collecting Air Samples



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dependable execution

One of our standing goals is to provide you with practical information to help you obtain reliable data from your chromatographic and peripheral systems. This guide describes equipment needed and practical techniques to follow when collecting passive air samples, using canisters. It is a compilation of information based on our experience and that of experts in this field. We would like to thank the individuals who provided invaluable assistance in the development of this guide: Dr. Eric Winegar of Applied Measurements Science, Joachim Pleil of the US Environmental Protection Agency, John Yokoyama of Performance Analytical, and Jerry Winberry of Envirotech Solutions.

If you have any questions, or have input for future editions of this guide, please feel free to contact us at Restek Corporation.



David M. Shelow

Air Monitoring Products

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Freon, Tedlar, Teflon, Viton (E.I. du Pont de Nemours & Co., Inc.), SUMMA (Molextrics), Veriflo (Veriflo Corp., div. of Parker Hannifin Corp.)

I. Introduction

Ambient air sampling involves collecting a representative sample of ambient air for analysis. If the environment is not changing, or if only a qualitative sample is needed, a simple “grab” sample can be obtained. For example, an evacuated sample canister can be opened and sample rapidly collected at a non-controlled rate, usually over several seconds, until the container attains equilibrium with atmospheric pressure. Generally this qualitative approach is used when unknown analytes must be identified, when the air contains high concentrations of analytes at certain (short) times, or when an odor is noticed and a sample must be obtained quickly. Paired grab samples (before/after or smell/no smell) often are employed to qualitatively diagnose a perceived problem.

To obtain a more representative sample requires time-integrated sampling. A flow restrictor is used to spread the sample collection flow over a specific time period, to ensure an “average” composited or time-weighted average (TWA) sample. A TWA sample will accurately reflect the mean conditions of the ambient air in the environment and is preferred when, for regulatory or health reasons, a typical exposure concentration is required for a situation that may have high variability, as in an occupational setting.

There are two general approaches to collecting air samples: 1) “whole air” sampling with canisters or Tedlar® bags and 2) “in-field concentration” sampling using sorbent tubes or cold traps. In this guide we focus on collecting whole air samples in canisters. Within this approach, two sampling techniques commonly are used: passive sampling and non-passive (active) sampling, distinguished by the absence or use of an active pumping device, respectively.

In passive sampling an air sample is pulled through a flow controller into an evacuated canister over a chosen period of time, ranging from 5 minutes to 24 hours. The sampling period and the flow rate determine the canister volume required. In active sampling, a pump is used to push the sample through a mass flow controller and into the canister. Additional sample can be collected, relative to the amount that can be collected by passive sampling, by pressurizing the canister with sample. Commonly the sample is pressurized to 15psig, effectively doubling the sample volume. Sampling can be time-integrated (e.g., an 8-hour sample), or a dip tube design can be used to establish a flow through the system and flush the sample container with sample, then, after a specified time, the exit valve is closed and the container is pressurized with sample.

Although active sampling is very flexible, a drawback to using a pump is the need for additional quality assurance requirements for sample integrity (i.e., no artifacts or loss of analytes). Additionally, a pump requires a battery or line power source, which may pose logistic difficulty in remote field-site sampling.

II. Equipment Used for Passive Air Sampling

To ensure a valid sample when using a passive sampling technique, it is important that the flow rate not change greatly during the time interval specified for the integrated sample. The proper sampling equipment helps accomplish this objective. A typical passive sampling train should include the following components, all constructed of stainless steel: a sampling inlet, a sintered metal particle filter, a critical orifice, a flow controlling device, a vacuum gauge, and a canister (Figure 1).

Sampling Inlet

The sampling inlet - the entrance to the sampling train - typically is cleaned stainless steel tubing, either 1/4" ID or 1/8" ID. US EPA Compendium Method TO-14/15 recommends sampling at a height of 2 meters above the ground. In a highly trafficked area, this would minimize the problem of dust particles entering the sampling train. This height is not mandatory, however, and it is common practice to use an inlet that is 12" (approximately 1/3 meter) high. The EPA also recommends having the entrance of the sampling inlet face



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wv

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downward, to prevent raindrops from entering the inlet. In some sampling trains a 1/8" or 1/4" nut at the entrance of the inlet keeps water droplets away from the edge of the inlet, where they could be drawn into the sampling train with the sample.

Particle Filter

Installed in the sampling train prior to the flow-controlling device, the particle filter prevents airborne particles from entering the sample flow path. Particles could partially obstruct the flow path and alter the flow rate during sampling. In extreme cases, particles could plug the flow path and stop the sample flow. The smallest orifice commonly used in a passive sampling train is 0.0012" (approximately 30 micrometers). Without a particle filter, dust particles could occlude this opening as they accumulate in the orifice fitting. Particles also can affect the leak integrity of the valve, and possibly can damage the valve. Two types of filters are used for this application, frit filters and in-line filters (Figure 2). A variety of models of each type are available; most are of sintered stainless steel and have 2-, 5-, or 7-micron pores. Obviously, the smaller the pores, the less likely are potential problems from airborne particles. EPA Compendium Method TO-14A/15 recommends using a particle filter with 2-micron pores.

Critical Orifice

The critical orifice (Figure 3, page 4) restricts the flow to a specified range. In conjunction with the flow controller, this allows the canister to fill at a specified rate over a specified time period. The most common critical orifice design is a series of interchangeable stainless steel 1/4" NPT to 1/4" compression unions, each fitted with a precisely bored sapphire orifice. Each orifice provides a specific flow range (Table 1). Stability over a wide range of temperatures makes sapphire the construction material of choice. Typically during field sampling, the sampling train is subjected to temperature fluctuations that would cause metals to contract or expand, affecting the diameter of the aperture and thereby affecting flow. Sapphire will not expand or contract across any ambient temperature extremes incurred during sampling.

A critical orifice can be used as the sole flow-restricting device, but it cannot ensure uniform flow. The source pressure of the flow changes during sampling, and the flow rate through the orifice also would change, producing an invalid time-integrated sample. It is important that a highly consistent flow rate be maintained during passive sampling. This is accomplished by the flow controller that incorporates the critical orifice.

Flow Controller

The flow controller (Figure 3, page 4) maintains a constant sample flow over the integrated time period, despite changes in the vacuum in the canister or in the environmental temperature (Figure 4, page 5). In the Veriflo™ Model SC423 XL Flow Controller shown in Figure 3, the critical orifice acts as a flow restrictor, upstream of a constant back pressure. This constant back pressure is established by the balance between the mechanical spring rate of the diaphragm and the pressure differential across the diaphragm. The latter is established by the pressure difference between the atmospheric pressure and the vacuum in the canister and the flow through the critical orifice. The net result is a constant flow.

Table 1 Critical orifice diameter vs flow rate.

Orifice Diameter (in.)	Flow Rate Range (sccm)	Canister Volume / Sampling Time			
		1L	3L	6L	15L
0.0008	0.5-2	24 hr.	48 hr.	125 hr.	--
0.0012	2-4	4 hr.	12 hr.	24 hr.	60 hr.
0.0016	4-8	2 hr.	6 hr.	12 hr.	30 hr.
0.0020	8-20	1 hr.	4 hr.	8 hr.	20 hr.
0.0030	20-40	--	2 hr.	3 hr.	8 hr.
0.0060	40-80	--	--	1 hr.	3 hr.

Figure 1 A complete sampling train is needed for reliable passive sampling.

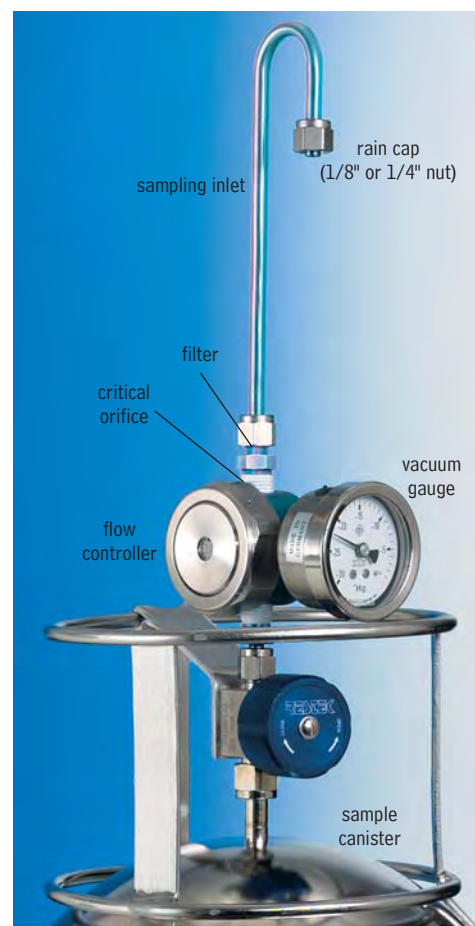


Figure 2 Filters used in sampling trains.



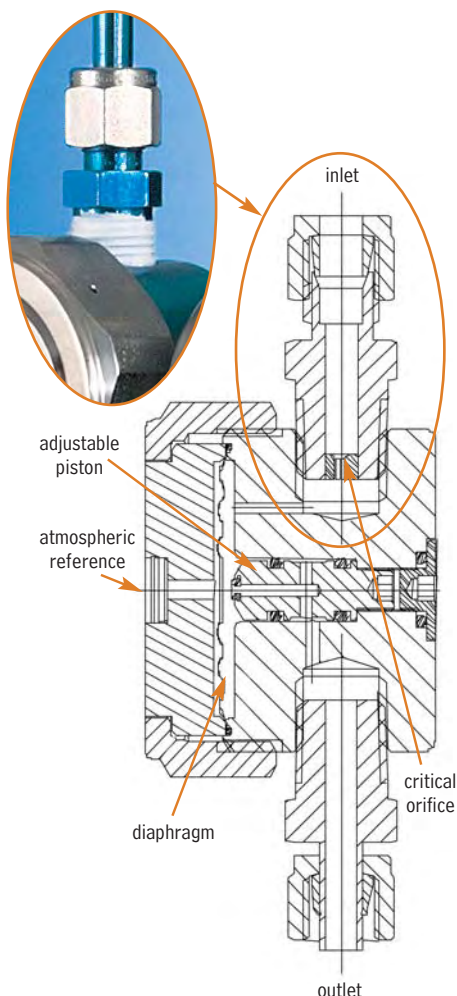
Figure 3 Flow controller & critical orifice.

Figure 3 courtesy of Veriflo Corp.,
a division of Parker Hannifin Corp.

The critical orifice determines the flow range. The adjustable piston is used to set a specific, fixed flow rate within the flow range. An adjustment to the position of the piston changes the back pressure, which changes the pressure differential across the critical orifice. If the piston is lowered away from the diaphragm, the flow rate will increase. If the piston is raised toward the diaphragm, the flow rate will decrease. This flow controller will accurately maintain a constant flow despite changes in vacuum over a range of 30" Hg to 7" Hg. Flow is constant until the vacuum range of the device is exceeded, making the flow controller unable to maintain the constant pressure differential. In Figure 5, for example, the flow rate is constant from 29.9" Hg to approximately 7" Hg, at which point the flow rate decreases because the flow controller is unable to maintain the proper pressure differential. This control will allow the user to collect approximately 5 liters of sample in a 6-liter canister. This is an extremely important factor in obtaining valid time-integrated samples through passive sampling. We will discuss this point further in the Field Sampling section of this guide.

Vacuum Gauge

A vacuum gauge enables sampling personnel to visually monitor changes in the vacuum in the canister during sampling. If the flow rate changes unexpectedly (e.g., due to a leak or an incorrect setting), the vacuum gauge will indicate a disproportionately high or low vacuum in the canister, and corrective action can be taken (i.e., flow adjusted) in time to ensure a valid sample. This type of vacuum gauge is attached to the sampling train, for use in the field. The vacuum gauge should be of high quality, to ensure that it does not introduce contaminants into the sample. All wetted parts in the vacuum gauge shown in Figure 6 (page 7) are constructed of stainless steel; the gauge is accurate to within 1% of full scale.

To monitor pressure in the canister before and after sampling, use a more accurate measuring device. Test gauges built by Ashcroft are accurate to 0.25% of full scale. These sensitive gauges should not be used in the field - they typically are wall mounted in the lab. Once used for sampling, a gauge must be cleaned, and should be certified clean. Procedures are described later in this guide.

Canister

The canister is a stainless steel vessel designed to hold vacuum to less than 10 mTorr or pressure to 40 psig. Canisters are available in a range of volumes: 850 mL, 1.0 liter, 1.8 liter, 3.0 liter, 6.0 liter, 15 liter, and 35 liter. The size of canister used usually depends on the concentration of the analytes in the sample, the sampling time, the flow rate, and the sample volume required for the sampling period (Table 1, page 3). Typically, smaller canisters are used for more concentrated samples, such as soil gas collection, 3-liter and 6-liter canisters are used to obtain integrated (TWA) ambient air samples at sampling times of up to 24 hours, and large 15-liter and 35-liter canisters are used for reference standards. Sampling time will be limited by the combination of canister size and the flow rate at which the sample is to be collected.

A well-designed canister is essential to the success of the sampling project. First, the canister should be made of stainless steel, so the collected sample will not permeate through the vessel wall or degrade due to exposure to light during shipment to the analytical laboratory. Second, the interior surface of the canister should be inert, to reduce the potential for interactions with the analytes in the sample. Third, all canisters involved in a particular application should be of consistent volume, to simplify calculating sample volumes. Finally, the canister should have a high quality valve that resists abuse in the field (e.g., overtightening that potentially could cause leaks). An inferior valve can fail, causing sample loss and incurring replacement costs. It can be more expensive to sample again than to replace a valve.

Two types of canisters are available, the difference being the interior surface. The traditional canister is the stainless steel SUMMA® canister. The interior of

Figure 4 A flow controller will maintain a constant sample flow despite changes in canister pressure or environmental temperature.

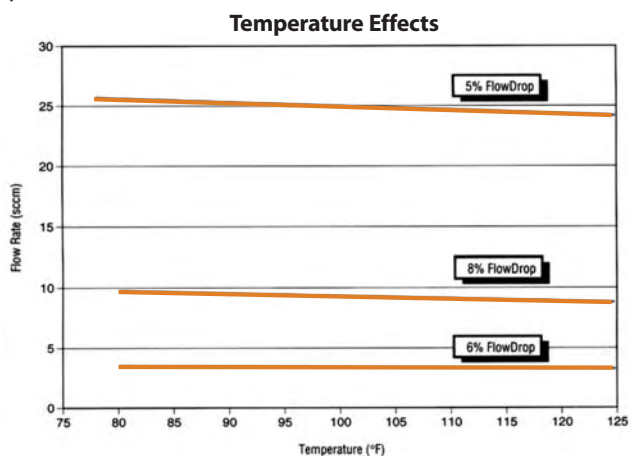
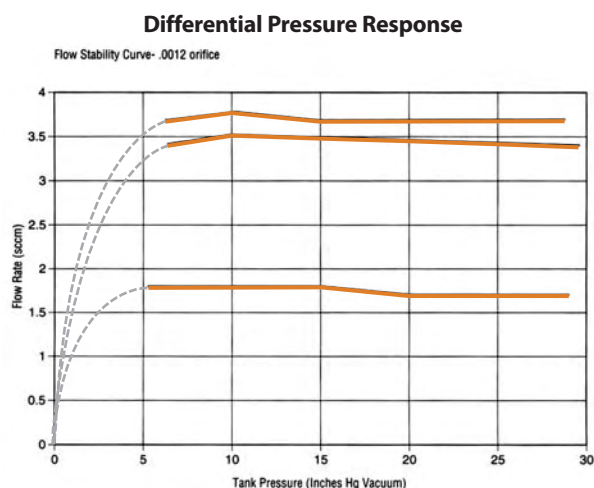


Figure 5 A flow controller will maintain a constant sample flow until it is unable to maintain a stable pressure differential across the critical orifice.



Figures 4 and 5 courtesy of Veriflo Corp., a division of Parker Hannifin Corp.

a SUMMA® canister is electropolished, using a polishing procedure (developed by Moleetrics) that enriches the nickel and chromium surface and makes it more inert than untreated stainless steel. The new generation of sampling canister is typified by the SilcoCan™ canister. Like the SUMMA® canister, the SilcoCan™ canister is made of stainless steel, and the interior is electropolished, but in an additional step - Siltek® treatment - an inert layer is chemically bonded to the interior surface. Siltek® treatment makes the surface inert not only for relatively inactive organic compounds, but also for compounds that are reactive with metal surfaces, such as sulfur-containing compounds. Thus, surface deactivation for SilcoCan™ canisters exceeds that for SUMMA® canisters.

Canister Valve

The valve on a sampling canister must be of high quality, with the following characteristics: leak integrity, a metal seat (to eliminate offgassing of seat components into the sample and memory effects in the seat material), stainless



a plus 1 story

Barry was asked to build and test 20 air sampling canisters, for shipping the same day at 11:30am. He worked nonstop, until the canisters were assembled, quality checked, and packaged for shipment, ensuring a customer had the canisters in time for an important project.

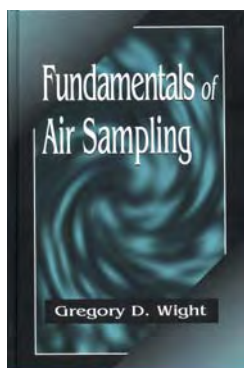
Barry Spicer, Jr.,
Restek Performance Coatings Technician

steel wetted surfaces, and a packless design (a completely enclosed system, to ensure no contamination from lubricants or packing material). Various valves are used on various models of canisters; the most commonly used valves are the Nupro 4H4 series metal bellows valve and the Parker Hannafin diaphragm valve with metal seat. At Restek we incorporate the Parker Hannafin diaphragm valve on canisters because of its ability to hold vacuum, its durability and longevity, and a maximum temperature limit (250°C) that is much higher than that for a bellows valve. Further, a Parker Hannafin diaphragm valve can be rebuilt if it is damaged; it does not have to be replaced.

The connection of the valve to the canister is critical. The connection must be leak tight, to ensure a correct sampling flow rate, but extreme caution must be taken to prevent overtightening the tube compression fittings.

Siltek® and Sulfinert® Treatment

Siltek® treatment is a proprietary process, developed by Restek Corporation, through which an inert layer is chemically bonded to a metal surface. The surface produced by this treatment is virtually inert to active compounds. The stainless steel pathway described in this guide is sufficient for sampling atmospheres containing only non-reactive compounds, but for reactive compounds the entire sampling pathway should be Siltek® treated to eliminate contact between the reactive analytes and the metal surfaces. Siltek® treatment can be applied to the interior surfaces of the canister and valve, to ensure an inert sample pathway. If the samples will contain reduced sulfur-containing analytes, an alternative proprietary Restek deactivation process, Sulfinert® treatment, is the most effective means of deactivating the sample pathway and canister.



Fundamentals of Air Sampling

This book explains the fundamentals of air sampling, develops the theory of gas measurement, and presents several how-to examples of calibration and use of air and gas sampling devices. Other topics include the basics of pressure measurement and units conversion, and specific discussions regarding the use of a Volatile Organic Sampling Train or a SUMMA®-polished canister sampling system.

G. D. Wight, CRC Press LLC, 1994, 272pp., **cat.# 20492**

III. Preparing the Sampling Train for Use

The sampling train must be prepared in the laboratory before it can be used in the field. The train must be assembled and leak tested, the flow rate must be set, and the train must be certified clean. All of the following information should be documented for the chain of custody for the passive sampling train and the sample collected with it.

Assemble, Leak Test, and Set the Flow Rate of the Passive Sampling Train

Choose the critical orifice (Table 1, page 3) according to the sampling period and flow rate you anticipate using (Table 2). This will ensure an accurate and valid sample. There should be a marking on the outside of the critical orifice fitting indicating the size of the orifice. In a clean environment, assemble the sampling train components as shown in Figure 1 (page 3). It is imperative that you leak test the assembled train. If the sampling train leaks during sampling, the final partial pressure in the canister will not be the desired final partial pressure, making the sample invalid. The most common reason for invalid samples is leaks within the sampling train. There are two ways to leak test the train:

1. Pass helium gas through the flow controller and use a sensitive helium leak detector to test for leaks (e.g., Restek Leak Detector).
2. Cap the inlet, attach the sampling train to an evacuated canister, open the valve on the canister and evacuate the sampling train.

Close the valve and monitor any pressure change in the static sampling train. Leaks of less than 1 mL/min. can be detected in 1-2 minutes.

This is a good practical test - the small internal volume of the passive sampling train, combined with even a small leak, will produce a large change in monitored pressure.

After you are certain the sampling train is leak-free, set the desired sampling flow rate.

To set the desired flow rate follow these steps:

1. Remove the protective cap from the back of the Veriflo™ Flow Controller SC423XL body.
2. Connect either an evacuated canister or a vacuum source to the outlet of the sampling train.
3. Connect a high quality calibrated flow meter (i.e., mass flow meter, rotameter, GC-type flow sensor, e.g., Restek Flowmeter 6000, cat. #21622) to the inlet of the train.
4. Apply vacuum by opening the canister or turning on the vacuum source.
5. With a 3mm hex (Allen) wrench, adjust the piston gap screw to achieve the desired flow rate (Table 2). Between adjustments allow the flow to equilibrate for several minutes.
6. Replace the protective cap onto the back of the Veriflo™ Flow Controller body.

Cleanliness: Certifying the Sampling Train for Use

US EPA Compendium Method TO-14A/TO-15 requires that the sampling train be certified clean prior to use. Certify the train by passing a humidified, high-purity air stream through the train, concentrating the exit gas on a trap, and analyzing the gas by gas chromatography / mass spectroscopy or other selective detector. For the sampling train to pass certification the analytical system should not detect greater than 0.2ppbv of any target VOC.

The certified sampling train should be carefully packaged in aluminum foil or in a clean container for storage or for shipment into the field. Care in packaging is critical. Careless handling could affect the preset flow rate. When the sampling train is ready for sampling, prepare the canister.

IV. Preparing the Canister for Sampling

Preparing a canister for sampling involves certifying the canister clean, evacuating the canister to final pressure for use, and identifying the canister. All information acquired during these processes is needed for the chain of custody.

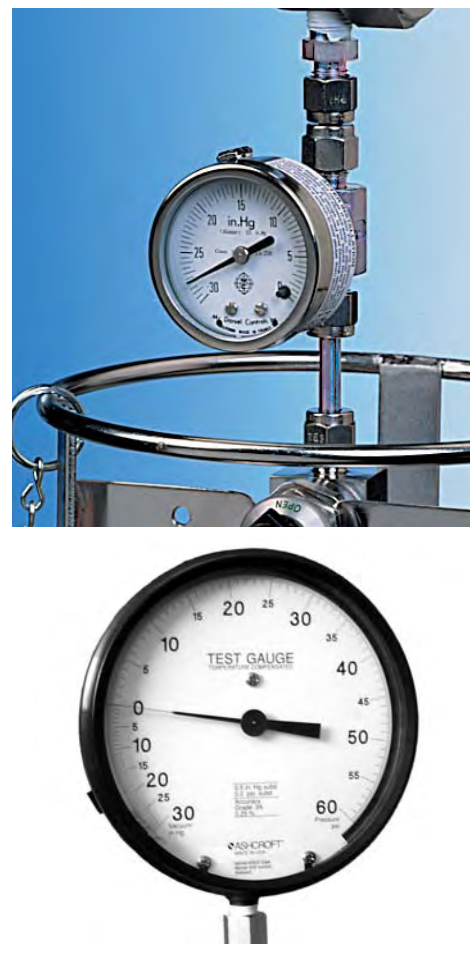
Certifying the cleanliness of the canister is important toward ensuring that results reported are solely from the site sampled, and not admixed with residue from a previous site or from contaminated laboratory air. To certify a canister clean, fill the canister with humidified air, pass the air from the canister through an adsorbent trap and analyze the adsorbent for target VOCs by GC/MS or other selective detector. Two US EPA methods discuss canister certification: EPA Compendium TO-12 and EPA Compendium TO-14A/TO-15. To comply with EPA Compendium Methods TO-14A/TO-15, the analytical system should not detect greater than 0.2ppbv of any target VOC. To comply with EPA Compendium Method TO-12 the analytical system, GC/FID, should not detect greater than 0.02ppmvC hydrocarbons. Although batch certification of canister cleanliness is a relatively common practice, we recommend certifying and documenting each canister individually. Detailed cleaning instructions are presented in Section VIII. Cleaning the Canister (page 12).

Table 2 Flow rates for integrated sampling, using a 6-liter canister and sampling on the flat portion of the flow curve for the flow controller (Figure 5).

Sampling Period (hours)	0.5	0.75	1	2	4	8	12	16	24
Flow Rate Range (mL/min.)	133-167	89-111	67-83	33-42	17-21	8-10	5.6-6.9	4.2-5.2	2.8-3.5

Collected volume is 4-5 liters (flow = volume in mL / sampling time in min.).

Figure 6 Conventional vacuum gauge and high-accuracy test gauge.



did you know?

Restek canisters are shipped in boxes with handles for ease of carrying and transporting, eliminating costly carrying cases.



Some laboratories certify a canister for VOC stability by introducing a low concentration test mixture into the canister and measuring degradation over a specified time period. If the canister meets the specification, it is certified for use. We recommend using such studies to ensure the effectiveness of a canister or group of canisters for a proposed application.

Once the canister is certified clean, evacuate the canister to a final vacuum of 10-50mtorr, using either the canister cleaning system or a clean final vacuum system. This vacuum is critical to ensure the correct amount of sample is collected. Use an accurate test gauge or digital pressure tester to ensure final vacuum has been reached and document the final vacuum reading for the chain of custody. Install a brass plug nut into the canister valve to ensure no contamination can enter the sample pathway during shipment to the field.

Allot an individual identity to the canister, either with a label and serial number or with a bar code.

Some analysts prefer to introduce surrogate standards into the canister prior to sampling. Debate on this practice revolves around theories that there are potential loss issues due to low humidity and inadequate surface passivation by water. Neither Restek chemists nor our consulting experts recommend adding surrogates to the canisters. If you choose to introduce surrogates into your canisters prior to sampling be sure to recheck the vacuum reading for each canister after adding the surrogates, and record the reading.

V. Field Sampling, Using a Passive Sampling Train and Canister

It is important to mention again that the sampling train and canister must be leak tested and certified clean prior to use. To properly begin field sampling, we recommend bringing a "practice" evacuated canister and a flow measuring device with you to the field. Use this canister to verify the flow rate through the passive sampling train prior to using the train to obtain samples of record. To verify the flow rate, connect the passive sampling train to the "practice" canister. Attach a flow meter to the inlet of the sampling train. Open the canister, and measure the flow rate through the sampling train. If the flow rate is within +/- 10% of the flow rate set in the lab, the train is ready to be used on the formal sampling canister. If the flow rate is not within these limits, adjust the flow rate by adjusting the piston gap screw.

When the flow rate is confirmed, record the rate as the canister flow rate for the chain of custody form.

To begin sampling, using the formal sampling canister, follow these steps:

1. Remove the brass plug nut from the canister valve.
2. If you are using a test gauge, attach the gauge to the canister and record the vacuum reading. If you choose not to use a test gauge under field conditions, record the reading on the vacuum gauge that is part of the passive sampling train.
3. Attach the verified passive sampling train to the canister.
4. Record the begin sampling time and necessary meteorological data.
5. Open the canister valve and begin sampling.
6. Periodically check the canister throughout the sampling period to ensure the partial pressure reading is accurate and sampling is proceeding as planned.
7. Once the sampling period is complete, close the valve and remove the sampling train. Check the final partial pressure within the canister, using the test gauge or the vacuum gauge in the sampling train.

There are four possible scenarios:

- A. Ideally there will be a vacuum of 7"-4" Hg in the canister (see, e.g., Table 3 on page 10).
 - B. If more than 7" Hg vacuum remains, less sample was collected than initially anticipated. The sample will be valid, but the detection limit may be higher than expected. You might have to pressurize the canister prior to the analysis, which will dilute the sample, then you will have to use a dilution factor to determine final concentrations of target compounds.
 - C. A vacuum of less than 4" Hg indicates the sample might be skewed toward the initial part of the sampling period. This assumption usually is valid because the flow rate through the flow controller will fall once the vacuum falls below 5" Hg (Figure 5, page 5), when the change in pressure across the flow controller diaphragm becomes too small and the flow controller is unable to maintain a constant flow. Although flow was not constant over the entire sampling period, the sample might be usable because sample was collected over the entire interval.
 - D. If the ending vacuum is less than 1" Hg the sample should be considered invalid because it will be impossible to tell when the sample flow stopped.
8. Record the final partial pressure in the canister and replace the plug nut.

Information that should be acquired at the sampling site includes the start time and interval time, the stop time, atmospheric pressure and temperature and, for ambient sampling, wind direction. Include elevation if it is a factor. These parameters often prove very useful toward interpreting results.

After sampling, the canisters are sent back to the laboratory, where the final vacuum is measured once again, with a test gauge. Using the initial vacuum and final vacuum, the sample volume collected can be determined from Equation 1:

Equation 1:

$$\text{sample volume} = \frac{\text{pressure change}^*}{\text{atmospheric reference pressure}} \times \text{canister volume}$$

*initial pressure - final pressure

Example: A sample is collected in a 6-liter canister. The initial gauge pressure reading when the canister left the lab was 29" Hg vacuum; the final gauge pressure reading when the canister was returned to the lab was 7" Hg vacuum.

$$\text{sample volume} = [(29\text{"Hg} - 7\text{"Hg}) / 29\text{"Hg}] \times 6\text{L} = 4.55 \text{ liters collected.}$$

It is also a good practice to recheck the flow rate after sampling, because this will affect the sample volume (Equation 2). Laboratories typically allow a maximum deviation of +/-10% to +/-25% between the initial flow rate and the post-sampling flow rate.

Equation 2:

$$\text{sample volume} = [(\text{initial flow rate} + \text{post-sampling flow rate})/2] \times \text{sampling time}$$

Example: A flow controller was set at 3.3mL/min. After obtaining a 24 hour sample the flow rate was 3.0mL/min.

$$\text{sample volume} = [(3.3\text{mL/min.} + 3.0\text{mL/min.}) / 2] \times 1440 \text{ min.} = 4536\text{mL.}$$



Methods of Air Sampling and Analysis, 3rd Edition

This book includes precise directions for analyzing a long list of air contaminants. All contaminants one can analyze or monitor using a given method are consolidated to facilitate use. An excellent reference manual for all analytical laboratories conducting air analyses.

J. P. Lodge, CRC Press LLC, 1988, 784pp., **cat.# 20493**

frequently asked question

Where can I find EPA Air Toxic Methods?

pdf files of US EPA Air Toxic Methods are available at this web address: www.epa.gov/ttn/amtic/airtox.html

VI. Analysis of Collected Samples

Once received by the lab, each canister is identified from the information in the chain of custody report. The final partial pressure is checked to ensure no leaks appeared during transport. It might be necessary to pressurize a canister prior to the analysis; do this by adding humidified nitrogen or air to the canister to a pressure greater than 5 psig or higher, depending on the sample volume needed for analysis or for suitably diluting the sample (e.g., Table 4). The need to dilute is determined by the preconcentrator instrument. Some air preconcentrators can be operated while the canister is under slight vacuum. Check with your instrument manuals, or with the manufacturer, to determine if you must dilute your samples prior to analysis.

Equation 3:

$$\text{dilution factor} = (P_{\text{after dilution}} + P_{\text{lab atmosphere}}) / (P_{\text{lab atmosphere}} - P_{\text{before dilution}})$$

The dilution factor is calculated from the post-sampling pressure (before dilution), the final pressure (after dilution), and the atmospheric pressure in the laboratory. The factor for converting "Hg to psi = 0.491.

Example: At the end of a sampling period the gauge pressure in a canister was 7"Hg. The canister was pressurized with nitrogen to 14.7psig (1 Atm.).

The dilution factor is $(14.7 + 14.7) / (14.7 - (7 \times 0.491)) = 2.61$

Table 3 Final vacuum and volume of sample collected in 6-liter canister.

Final Vacuum ("Hg)	29"	27"	25"	23"	20"	17"	15"	12"	10"	7"	5"	3"	0"
Sample Volume (liters)	0	0.414L	0.83L	1.24L	1.86L	2.48L	2.90L	3.52L	3.93L	4.55L	5.0L	5.38L	6L

To analyze the sample, withdraw an aliquot of the sample from the canister. For low level ambient air analysis, withdraw 250-500 mL of sample from the canister and concentrate the analytes by using a mass flow controller and a cryogenically cooled trap (e.g., glass beads and/or a solid sorbent). Desorb the concentrated analytes from the trap and deliver them to a cryofocuser, to focus the sample bandwidth prior to introduction onto the GC column. A 60m x 0.32mm ID x 1.0µm Rtx®-1 column typically is used for EPA Method TO-14A or Method TO-15 ambient air analysis; an MSD is a common detector. Figure 7 shows a typical TIC spectrum for a TO-14A/TO15 ambient air analysis.

Procedures used in these chromatographic analyses generally include a multi-point calibration, using gas standards. Therefore calculations of organic compounds in collected samples are straightforward - only volumes analyzed and dilution rates are needed to determine sample concentrations. High concentration calibration gas standards are commercially available (e.g., 1ppmv or 100ppbv); introduce an aliquot of stock material into a canister and dilute with humidified air or nitrogen. After analyzing the calibration standards, determine the response factor for each analyte, using the peak area counts per concentration.

After analyzing the multipoint calibration standards and calculating peak area/concentration response factors, analyze the "real world" samples. If an "unknown" sample has not been diluted apply the corresponding response fac-

Table 4 Dilution factors to adjust final sampling pressure to **14.7psig** for a 6-liter canister.

Final Vacuum ("Hg)	29"	27"	25"	23"	20"	17"	15"	12"	10"	7"	5"	3"	0"
Sample Volume (liters)	0	0.414L	0.83L	1.24L	1.86L	2.48L	2.90L	3.52L	3.93L	4.55L	5.0L	5.38L	6L
Dilution Factor	63.71	20.37	10.12	8.63	6.02	4.63	4.01	3.34	3.00	2.61	2.40	2.22	2.00



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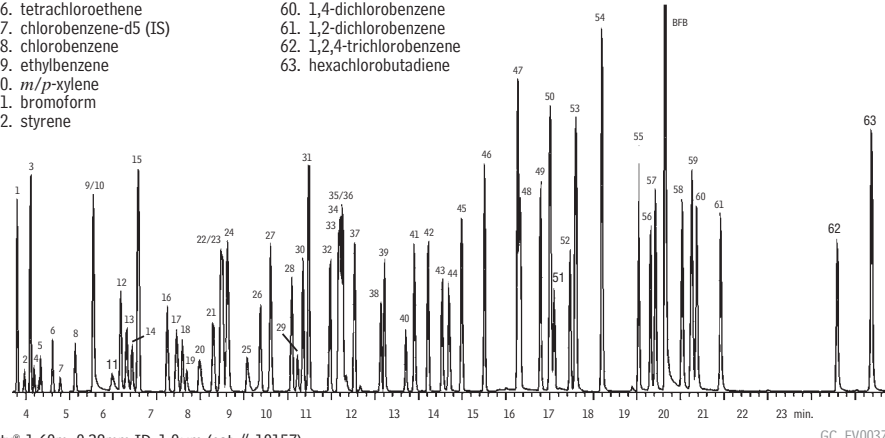
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Figure 7 TLC spectrum for a TO-14/TO-15 ambient air analysis.

1. dichlorofluoromethane
2. chloromethane
3. dichlorotetrafluoroethane
4. vinyl chloride
5. 1,3-butadiene
6. bromomethane
7. chloroethane
8. bromoethene
9. acetone
10. trichlorofluoromethane
11. isopropyl alcohol
12. 1,1-dichloroethene
13. methylene chloride
14. 3-chloropropene
15. carbon disulfide
16. Freon® TF
17. *trans*-1,2-dichloroethene
18. 1,1-dichloroethane
19. methyl *tert*-butyl ether
20. methyl ethyl ketone
21. *cis*-1,2-dichloroethene
22. bromochloromethane (IS)
23. *n*-hexane
24. chloroform
25. tetrahydrofuran
26. 1,2-dichloroethane
27. 1,1,1-trichloroethane
28. benzene
29. carbon tetrachloride
30. cyclohexane
31. 1,4-difluorobenzene (IS)
32. 1,2-dichloropropane
33. bromodichloromethane
34. trichloroethene
35. 1,4-dioxane
36. 2,2,4-trimethylpentane
37. *n*-heptane
38. *cis*-1,3-dichloropropene

39. methyl isobutyl ketone
40. *trans*-1,3-dichloropropene
41. 1,1,2-trichloroethane
42. toluene
43. methyl butyl ketone
44. dibromochloromethane
45. 1,2-dibromoethane
46. tetrachloroethene
47. chlorobenzene-d5 (IS)
48. chlorobenzene
49. ethylbenzene
50. *m/p*-xylene
51. bromoform
52. styrene

53. 1,1,2,2-tetrachloroethane
54. *o*-xylene
55. 2-chlorotoluene
56. 4-ethyltoluene
57. 1,3,5-trimethylbenzene
58. 1,2,4-trimethylbenzene
59. 1,3-dichlorobenzene
60. 1,4-dichlorobenzene
61. 1,2-dichlorobenzene
62. 1,2,4-trichlorobenzene
63. hexachlorobutadiene



Rtx®-1 60m, 0.32mm ID, 1.0µm (cat.# 10157).

Sample: 200mL of 10ppbv TO-15 standard (cat.# 34436), injected into TO-Can™ canister and humidified to 70% RH.

Concentrator: Nutech 3550 Preconcentrator

200mL of sample concentrated at 160°C, thermally desorbed at 150°C, and cryofocused at 185°C.

Oven temp.: 30°C (hold 4 min.) to 175°C @ 9°C/min. to 220°C @ 40°C/min.

Carrier gas: helium @ 1.2mL/min.

Det.: Agilent 5971 MS

Scan range: 35-265amu

tor to each “unknown” analyte peak area to get the reporting limit concentration of the “unknown” in the analysis (typically in ppbv). If you have diluted the canister to get a positive pressure, however, you must apply the dilution factor to the concentration values. This is done by multiplying the reporting limit by the dilution factor.

VII. Cleaning the Passive Sampling Train

The cleanliness of the sampling train is critical to collecting accurately representative samples. Practices followed for cleaning passive sampling equipment between uses range from purging the sampling pathway with humidified nitrogen or air for many hours to heating the pathway during a purge to disassembling each component, sonicating the pieces in solvent, and oven baking the pieces prior to re-assembly. The most suitable mode of cleaning depends on the concentrations of analytes of interest, and contaminants, in the previous sample collected.

The particle filter must be thoroughly cleaned between uses. Disassemble the filter, then remove the larger particles from the frit by blowing particle-free nitrogen through the frit from the outlet surface toward the inlet surface. After the larger particles are removed sonicate or rinse the filter parts in methanol, then bake the parts in an oven at 130°C to remove any residual organic vapors.

The critical orifice and flow controller can be cleaned in either of two ways. The first method is to disassemble the flow controller and clean all the metal parts with methanol. This will remove any high boiling compounds that have condensed onto the wetted areas of the controller. Heat the cleaned parts in an oven at 130°C to remove residual organic vapors. Do not sonicate in solvent or bake any of the non-metallic parts, such as O-rings; they will be damaged during these steps. Do not rinse the vacuum gauge with methanol. The vacuum gauge may be heated, but do not exceed 80°C; higher temperatures will damage the face and the laminated safety glass lens. Heating to 80°C will not affect the mechanical operation of the spiral bourdon tube in the vacuum gauge.

A less involved method of cleaning the flow controller is to use a heating jacket or heat gun to heat the components of the assembled sampling train, while purging the system with nitrogen. As organic compounds are heated and desorbed from the interior surfaces the nitrogen gas sweeps them out of the sampling equipment.

Preparing the Clean Passive Sampling Train for Re-use

After the sampling train components have been cleaned, reassemble the system, check for leaks, set the desired flow rate, and certify the sampling system clean. Follow the procedures described previously in this guide. Package the clean sampling train to prevent contact with airborne contaminants.

VIII. Cleaning the Canister

Every air sampling canister, whether new or previously used, must be cleaned and certified before it is used for sampling. Some laboratories batch test and certify canisters, in which after cleaning, one canister out of 10 is tested and certified clean. We recommend certifying each canister clean prior to use, however, especially if there is potential for litigation.

For many years there has been much discussion as to what constitutes a proper procedure for cleaning canisters. US EPA Method TO-14A has provided guidance, and in the last 5-10 years many automated commercially available canister cleaning systems have evolved. Unfortunately, because these systems are quite expensive, and some designs have limitations, many analysts design their own systems and methodologies for cleaning canisters. The cleaning procedure described in this section is a practical approach that will ensure canisters are suitably cleaned for ambient air sampling, whether you are using a commercially available cleaning system or a system of your own design. There are minor differences when cleaning SilcoCan™ or SUMMA® canisters. We will discuss these differences in this procedure.

Air Versus Nitrogen

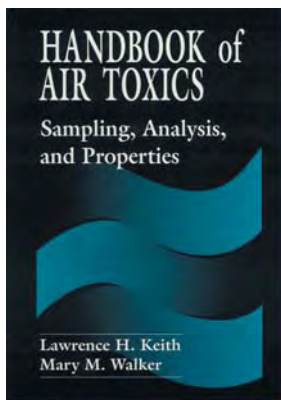
The two gases recommended for cleaning canisters are humidified ultra-high purity air and ultra-high purity nitrogen. The water in the humidified gas hydrolyzes impurities in the canister and, according to theory, will occupy the active sites on the interior surface, displacing the impurities and allowing them to be removed. Air is recommended when oxidation of the interior surface is desired. The oxygen content of air, 21%, is sufficient for this surface oxidation; it is not necessary to use pure oxygen gas. Nitrogen is equally effective for cleaning ambient air canisters, but, of course, nitrogen will not oxidize the surface of the canister.

Heat or No Heat

Many user-designed canister cleaning systems do not heat the canisters. Typically this does not create a problem when cleaning canisters that are used in ambient air collection, but as a safeguard we recommend heating the canisters during the cleaning process. Compounds collected in most ambient air samples are in the low ppbv range, and can be removed from a canister by multiple cycles of pressurization with humidified air or nitrogen followed by evacuation. If there are higher concentrations of contaminants in the canister, however, heat might be required to clean the canister satisfactorily.

Be aware that adding heat and humidified gas to a canister potentially can create a steam pressure vessel. Some commercial cleaning systems incorporate a pressure release valve to ensure the pressure does not exceed the pressure rating of the canisters.

A heating option can be added to a user-designed canister cleaning system by using an oven, heat bands, insulated jackets, or an infrared source.



Handbook of Air Toxics. Sampling, Analysis and Properties

This reference book contains physical and chemical data for all the chemicals in the National Toxicology Program's Chemical Database and all substances indicated in the US EPA Clean Air Act Amendments.

L. H. Keith and M. M. Walker, CRC Press LLC, 1995, 640pp., cat.# 21373

Oven

Some canister cleaning systems are incorporated within an oven. The supply line for the humidified air stream and the line to the vacuum system are plumbed directly into the oven. In this arrangement the entire canister, including the valve, will be heated, and this will help remove contaminants if both the valve and the canister are dirty. Typically, when using heat, it is helpful to create steam from the humidified air stream. An oven temperature of at least 120°C is required, but higher temperatures often are used. Remember that heat can shorten the lifetime of the Nupro valve on a SUMMA® canister (see step 3 in Cleaning Method, below).

Space is a concern with oven systems. Most commercial ovens are not very large and this restricts the number of canisters that can be cleaned at one time. However, clean-up times are shorter with heat than without heat, so more cleaning cycles can be completed in a week.

Heat Bands

A band heater placed around the equator of the canister typically will be capable of heating the canister to approximately 130°C. There is a heat gradient, and the valve might only receive radiant heat (approximately 70-100°C). In most sampling situations, however, this lower temperature should be sufficient for effectively removing contaminants from the valve.

Insulated Heat Jackets

Insulated heat jackets can be obtained to surround and heat each canister. These jackets typically have a silicone or Teflon®-coated fiberglass fabric exterior and a fiberglass insulation interior. Some operate at a fixed temperature; others can provide variable temperature, up to 400-500°F. Restek's heating jacket, described at right on this page, offers significant advantages over most other commercial designs, which do not encompass the valve area.

Infrared Heat

An infrared heating system includes an infrared heat source and a reflective panel similar to the cylinder drying rack on a gas cylinder system. The infrared source and the reflective panel are placed on opposing sides of the canisters. Infrared rays from the source heat the canisters; rays that pass the canisters strike the reflective panel and heat the canisters from the opposing side.

The Cleaning System

The cleaning system must provide a humidified air stream and include a good vacuum source, a cold trap to collect impurities during cleaning, and accurate gauges to read vacuum and pressure. A heat source is optional, but is highly recommended in some circumstances, as discussed above. The system can be designed to clean 4 to 24 six-liter canisters. Figure 8 (page 14) shows an example of a "homemade" system designed to clean 24 six-liter canisters. This design does not employ heat, but a heater easily can be added (see Heat or No Heat, page 12). It provides a humidified air stream to all canisters and the roughing pump on the bottom shelf is the vacuum source. This system is computer operated to minimize labor, but this is not necessary.

Cleaning Method

1. Connect all canisters to the cleaning system, then release any pressure within any of the canisters. Put the system under vacuum, to evacuate the canisters. US EPA Method TO-14A/15 recommends evacuating the system to 50 mTorr for 1 hour, but a reduced pressure of 23-25" Hg is sufficient for general cleaning.
2. After the canisters have been under vacuum for approximately 1 hour, pressurize the canisters with humidified air or nitrogen*. Pressurization will dilute the impurities and the moist air will hydrolyze them. Pressurize canisters to 5 psig if they will be heated or to 30 psig if they will not be heated. Proceed to step 3 when the system has equilibrated at the designated pressure.



The ultimate in controlled heating, for reliably cleaning your air canisters!

Air Canister Heating Jacket

- Closely simulates oven environment—heats entire canister.
- Two temperature settings, 75°C and 150°C.
- Prevents sample condensation, for accurate sub-sampling.
- Easily fits canister up to 6 liters.
- Lightweight; comfortable to the touch when heated.
- Connect up to five Canister Heating Jackets to one 15 amp circuit.

The Restek Canister Heating Jacket will help you clean your canisters faster and more efficiently. The novel design ensures the entire canister, including the valve, is heated during the cleaning cycle, to remove contaminants most effectively. It also can be used to keep the sample heated during aliquot removal, which helps prevent condensation and assure accurate data for larger molecules. The two heat settings let you match the temperature to the volatility of your sample components. If you try one in your system, we think you'll want more.

Description	qty.	cat.#
Air Canister Heating Jacket (110 volt)	ea.	24123

*please note

If you are cleaning SilcoCan™ canisters, and will be using heat, use humidified nitrogen, not air.

Figure 8 User-designed system for cleaning 24 six-liter canisters.



- Heat the pressurized canisters to 120 - 250°C, depending on the type of canister being cleaned. Do not allow the temperature of a SUMMA® canister to exceed 155°C, because the Nupro valve it employs has Viton® O-rings and requires greases that cannot be exposed to high temperatures. Many commercial cleaning systems avoid this problem by ensuring the valve is not within the heated zone. The canister below the valve is heated but the valve receives only radiant heat. In contrast, the Parker Hannifin diaphragm valve in a SilcoCan™ canister is far less heat sensitive, allowing the canister to be cleaned at temperatures up to 250°C, to help remove less labile impurities.

Heat the canisters filled with humidified air for at least 1 hour.

- Re-evacuate the canisters to remove the desorbed impurities. Allow the canisters to equilibrate for 1 hour.
- Determine if the canisters have been cleaned effectively by following the procedure in Certifying the Canister, below. US EPA methods recommend testing every canister until a reliable procedure is developed.

Repeat steps 1-5 as necessary; the number of cycles will be determined by how dirty the canisters are and how easily they are cleaned. We recommend developing a cleaning procedure that matches your specific sampling procedure, by testing the canisters for cleanliness after each cycle and determining the number of cycles necessary for proper cleaning. If the canisters are not heated, the number of cycles required to clean the canisters might be higher.

- Once a canister is clean, prepare it for collecting a sample by evacuating it to 10-50 mTorr. If your system is leak-tight, you can do this by using a roughing pump, but many commercial systems include a molecular drag pump to reach final vacuum quickly.

IX. Certifying the Canister

We recommend certifying canisters for both cleanliness and for analyte stability. To certify a canister clean, pressurize the canister to 14.7 psig with humidified ultra-high purity air or nitrogen after it has gone through the cleaning cycles. The humid air or nitrogen stream must be certified clean before it can be used for canister certification. Analyze an aliquot of the canister content by GC/MS or GC/FID/ECD. US EPA Method TO-14A/15 specifies a canister must contain less than 0.2 ppbv of any target VOC compound (Figure 9); EPA Method TO-12 specifies less than 0.02 ppmC, as detected by GC/FID. If a canister does not meet specification, it must be re-cleaned and re-tested for certification.

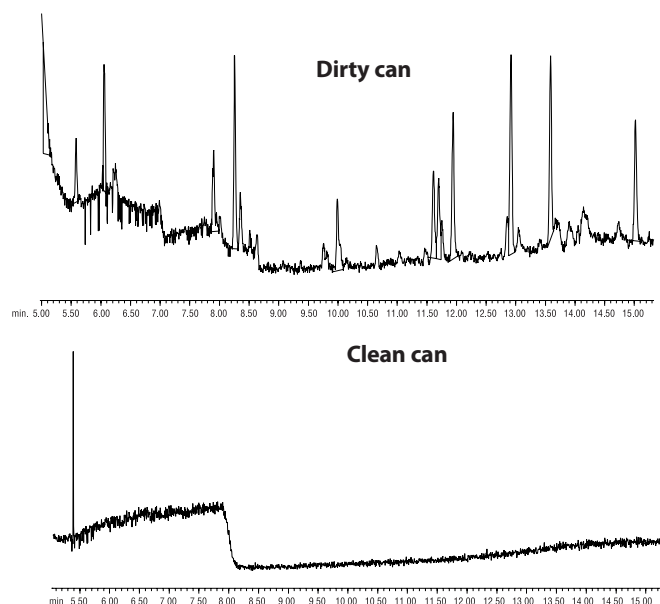
To certify a canister for analyte stability, introduce a low working concentration of a characterized test mix into the canister. Analyze an aliquot of the contents of the canister immediately after introducing the test mixture and at periodic intervals. We recommend monitoring for changes for a minimum of 2 weeks, or for a timeframe similar to your anticipated holding period. Responses should not decrease more than 20% over this period.

Commercially available standards are available for stability testing, but we recommend you make your own test mixture that is comparable to the target compound list that the canister will hold. For example, if you are analyzing sulfur compound content in ambient air, prepare a sulfur-specific test mix and evaluate the canister's performance for sulfurs. Maintain a log sheet for each canister, and record the test results and certification. This will be a permanent record for each canister. Some labs certify canisters for certain compounds and use a canister only for this specific application.

X. Conclusion

A well designed and properly prepared passive sampling system helps ensure accurate, useful information is obtained from an air sampling project. In this guide, we describe the components of the system, procedures for assembling the system and preparing it for sampling, and the sampling procedure. Cleaning system options and procedures for cleaning a used sampling train and canister for certification prior to a subsequent sampling also are presented. The following section describes Restek products designed to help collect and analyze air samples.

Figure 9 Aliquots from a canister before and after cleaning with 2 cycles of humidified air while heated to 200°C.



Rtx®-1 60m x 0.32mm ID x 1.0µm (cat. #10157)
50°C (hold 1min) to 165°C @ 8°C/min.
to 220°C @ 15°C/min. (hold 5 min.)
flow rate = 1.4mL/min.

Nutech 3550 Preconcentrator
conditions
sample = 300cc from canister
cryotrap = -160°C
desorb = 150°C
cryofocuser = -190°C
desorb = 150°C

MSD conditions
Instrument: HP5971 GC/MSD
5 minute solvent delay
scan range = 25-260amu
detector temp. = 250°C

Pressure Conversion Table

Pressure	PSI	atm	kg/cm ²	torr	kPa	bar	inches Hg
PSI =	1	0.068	0.0703	51.713	6.8948	0.06895	2.0359
atm =	14.696	1	1.0332	760	101.32	1.0133	29.921
kg/cm ² =	14.223	0.967	1	735.5	98.06	0.9806	28.958
torr =	0.0193	0.00132	0.00136	1	0.1330	0.00133	0.0394
kPa =	0.1450	0.00987	0.0102	7.52	1	0.0100	0.2962
bar =	14.5038	0.9869	1.0197	751.88	100	1	29.5300
in Hg =	0.49612	0.0334	0.0345	25.400	3.376	0.03376	1

Multiply units in the left-most column by the conversion factors listed in the columns to the right.
e.g., 10PSI x 0.068 = 0.68atm, 10 bar x 29.5300 = 295.300 inches Hg



Alternative Vacuum/Pressure Gauges

The standard vacuum/pressure range on a SilcoCan™ or TO-Can™ canister fitted with a gauge is 30" Hg to 60psig. To order a different gauge for the canister, add the appropriate suffix number to the canister catalog number. There is no price difference for these alternative gauges.

Gauge	Suffix
30" Hg/15psi	-651
30" Hg/30psi	-652

did you know?

We ship our canisters cleaned, batch-tested per USEPA TO-14, and under 30psig pressure with dry nitrogens.

Refer to our catalog or our website for replacement pressure gauges.



XI. Air Sampling Products

SilcoCan™ Air Monitoring Canisters

Siltek® treated - ideal for low-level reactive sulfur compounds (1-20ppb)

- Unsurpassed inertness, even for sulfur-containing or brominated compounds.
- Sizes from 1 to 15 liters support a wide range of sampling needs.
- Optional 3-port valve allows attachment of vacuum/pressure gauge for monitoring canister pressure.
- For critical applications, order a Siltek® treated valve - add suffix "-650" to the catalog number of the canister.

For ultimate inertness, we treat SilcoCan™ air monitoring canisters with our unique Siltek® passivation technology. Even highly active components, at low parts-per-billion concentrations, can be readily sampled and stored without loss. The valve is a high quality, metal-to-metal seal, 2/3-turn valve with metal diaphragms. Both stainless steel and Siltek® treated valves are available, in both the 2-port and 3-port configurations.

Description	qty.	cat.#
1L Volume		
SilcoCan™ Canister, 1/4" Valve	ea.	24180
SilcoCan™ Canister, Siltek®-Treated 1/4" Valve	ea.	24180-650
SilcoCan™ Canister with Gauge, 1/4" Valve	ea.	24140
SilcoCan™ Canister with Gauge, Siltek®-Treated 1/4" Valve	ea.	24140-650
3L Volume		
SilcoCan™ Canister, 1/4" Valve	ea.	24181
SilcoCan™ Canister, Siltek®-Treated 1/4" Valve	ea.	24181-650
SilcoCan™ Canister with Gauge, 1/4" Valve	ea.	24141
SilcoCan™ Canister with Gauge, Siltek®-Treated 1/4" Valve	ea.	24141-650
6L Volume		
SilcoCan™ Canister, 1/4" Valve	ea.	24182
SilcoCan™ Canister, Siltek®-Treated 1/4" Valve	ea.	24182-650
SilcoCan™ Canister with Gauge, 1/4" Valve	ea.	24142
SilcoCan™ Canister with Gauge, Siltek®-Treated 1/4" Valve	ea.	24142-650
15L Volume		
SilcoCan™ Canister, 1/4" Valve	ea.	24183
SilcoCan™ Canister, Siltek®-Treated 1/4" Valve	ea.	24183-650
SilcoCan™ Canister with Gauge, 1/4" Valve	ea.	24143
SilcoCan™ Canister with Gauge, Siltek®-Treated 1/4" Valve	ea.	24143-650

TO-Can™ Air Monitoring Canisters

Optimized for US EPA Methods TO-14 and TO-15

- High quality, metal-to-metal seal, 2/3-turn valve with metal diaphragms.
- Sizes from 1 to 15 liters.
- Optional 30" Hg/60psig vacuum/pressure gauge (other gauges available).

Description	qty.	cat.#
1L Volume		
TO-Can™ Canister, 1/4" Valve	ea.	24172
TO-Can™ Canister with Gauge, 1/4" Valve	ea.	24176
3L Volume		
TO-Can™ Canister, 1/4" Valve	ea.	24173
TO-Can™ Canister with Gauge, 1/4" Valve	ea.	24177
6L Volume		
TO-Can™ Canister, 1/4" Valve	ea.	24174
TO-Can™ Canister with Gauge, 1/4" Valve	ea.	24178
15L Volume		
TO-Can™ Canister, 1/4" Valve	ea.	24175
TO-Can™ Canister with Gauge, 1/4" Valve	ea.	24179

1/4" Replacement Valves for Air Monitoring Canisters*

Description	Stainless Steel Valve		Siltek®-Treated Valve	
	qty.	cat.#	qty.	cat.#
1/4" Replacement Valve (2-port)	ea.	24145	ea.	24144
1/4" Replacement Valve (3-port)	ea.	24147	ea.	24146

*All Restek canisters are originally equipped with these high-quality Parker Hannifin diaphragm valves. Each valve is helium leak-tested to 4 x 10⁻⁶ cc/sec. The all-stainless steel construction eliminates contamination and withstands temperatures from -100°C to 250°C. Compression outlet fitting, indicator plate to display open or closed position, 1/4" inlet and outlet.



Phone 800 356 1600 or 014 322 1200

www

HRMalytic +61(0)3 9762 2034
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Importers & Manufacturers
www.chromtech.net.au

Website NEW : www.chromalytic.com.au E-mail : info@chromtech.net.au Tel: 03 9762 2034 . . . in AUSTRALIA

Miniature Air Sampling Canisters—an alternative to tube and pump samplers

- Ideal for indoor air, personal, emergency response, or soil gas sampling (applications ≤ 40psig).
- Available with quick-connect (1/4" tube) fitting, compatible with sampling and analysis instruments.
- Available with non-treated or Sulfinert®-treated valve.
- 1000cc canister suitable for US EPA Methods TO-14 and TO-15.

Description	Volume	qty.	cat.#
Electro-Polished Miniature Canister with Quick-Connect Stem Fitting	400cc	ea.	24188
	1000cc	ea.	24194
Sulfinert®-Treated Miniature Canister with Quick-Connect Stem Fitting	400cc	ea.	24189
	1000cc	ea.	24195
Sulfinert®-Treated Miniature Canister with Sulfinert®-Treated Quick-Connect Stem Fitting	400cc	ea.	24190
	1000cc	ea.	24196
Electro-Polished Miniature Canister with Metal-Seated Diaphragm Valve	400cc	ea.	24191
	1000cc	ea.	24197
Sulfinert®-Treated Miniature Canister with Metal-Seated Diaphragm Valve	400cc	ea.	24192
	1000cc	ea.	24198
Sulfinert®-Treated Miniature Canister with Sulfinert®-Treated Diaphragm Valve	400cc	ea.	24193
	1000cc	ea.	24199
Electro-Polished Miniature Canister with Nut & Ferrule	400cc	ea.	24205
	1000cc	ea.	24206
Sulfinert®-Treated Miniature Canister with Nut & Ferrule	400cc	ea.	24207
	1000cc	ea.	24208



Dimensions:
400cc = 2.75" diameter,
5.35" long
1000cc = 2.75" diameter,
11.92" long

Quick-Connect Fittings for Miniature Air Sampling Canisters (1/4" tube fitting)

Description	qty.	cat.#
Quick-Connect Stem Fitting	ea.	24185
Sulfinert®-Treated Quick-Connect Stem Fitting	ea.	24186
Quick-Connect Body Fitting	ea.	24187

Air Monitoring Gas Standards (see our catalog or website for others)

TO-14A Calibration Mix (39 components)

benzene	ethyl chloride
bromomethane	hexachloro-1,3 butadiene
carbon tetrachloride	methylene chloride
chlorobenzene	styrene
chloroform	1,1,2,2-tetrachloroethane
chloromethane	tetrachloroethylene
1,2-dibromoethane	toluene
m-dichlorobenzene	1,2,4-trichlorobenzene
o-dichlorobenzene	1,1,1-trichloroethane
p-dichlorobenzene	1,1,2-trichloroethane
dichlorodifluoromethane	trichloroethene
1,1-dichloroethane	trichlorofluoromethane
1,2-dichloroethane	1,1,2 trichlorotrifluoroethane
1,1-dichloroethene	1,2,4-trimethylbenzene
cis-1,2-dichloroethene	1,3,5-trimethylbenzene
1,2-dichloropropane	vinyl chloride
cis-1,3-dichloropropene	m-xylene
trans-1,3-dichloropropene	o-xylene
dichlorotetrafluoroethane	p-xylene
ethyl benzene	

In nitrogen, 104 liters @ 1800psig

1ppm	100ppb
34400 (ea.)	34421 (ea.)

TO-15 62 Component Mix (62 components)

acetone	4-ethyltoluene
benzene	trichlorofluoromethane (Freon® 11)
benzyl chloride*	dichlorodifluoromethane (Freon® 12)
bromodichloromethane	1,1,2-trichloro-1,2,2-trifluoroethane (Freon® 113)
bromoform	1,2-dichlorotetrafluoroethane (Freon® 114)
bromomethane	heptane
1,3-butadiene	hexachloro-1,3-butadiene
2-butanone (MEK)	hexane
carbon disulfide*	2-hexanone (MBK)
carbon tetrachloride	4-methyl-2-pentanone (MIBK)
chlorobenzene	methylene chloride
chloroethane	methyl tert-butyl ether (MTBE)
chloroform	2-propanol
chloromethane	propylene
cyclohexane	styrene
dibromochloromethane	1,1,2,2-tetrachloroethane
1,2-dichlorobenzene	tetrachloroethene
1,3-dichlorobenzene	tetrahydrofuran
1,4-dichlorobenzene	toluene
1,1-dichloroethane	1,2,4-trichlorobenzene
1,2-dichloroethane	1,1,1-trichloroethane
cis-1,2-dichloroethene	1,1,2-trichloroethane
trans-1,2-dichloroethene	trichloroethene
1,2-dichloropropane	1,2,4-trimethylbenzene
cis-1,3-dichloropropene	1,3,5-trimethylbenzene
trans-1,3-dichloropropene	vinyl acetate
1,4-dioxane	vinyl chloride
ethanol*	m-xylene
ethyl acetate	o-xylene
ethyl benzene	p-xylene
ethyl dibromide (1,1-dibromoethane)	

In nitrogen, 104 liters @ 1800psig

6-month stability

1ppm	100ppb
34436 (ea.)	34437 (ea.)

*Stability of this compound cannot be guaranteed.

cylinder design

Aluminum construction.

Size: 8 x 24 cm.

Volume/Pressure: 104 liters of gas @ 1800psig.

Outlet Fitting: CGA-180.

Weight: 1.5 lbs.



did you know?

Spectra Gas manufactures our high-quality air monitoring gas standards and is:

- Official supplier of PAMS (ozone precursor) calibration gas to US EPA.
- Only vendor of 62-component TO-15 gas standard.
- Rigorous quality control guarantees the stability and reproducibility of every Spectra Gases mix.

Passive Air Sampling Kits

Better Performance at a Better Value

- Improved design eliminates leaks at the filter.
- Siltek®-treated components ensure a very inert surface.
- Excellent for sampling times from 1 hour to 125 hours, or grab sampling.

Restek's passive air sampling kit incorporates all hardware necessary to collect air samples, and is easy to assemble for field sampling.* The improved filter design greatly reduces the number of potential leak sites.

The passive air sampling kit is available in six sampling flow ranges, and in stainless steel or Siltek® treated finish. The stainless steel kit is ideal to partner with the Restek TO-Can™ air sampling canister for TO-14A and TO-15 methods. Use the Siltek®-treated version with the Restek SilcoCan™ air sampling canister when collecting low-level volatile sulfur compounds, or other active compounds.

Air Sampling Kits

400cc	Canister Volume*/Sampling Time				Flow (sccm)	Orifice size	Siltek®-Treated Sampling Kits	Stainless Steel Sampling Kits
	1 Liter	3 Liter	6 Liter	15 Liter				
8 hour	24 hour	48 hour	125 hour	—	0.5–2	0.0008"	24217	24216
2 hour	4 hour	12 hour	24 hour	60 hour	2–4	0.0012"	24160	24165
1 hour	2 hour	6 hour	12 hour	30 hour	4–8	0.0016"	24161	24166
—	1 hour	4 hour	8 hour	20 hour	8–20	0.0020"	24162	24167
—	—	2 hour	3 hour	8 hour	20–40	0.0030"	24163	24168
—	—	—	1 hour	3 hour	40–80	0.0060"	24164	24169

*Air sampling canisters sold separately.

1. Veriflo™ SC423XL flow controller

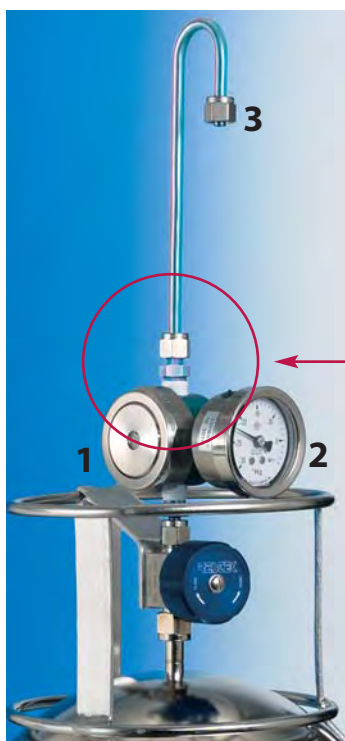
This flow controller is the heart of the sampling train. It is a high-quality device designed to maintain a constant mass flow as the pressure changes from 30" Hg to 5" Hg (we recommend you stop sampling at or before 5" Hg of vacuum). All wetted parts of the flow controller can be Siltek®-treated.

2. Stainless steel vacuum gauge

Fitted to the flow controller, the gauge monitors canister pressure change during sampling.

3. 1/4-inch Siltek® sample inlet

The 2m x 1/4-inch tubing includes a stainless steel nut on the inlet end, to prevent water droplets from accumulating at the edge of the tubing, where they could be pulled into the sampling train.



All fitting connections are 1/4" tube, except where noted.



4. 2-micron frit filter and washer

Located prior to the critical orifice to prevent airborne particles from clogging the critical orifice. Replaceable. Available in stainless steel, or Siltek®-treated for optimum inertness.

5. Interchangeable critical orifice

An interchangeable sapphire critical orifice allows you to control the flow with very high precision. To select the correct critical orifice for your sample, see table above. Available in stainless steel, or Siltek®-treated for optimum inertness.

a plus 1 story

"Restek has consistently provided high quality chromatography columns and supplies to me for well over a decade. Over the last two years, I have extensively been involved with air analysis, TO-15, etc., and Restek provides the highest quality canisters, mini-cans, and flow controllers in the market today."

Scott Van Etten, IH Laboratory Manager, EMSL Analytical

Buy only the parts you need!

Replacement Orifices

Use these orifices to change the flow range for alternative sampling times. Interchangeable with Veriflo™ 423XL orifices.

Flow (sccm)	Orifice size	Siltek®-Treated cat.#	Stainless Steel cat.#
0.5–2	0.0008"	24219	24218
2–4	0.0012"	24233	24245
4–8	0.0016"	24234	24246
8–20	0.0020"	24235	24247
20–40	0.0030"	24236	24248
40–80	0.0060"	24237	24249



Siltek®
critical orifice

2µm Frit Filters

For use in critical orifice fitting. Includes washers.

Description	qty.	cat.#
Siltek® Replacement Frit Filter	3-pk.	24171
Stainless Steel Replacement Frit Filter	3-pk.	24170



Veriflo™ Flow Controllers

Veriflo™ 423XL flow controllers are offered in a Siltek® and a stainless steel version, with or without a critical orifice. (Vacuum gauge sold separately.) The critical orifice is interchangeable. Order replacement orifices or orifices for alternate sampling times separately.

Flow (sccm)	Orifice size	Siltek®-Treated cat.#	Stainless Steel cat.#
0.5–2	0.0008"	24232	24229
2–4	0.0012"	24255	24260
4–8	0.0016"	24256	24261
8–20	0.0020"	24257	24262
20–40	0.0030"	24258	24263
40–80	0.0060"	24259	24264
—	no orifice	24238	24239

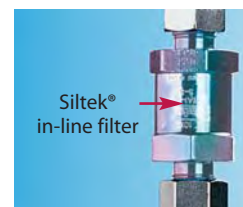


Veriflo SC423XL
flow controller

7µm In-Line Filter

This 316 stainless steel filter is designed to collect particles larger than 7 microns. We offer a Siltek® version and a stainless steel version.

Description	qty.	cat.#
Siltek® 7µm In-Line Filter	ea.	24265
Stainless Steel 7µm In-Line Filter	ea.	24266



Siltek®
in-line filter

Siltek® tee

2-Inch Vacuum Gauge

Restek's high-quality 2-inch vacuum gauge incorporates 316 stainless steel wetted surfaces.

Description	qty.	cat.#
2-Inch Vacuum Gauge; 1/8" NPT	ea.	24269
2-Inch Vacuum Gauge; 1/4" NPT	ea.	24270



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Analyzing Cleaning and Personal Care Products by Gas and Liquid Chromatography



Inside:

Product Types

Ingredients

Product Listing



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Introduction

The cleaning products industry is a multi-billion dollar industry.¹ The average consumer uses a wide range of products to promote both personal and public health. Of the products used in one's home, there are several basic categories: personal cleansing, laundry, dishwashing, and household cleaning. These products are designed to improve personal hygiene, reduce levels of microorganisms, and improve personal appearance.

As with all consumer products, there is a need to test both final products and raw materials. This helps to ensure consistent product performance, as well as personal and environmental safety. Quantifying individual components also is useful for optimizing the manufacturing process, for determining product shelf life, and for comparing competitive products.

Chromatographic techniques such as gas chromatography (GC) and high pressure liquid chromatography (HPLC) are powerful tools in the analysis of cleaning and personal care products. In this technical guide, we explore how GC and HPLC can be used to quantify components of these important products. For GC assays, general detectors, such as the flame ionization detector (FID), or more information-rich detectors, such as the mass spectrometer (MS), can be used. GC/MS is particularly useful for analyzing complex formulations, such as fragrance blends, and for identifying unknown components or contaminants. HPLC is applicable to a wide range of personal care product ingredients, such as antimicrobial agents, preservatives, and some surfactants. In general, UV-visible or light-scattering detectors can be used.

Product Types

Cleaning and personal care products can be categorized in a number of ways. The Soap and Detergent Association (SDA)² groups soaps and detergents into four general categories: personal cleansing, laundry, dishwashing, and household cleansing. Personal cleansing products include liquid and bar soaps, and heavy duty cleaners. Laundry detergents and laundry cleaning aids can be purchased in a variety of forms: powders, gels, liquids, sprays, and sheets. In addition to dirt and stain removal, they are used to bleach, soften, and freshen laundry. Dishwashing products also are marketed in a variety of forms: liquids, gels, and powders. Although they fall within the same category, hand dishwashing detergents and automatic dishwashing detergents generally have different formulations, as conditions for their use are quite different.

Household cleaners include a wide variety of products, as no single product will work well on all surfaces and soils. All-purpose cleaners are intended for general use, and can be used on a variety of surfaces, including various combinations of plastic, paint, metal, porcelain, glass, and wood. Specialty cleaners, for more specific applications, include products for glass, tubs and tile, ovens, toilet bowls,

or rugs and upholstery. Abrasive cleaners contain small mineral or metal particles for removing heavy soil loads from small areas. For unclogging kitchen and bathroom drains, drain openers incorporate caustic ingredients that generate heat to melt fatty deposits and chemicals that oxidize soil deposits.

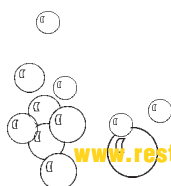
In addition to these cleaning products, a wide range of products promote personal hygiene: deodorants, mouthwashes, oral hygiene products, moisturizing lotions, and more. These products contain ingredients to cleanse, disinfect, deodorize, moisturize, and/or scent the user.

Basic Ingredients in Cleaning and Personal Care Products

Surfactants and Builders

The major components of cleaning products are surfactants and builders.¹

Surfactants (surface active agents) are used to reduce the surface tension of water, enabling the cleaning solution to more efficiently wet the surface to be cleaned. Without the surfactant, water's high surface tension causes it to bead on a surface, and cleaning is much more difficult. In addition, surfactants emulsify oils and other soils, and hold them in solution so they can be rinsed away.



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Surfactant-containing solutions can be applied to a wide variety of surfaces, including tile, ceramic, and cloth - and hair. Builders often are used to increase the effectiveness of a surfactant. Builders reduce water hardness by "tying up" hardness minerals, through chelation with the minerals or by forming an insoluble precipitate. Examples of builders/chelating agents include sodium citrate (the sodium salt of citric acid) and ethylenediamine tetraacetic acid (EDTA). Other builders, such as sodium carbonate, reduce water hardness by forming insoluble precipitates (e.g., calcium carbonate).

Surfactants generally are classified by their ionic properties in water. Anionic surfactants, such as alcohol ethoxylates, alkyl sulfates, and soaps, are negatively charged in solution. Anionic surfactants are used in laundry detergents and some dishwashing detergents, household clean-

ers, and personal cleaning products. Cationic surfactants, such as quaternary ammonium compounds, carry a positive charge in solution. They are used in products such as fabric softeners. Amphoteric surfactants, which can be either positively or negatively charged, often are used in personal cleansing products, due to their mildness. Nonionic surfactants, such as alcohol ethoxylates, are uncharged in solution; they are used in laundry detergents and automatic dishwasher detergents. An example analysis of a nonionic surfactant, Triton® X-100, an octylphenol ethylene oxide with an average of 9.5 ethylene oxide units per molecule, is shown in Figure 1. This surfactant can be analyzed by GC, using a nonpolar phase, such as MXT®-1.

As described above, soaps are anionic surfactants. Basically, soaps are sodium or potassium salts of fatty acids, produced by reacting animal or vegetable fats or oils with a strong alkali. The fat or oil, in its original form, consists primarily of triglycerides—three fatty acids attached to a glycerol backbone. After conversion to the soap—saponification—there is both a hydrophilic (car-

boxylate group) and a hydrophobic end (alkyl chain) to the molecule. Water, a polar molecule, can now interact with the hydrophilic alkyl chains, while the alkyl chain can interact with relatively non-polar surfaces such as countertops, tile, or skin.

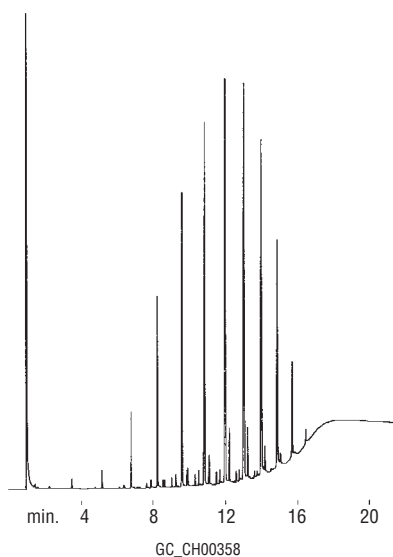
Fatty acids that make up a soap can be analyzed either in the free fatty acid form or after derivatization to the methyl esters (FAMES). Figure 2 shows an analysis of free fatty acids by GC, using a Stabilwax®-DA capillary column. The acid-deactivated phase in the Stabilwax®-DA column gives excellent peak shapes for free fatty acids. Figure 3 is an analysis of fatty acids as methyl esters, separated on an Rtx®-Wax column. FAMES also can be easily quantified by using a Stabilwax® column.

Solvents

Solvents are used primarily to dissolve organic soils. They also clean without leaving residue, making them very useful in products such as glass cleaners. The main criterion for cleaning product solvents is water miscibility, as the solvent must form a solution with the other water-soluble components. Alcohols and

Figure 1

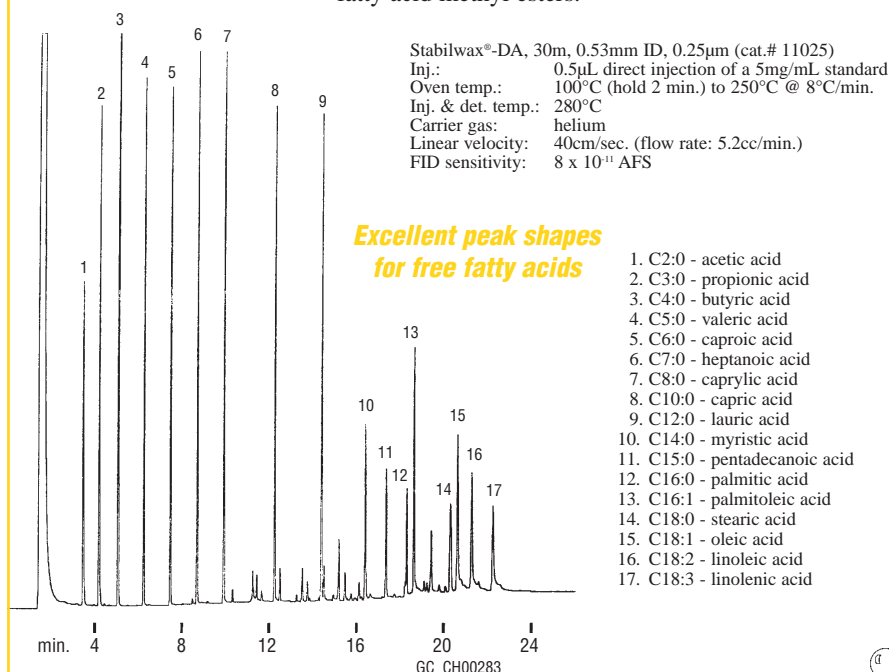
Triton® X-100 surfactant separated by number of ethylene oxide units on an MXT®-1 column.



MXT®-1, 30m, 0.28mm ID, 0.10µm (cat.# 70109)
Inj.: 1.0µL split injection of Triton® X-100 (40:1) in methylene chloride
Oven temp.: 150°C to 400°C @ 15°C/min. (hold 10 min.)
Inj. / det. temp.: 250°C / 400°C
Carrier gas: hydrogen
Linear velocity: 40cm/sec.
FID sensitivity: 102 x 10⁻¹¹ AFS

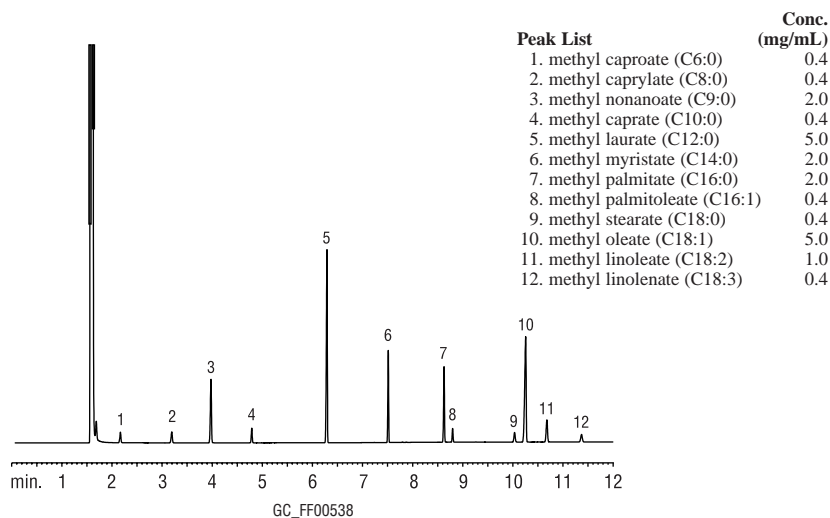
Figure 2

Free fatty acids analysis saves time and materials, relative to preparing and analyzing fatty acid methyl esters.



**Figure 3**

FAMES analysis on an Rtx®-Wax column.



Rtx®-Wax, 30m, 0.25mm, 0.25µm (cat.# 12423)

Inj.: 1µL split injection (100:1) of FAME standard; see peak list

Oven temp.: 120°C (hold 3 min.) to 220°C at 20°C/min. (hold 12 min.)

Inj./det. temp.: 250°C/300°C

Carrier gas: helium

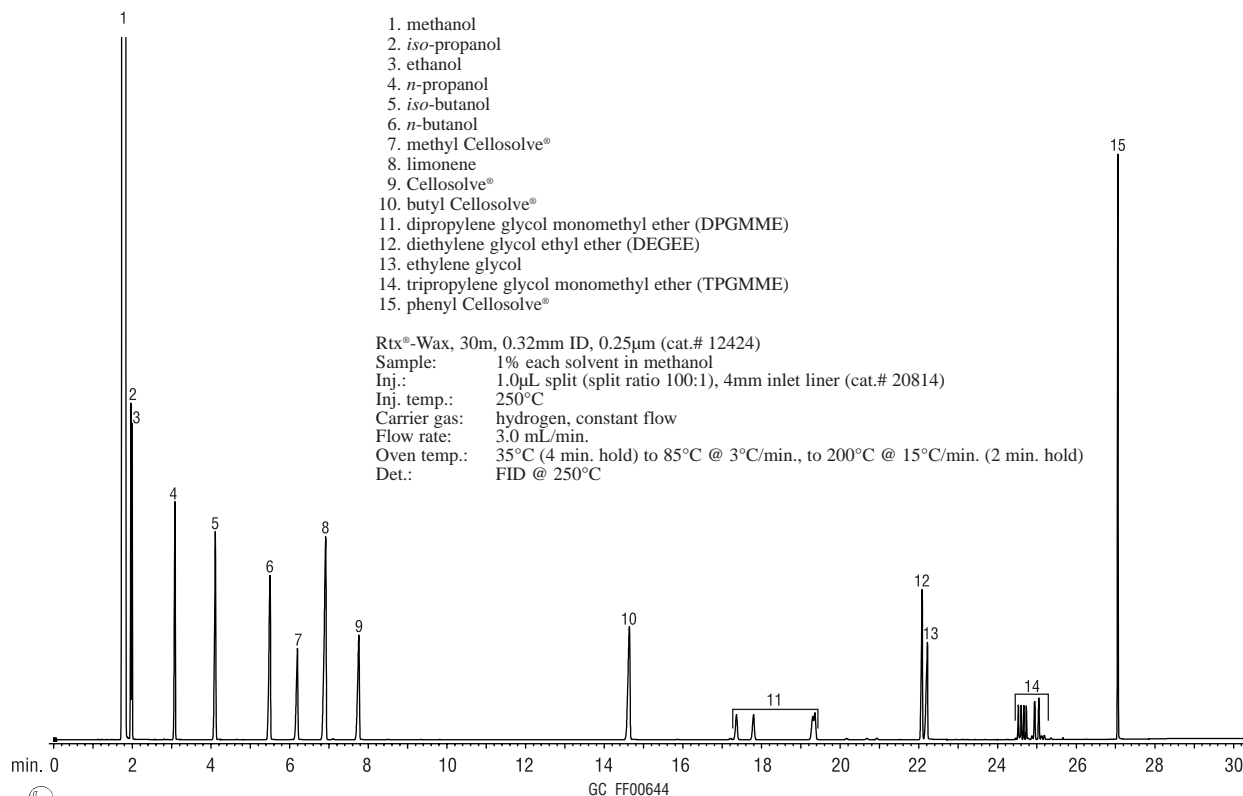
Linear velocity: 1mL/min. (34 cm/sec.)

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Figure 4

Alcohols, glycols, and other cleaning solvents can be quantified, using an Rtx®-Wax column.



Rtx®-Wax, 30m, 0.32mm ID, 0.25µm (cat.# 12424)

Sample: 1% each solvent in methanol

Inj.: 1.0µL split (split ratio 100:1), 4mm inlet liner (cat.# 20814)

Inj. temp.: 250°C

Carrier gas: hydrogen, constant flow

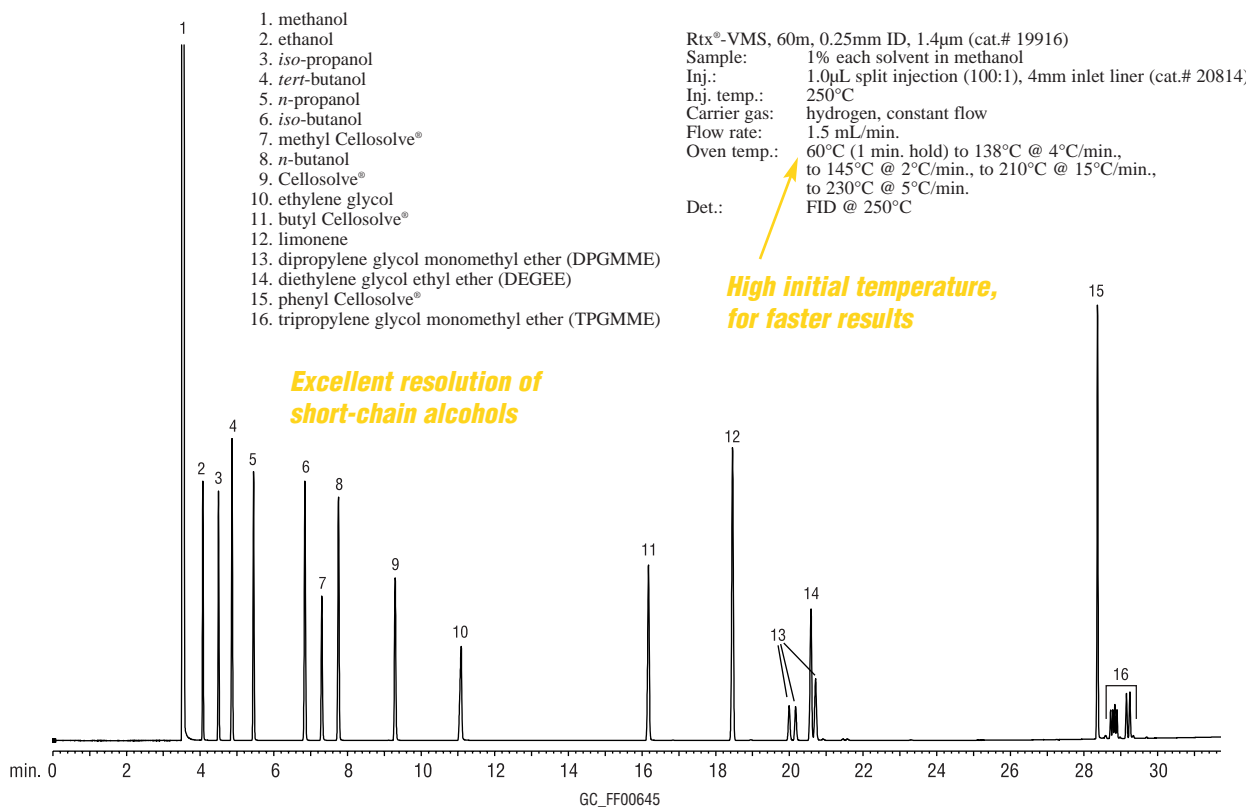
Flow rate: 3.0 mL/min.

Oven temp.: 35°C (4 min. hold) to 85°C @ 3°C/min., to 200°C @ 15°C/min. (2 min. hold)

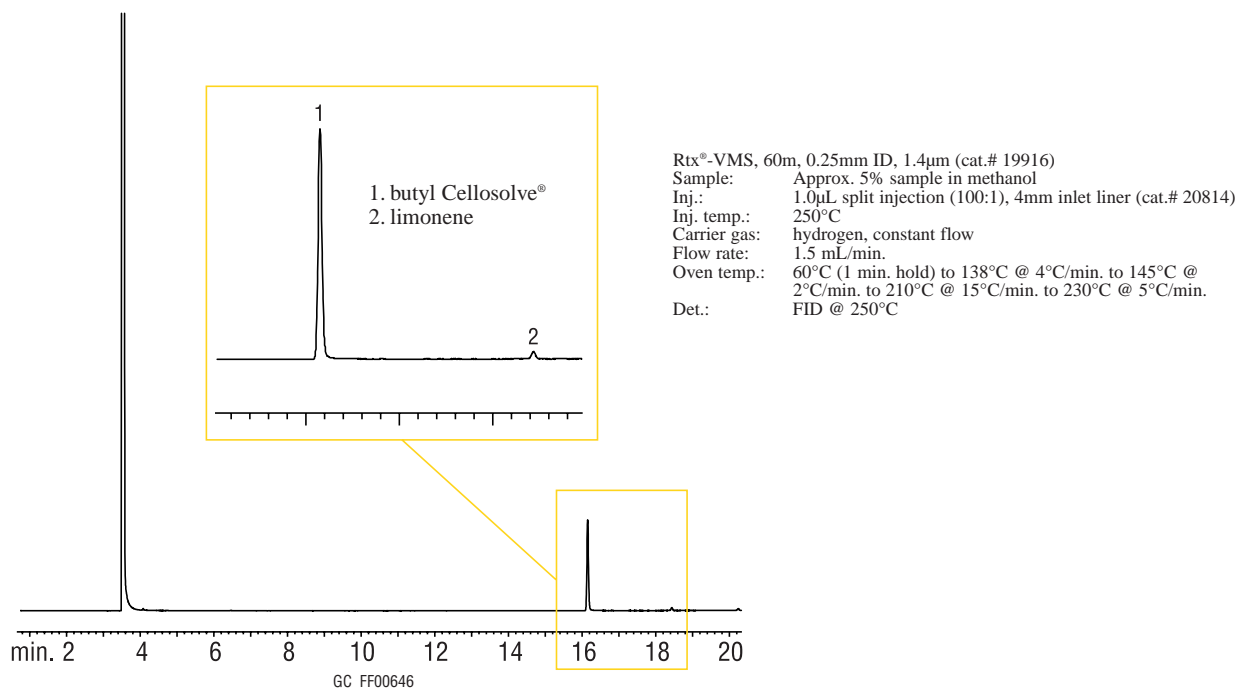
Det.: FID @ 250°C

Figure 5

Excellent, alternative selectivity for cleaning solvents, using an Rtx®-VMS column.

**Figure 6**

Quantify volatile ingredients in an all-purpose cleaner, using an Rtx®-VMS column.





glycols are popular choices. Cleaning solvents can be resolved using an Rtx®-Wax column (Figure 4) or an Rtx®-VMS column (Figure 5). The latter column gives excellent selectivity and peak shape for a wide range of cleaning solvents.

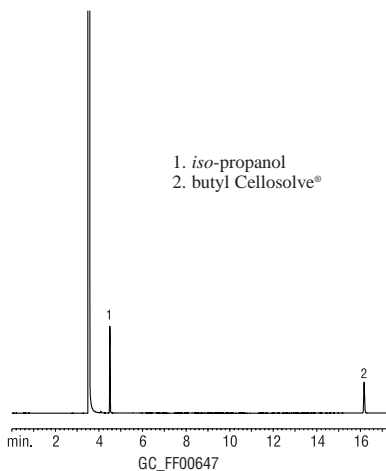
Analysis of an all-purpose cleaner is shown in Figure 6, and a glass cleaner is shown in Figure 7. Analyses of glycols and alcohols are shown in Figures 8–10.

Acids

Organic acids, such as acetic and citric acids, are used to reduce the pH of cleaning products, to remove mineral build-up. Inorganic acids, such as hydrochloric, phosphoric, and sulfuric acid also can be included in a formulation. Organic acids can be analyzed either by HPLC or by GC, but HPLC is a better technique for dicarboxylic acids. Figure 11 shows a separation of organic acids on an Ultra Aqueous C18 HPLC column. A GC analysis of short-chain free fatty acids is shown in Figure 12.

Figure 7

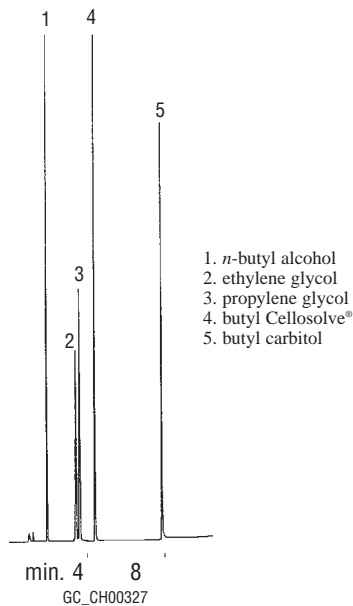
Glass cleaner on an Rtx®-VMS column.



Rtx®-VMS, 60m, 0.25mm ID, 1.4µm (cat.# 19916)
 Sample: Approx. 5% sample in methanol
 Inj.: 1.0µL split injection (100:1), 4mm inlet liner (cat.# 20814)
 Inj. temp.: 250°C
 Carrier gas: hydrogen, constant flow
 Flow rate: 1.5 mL/min.
 Oven temp.: 60°C (1 min. hold) to 138°C @ 4°C/min. to 145°C @ 2°C/min. to 210°C @ 15°C/min. to 230°C @ 5°C/min.
 Det.: FID @ 250°C

Figure 8

Glycols and alcohols on an ultra-low-bleed column.

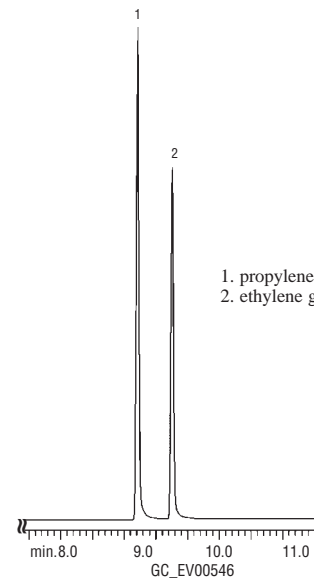


1. *n*-butyl alcohol
 2. ethylene glycol
 3. propylene glycol
 4. butyl Cellosolve®
 5. butyl carbitol

XTI®-5, 30m, 0.53mm ID, 1.0µm (cat.# 12255)
 Inj.: 1.0µL direct injection of glycols and alcohols, 100ppm each (hold 5 min.)
 Oven temp.: 40°C to 185°C @ 15°C/min.
 Inj. / det. temp.: 150°C / 200°C
 Carrier gas: helium
 Linear velocity: 40cm/sec. (flow rate: 5cc/min.)
 FID sensitivity: 8×10^{-11} AFS

Figure 9

Glycols on a Stabilwax® column.

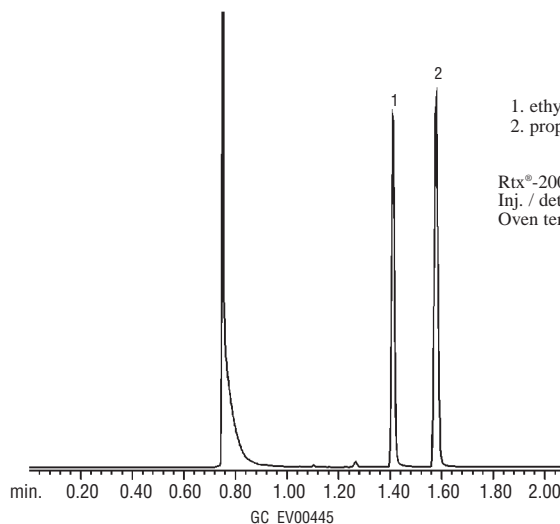


1. propylene glycol
 2. ethylene glycol

Stabilwax®, 30m, 0.53mm ID, 1.0µm (cat.# 10655)
 Inj.: 1.0µL direct injection of ethylene glycol and propylene glycol, 100 ppm each, in water. Open-top Uniliner® direct injection liner without wool (cat.# 20843-205).
 Septum purge: 5.0cc/min.
 Oven temp.: 80°C (hold 1 min.) to 200°C @ 8°C/min. (hold 5 min.)
 Inj./det. temp.: 225°C/250°C
 Carrier gas: helium
 Linear velocity: 50cm/sec.
 Detector: FID

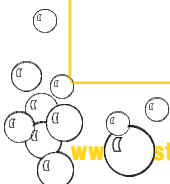
Figure 10

Glycols on a uniquely selective trifluoropropyl phase column.



1. ethylene glycol
 2. propylene glycol

Rtx®-200, 30m, 0.53mm ID, 1.0µm (cat.# 15055)
 Inj. / det. temp.: 220°C / 270°C
 Oven temp.: 80°C (hold 1 min.) to 200°C @ 8°C/min. (hold 3 min.)
 10psi pressure



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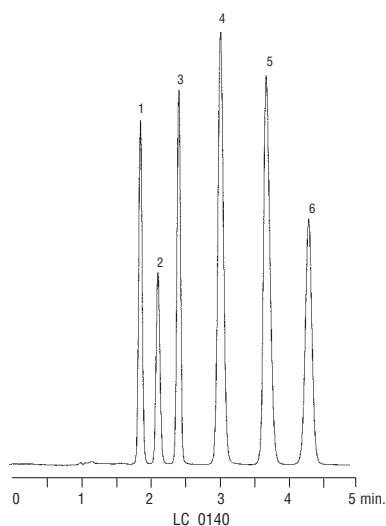
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Figure 11

Organic acids on an Ultra Aqueous C18 HPLC column.



Peak List:	Conc. (µg/mL)
1. malonic acid	500
2. lactic acid	500
3. acetic acid	1000
4. citric acid	1000
5. succinic acid	2000
6. fumaric acid	10

Sample:

Solvent: HPLC-grade water
Inj.: 10µL

Column: Ultra Aqueous C18

Catalog #: 9178565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:

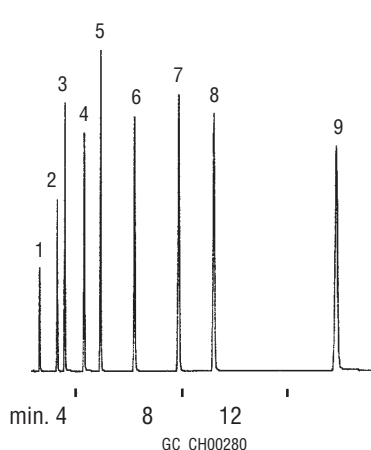
Mobile phase: 50mM potassium phosphate, pH 2.5; acetonitrile (99:1)
Flow: 1.5mL/min.
Temp.: 25°C
Det.: UV @ 210nm

HOT tech tip

The Ultra Aqueous C18 column is an excellent choice when using highly aqueous mobile phases. Embedded polar groups prevent collapse of the alkyl chains—even in 100% aqueous environments. See page 12 for more information.

Figure 12

Organic Acids on a Stabilwax®-DA column.



1. acetic acid
2. propionic acid
3. isobutyric acid
4. *n*-butyric acid
5. isovaleric acid
6. *n*-valeric acid
7. isocaproic acid
8. caproic acid
9. heptanoic acid

Stabilwax®-DA, 30m, 0.25mm ID, 0.25µm
(cat.# 11023)

Inj.: 1.0µL split injection (50:1) of a free acid standard, approximately 10 to 20ng/µL each analyte

Oven temp.: 145°C
Inj. & det. temp.: 250°C
Carrier gas: hydrogen
Linear velocity: 40cm/sec.
FID sensitivity: 2 x 10⁻¹¹ AFS

Alkalis

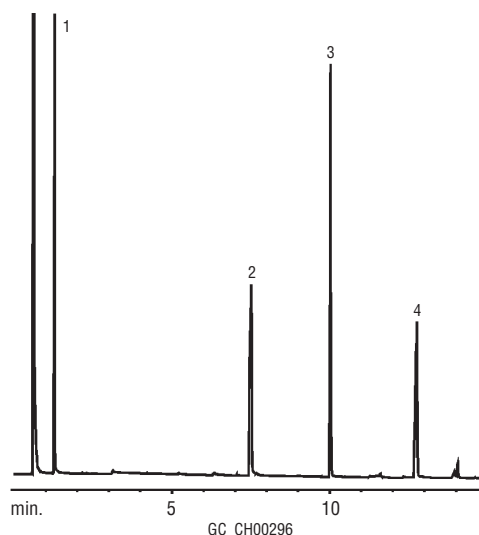
Products with higher pH are useful for dissolving fatty or oily soils. Alkalis, or bases, are used to neutralize acidic ingredients, or to raise the pH. Suitable alkalis include ethanolamines, ammonium hydroxide, and sodium silicate. The more basic compounds, such as ethanolamines, can be analyzed by GC, but a base-deactivated column should be used. Ethanolamines analysis on an Rtx®-5 Amine capillary GC column is shown in Figure 13.

Antimicrobial Agents

Antimicrobial agents are included in soaps, detergents, health and skincare products, and household cleaners. By controlling microbial growth, they control disease and odor. More than 300 active ingredients currently are used to control microorganisms.³ These agents can be categorized as sterilizers, disinfectants, sanitizers, or antiseptics/germicides. Sterilizers are used to eliminate fungi, viruses, and bacteria; disinfectants, to destroy or control fungi and bacteria, but not necessarily their spores; sanitizers, to reduce microorganisms on surfaces. Antiseptics or germicides are used on living people and animals. In the United States, a product used in or on the body, or in processed food, is regu-

Figure 13

Ethanolamines on a base-deactivated column.



1. monoethanolamine
2. diethanolamine
3. triethylene glycol monomethylether (IS)
4. triethanolamine

Rtx®-5 Amine, 15m, 0.25mm ID, 0.50µm
(cat.# 12335)

Inj.: 1.0µL split injection (58:1) of ethanolamines in methanol; on-column conc. 34ng each analyte
Oven temp.: 50°C (hold 2 min.) to 180°C @ 10°C/min. (hold 2 min.)

Inj. & det. temp.: 280°C / 300°C
Carrier gas: hydrogen
Linear velocity: 43cm/sec. set @ 50°C
FID sensitivity: 6.4 x 10⁻¹¹ AFS



lated by the Food and Drug Administration (FDA). Other products fall under the guidelines of the US Environmental Protection Agency (EPA). Examples of antimicrobial agents are: quaternary ammonium compounds, sodium hypochlorite, organic acids, alcohols, iodine, Triclosan, and 4-chloro-3,5-dimethylphenol (*para*-chloro-*meta*-xylenol/PCMX). A PCMX assay by HPLC is shown in Figure 14; Figure 15 demonstrates the separation of benzoic and sorbic acids on an Ultra Phenyl HPLC column.

Preservatives

Preservatives are used to extend product shelf life. Examples of preservatives used in cleaning and personal care products are BHT (3,5-di-*tert*-butyl-4-hydroxytoluene), BHA (2- & 3- *tert*-butyl-4-hydroxyanisole), EDTA (ethylenediamine tetraacetic acid), and glutaraldehyde. BHT and BHA are phenolic antioxidants that can be very effective, even at low concentrations. These compounds can be analyzed either by GC (Figure 16) or by HPLC (Figure 17).

Figure 14

PCMX in hand soap on a Pinnacle™ DB C18 HPLC column.

Peak List:

1. PCMX (4-chloro-3,5-dimethylphenol)

Column:

Pinnacle™ DB C18
Catalog #: 9414565
Dimensions: 150 x 4.6mm
Particle Size: 5µm
Pore Size: 140Å

Conditions:

Mobile Phase: water:methanol (35:65 v/v)
Flow: 1.0mL/min.
Temp.: ambient
Det.: UV @ 280nm

Sample:

Inj.: 10µL
Conc.: 5% solution of hand soap in methanol
Sample Diluent: methanol
Sample Temp.: ambient

LC_0293

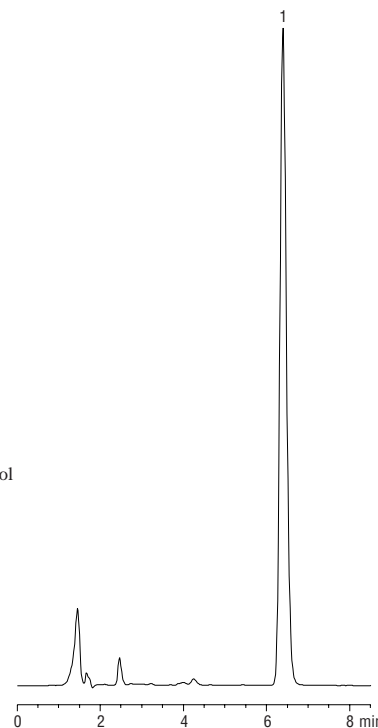


Figure 15

Resolution and symmetric peaks for sorbic and benzoic acids on an Ultra Phenyl HPLC column.

1. sorbic acid
2. benzoic acid

Sample:

Inj.: 10µL
Conc.: 100 ppm sorbic acid,
200 ppm benzoic acid
Solvent: mobile phase

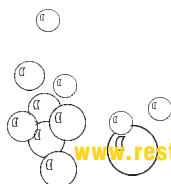
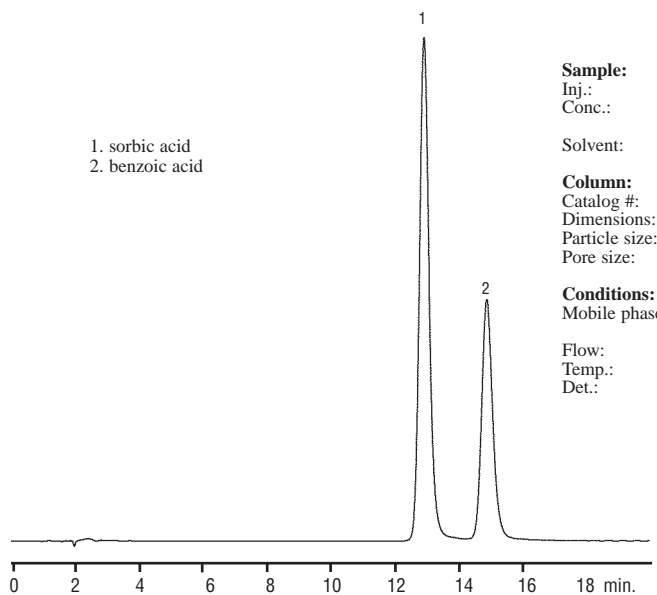
Column:

Ultra Phenyl
Catalog #: 9105565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:

Mobile phase: 1% acetic acid:methanol
(80:20, v/v)
Flow: 1.2 mL/min.
Temp.: ambient
Det.: UV @ 245nm

LC_0150



www.restekcorp.com

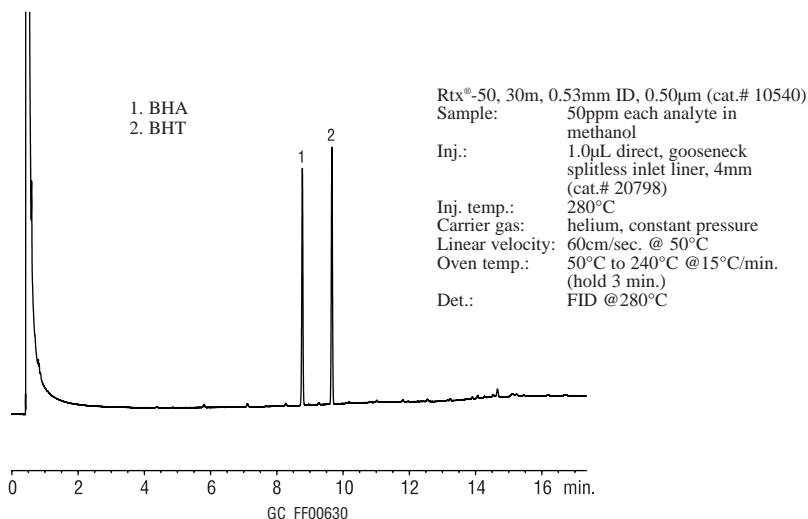
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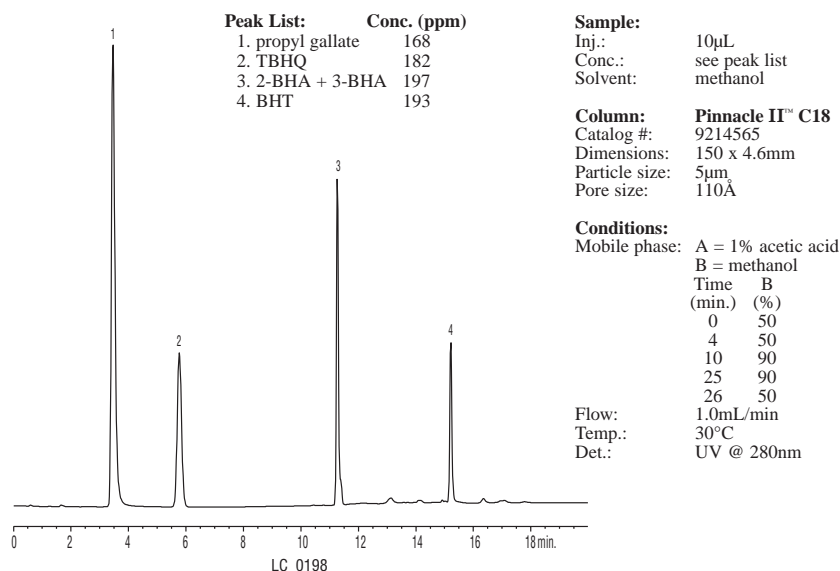
Website NEW : www.chromalytic.com.au E-mail : info@chromtech.net.au Tel: 03 9762 2034 . . . in AUSTRALIA

Figure 16

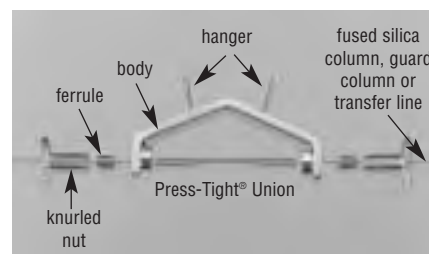
Preservatives BHA and BHT on an Rtx®-50 intermediate polarity column.

**Figure 17**

Phenolic preservatives, including BHA and BHT, on a Pinnacle II™ C18 HPLC column.

**Secure, Reliable Column-to-Column Connections***Use a Vu2 Union™ connector when you:*

- Connect a guard column to an analytical column.
- Connect a column to a transfer line or restrictor line.
- Connect two columns in a series.
- Repair a broken column.



The Vu2 Union™ connector's open design allows visual confirmation of the seal; secondary seals ensure a leak-tight connection.

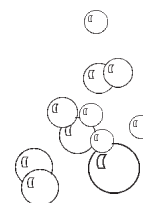
Vu2 Union™ Connector Kits

Kits include: Vu2 Union™ body, 2 knurled nuts, 2 Press-Tight® unions, and 4 ferrules

Fits Column ID	qty.	cat.#
0.15–0.25mm	kit	21105
0.28/0.32mm	kit	21106
0.45/0.50 & 0.53mm	kit	21107
Knurled nut	2-pk.	21108

Questions?

Contact Restek's Technical Service Team! We have answers to your toughest analytical questions. Call 800-356-1688 or 814-353-1300, ext. 4, email us at support@restekcorp.com or contact your local Restek representative.



Fragrances & Colorants

Fragrances and colorants give a unique look or scent to a product. Blue dyes or pigments—bluing agents—absorb in the yellow region of the spectrum, masking age- and use-associated yellowing of clothing and bedding, and making these articles look brighter. Colorants also make a product “seen” in use, as in toilet bowl cleaners and floor sanitizers. Fragrances disguise odors from soils, or from the product itself, as well as provide the desired scent. In general, GC is effective for monitoring or identifying fragrance components. Examples of fragrance assays by GC are shown in Figures 18–20.

Figure 18

Personal care product fragrance compounds on an Rtx®-1 column.

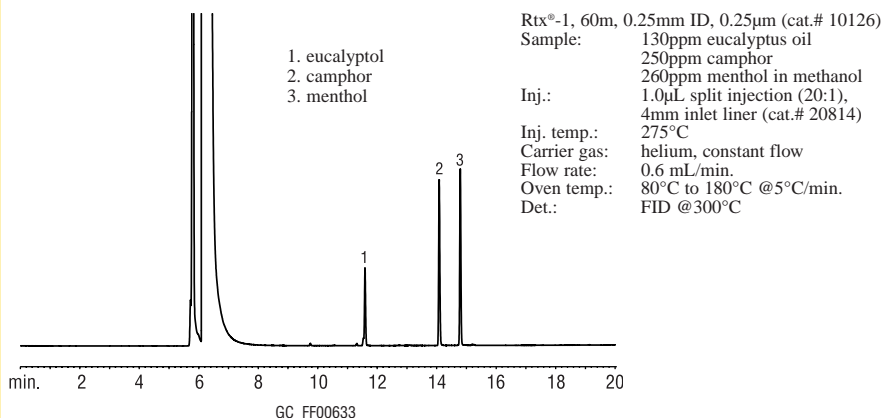


Figure 19

A complex fragrance, lemon oil, resolved on an Rtx®-5 column.

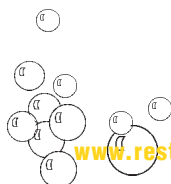
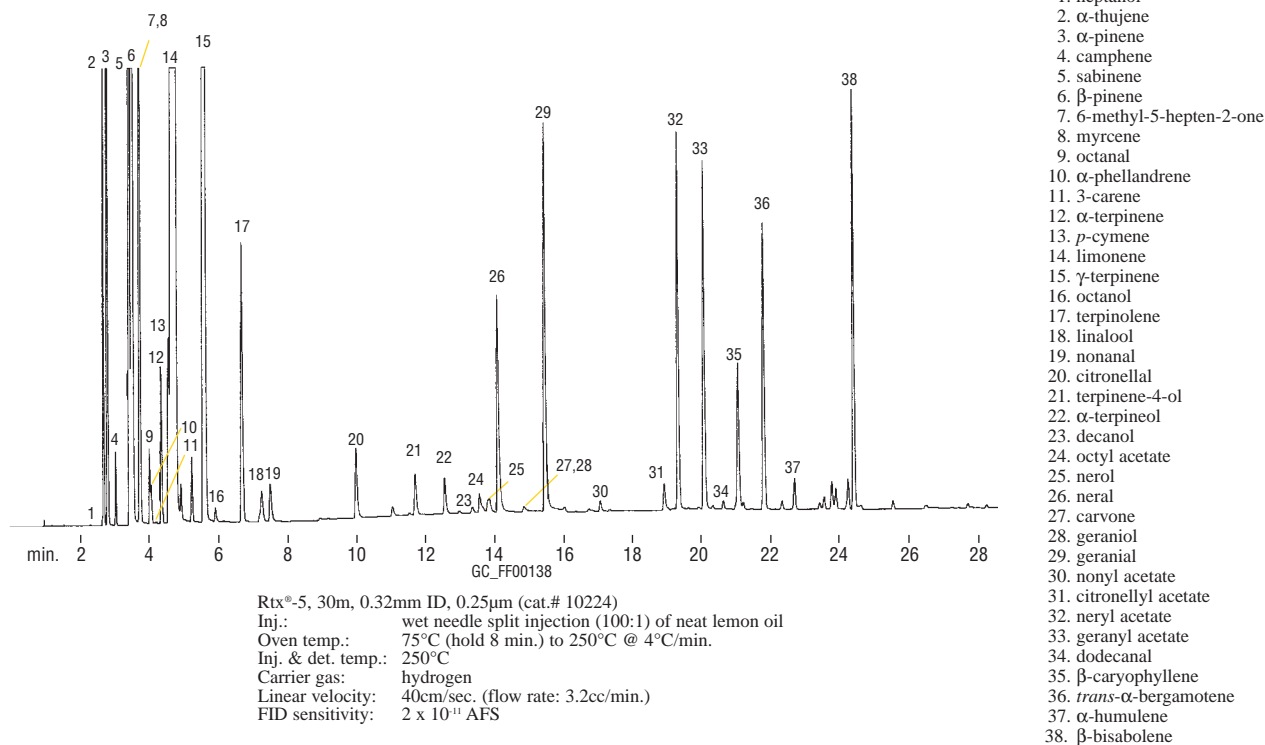
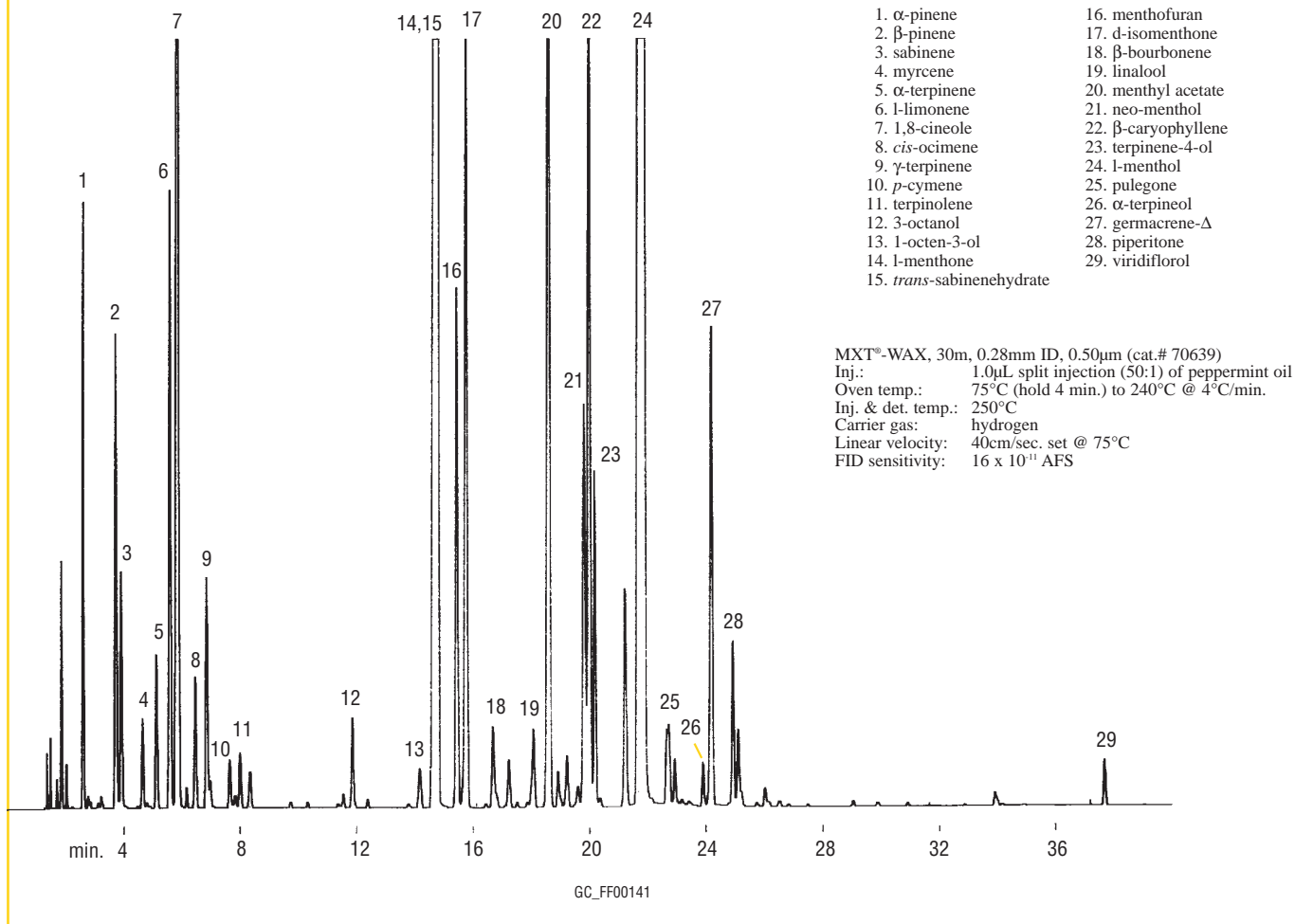


Figure 20

Peppermint oil components resolved on an MXT®-WAX column.



Miscellaneous Ingredients

Other ingredients used in cleaning, sanitizing, and personal care products include abrasives, such as quartz or sand; anti-redeposition agents, such as carboxymethylcellulose, that prevent soils from resettling on cleaned surfaces; bleach (e.g., sodium hypochlorite), for whitening and stain removal; enzymes, for removing specific soils, such as proteins; and fabric softeners, such as quaternary ammonium compounds.

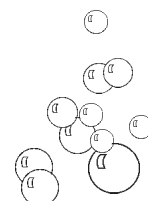
Summary

A wide and disparate list of ingredients is used in cleaning and personal care products, to solubilize soils, wet surfaces, mask odors, or perform a variety of other functions. Gas chromatography and liquid chromatography are used to

monitor specific components, to ensure product quality. Restek chromatographic columns and supplies ensure peak performance of these chromatographic assays. For assistance with your specific applications, please call Restek's Technical Service Team at 800-356-1688 or 814-353-1300, ext. 4, or email us at support@restekcorp.com. We will be happy to work with you.

References

1. Branna, Tom *The I&I Market* in *Happi*, Nov. 2000.
2. The Soap and Detergent Association. www.sdahq.org
3. US Environmental Protection Agency. www.epa.gov/pesticides/citizens/antimic.htm



HPLC Columns



For a complete listing of our HPLC columns and accessories, request our HPLC catalog (lit. cat.# 59241A), or visit our website.

PEEK® Fitting Extractor

Drill into the broken fitting, then screw the extractor into the fitting and remove it easily.

cat.# 25325, (ea.)



Sonic Debubbler

Just touch the Sonic Debubbler to the inlet line or check valve—ultrasonic vibrations will quickly dislodge or redissolve trapped air bubbles. Reduces downtime or conversion time from one mobile phase to another.

cat.# 20444, (ea.)



Ultra Phenyl 5µm Columns (USP L11)

Physical Characteristics:

particle: 5µm spherical fully end-capped pore size: 100Å
pH range: 2.5 to 7.5 carbon load: 10% temperature limit: 80°C

Chromatographic Properties:

High-purity, highly retentive, base-deactivated phase with alternative selectivity to hydrocarbon phases, especially for aromatic analytes.

	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
Length	cat.#	cat.#	cat.#	cat.#
30mm	9105531	9105532	9105533	9105535
50mm	9105551	9105552	9105553	9105555
100mm	9105511	9105512	9105513	9105515
150mm	9105561	9105562	9105563	9105565
200mm	9105521	9105522	9105523	9105525
250mm	9105571	9105572	9105573	9105575

Ultra Aqueous C18 5µm Columns (USP L1)

Physical Characteristics:

particle: 5µm spherical not end-capped pore size: 100Å
pH range: 2.5 to 7.5 temperature limit: 80°C

Chromatographic Properties:

Highly retentive and selective for reversed phase separations of polar analytes. Highly base deactivated. Compatible with highly aqueous (up to 100%) mobile phases.

	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
Length	cat.#	cat.#	cat.#	cat.#
30mm	9178531	9178532	9178533	9178535
50mm	9178551	9178552	9178553	9178555
100mm	9178511	9178512	9178513	9178515
150mm	9178561	9178562	9178563	9178565
200mm	9178521	9178522	9178523	9178525
250mm	9178571	9178572	9178573	9178575

Pinnacle II™ C18 5µm Columns (USP L1)

Physical Characteristics:

particle: 5µm spherical fully end-capped pore size: 110Å
pH range: 2.5 to 7.5 carbon load: 13% temperature limit: 80°C

Chromatographic Properties:

Excellent choice as a general purpose C18 column. Intermediate carbon loading and surface area, suitable for a wide range of neutral hydrophobic compounds.

	1.0mm ID	2.1mm ID	3.2mm ID	4.0mm ID	4.6mm ID
Length	cat.#	cat.#	cat.#	cat.#	cat.#
30mm	9214531	9214532	9214533	—	9214535
50mm	9214551	9214552	9214553	—	9214555
100mm	9214511	9214512	9214513	9214514	9214515
150mm	9214561	9214562	9214563	9214564	9214565
200mm	9214521	9214522	9214523	—	9214525
250mm	9214571	9214572	9214573	—	9214575

Pinnacle™ DB C18 5µm Columns (USP L1)

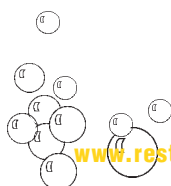
Physical Characteristics:

particle: 5µm spherical endcap: yes pore size: 140Å
pH range: 2.5 - 7.5 carbon load: 11% temperature limit: 80°C

Chromatographic Properties:

Highly base-deactivated spherical silica manufactured by Restek Corp. Monomeric C18 bonding. Hydrophobic C18 phase suitable for analyses of a wide range of compounds, from acidic through slightly basic. Replaces Hypersil® BDS C18.

	1.0mm ID	2.1mm ID	3.2mm ID	4.6mm ID
Length	cat.#	cat.#	cat.#	cat.#
30mm	9414531	9414532	9414533	9414535
50mm	9414551	9414552	9414553	9414555
100mm	9414511	9414512	9414513	9414515
150mm	9414561	9414562	9414563	9414565
200mm	9414521	9414522	9414523	9414525
250mm	9414571	9414572	9414573	9414575



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Restek's Trident™ Integral System

- Convenient and economical leak-free guard column system, extremely easy to install.
- Versatile configuration protects against all levels of contamination.
- Integral design eliminates troublesome tubing connections.

The system's foundation consists of the analytical column configured with our exclusive Trident™ end fitting and XF fitting. This configuration contains the standard internal frit as well as a replaceable cap frit, which can be easily changed without disturbing the packed bed. Changing the external frit can reverse the effects of accumulated particles, such as high backpressure or peak distortion. To obtain this basic configuration, simply order any Restek HPLC column, and add the suffix -700 to the catalog number.

For maximum protection against contaminants and particulate matter, the system can be configured with an integral guard cartridge holder (XG-XF), a guard cartridge, and a replaceable external frit. To obtain this configuration, simply order any Restek HPLC column, add the suffix -700 to the catalog number, and order the appropriate XG-XF male fitting.

Description	qty.	cat.#
XG-XF Fitting for 1cm Guard Cartridge	ea.	25026
XG-XF Fitting for 2cm Guard Cartridge	ea.	25062
Replacement XF Filter Fitting	ea.	25024
Replacement cap frits: 4mm, 2.0µm	5-pk.	25022
Replacement cap frits: 4mm, 0.5µm	5-pk.	25023
Replacement cap frits: 2mm, 2.0µm	5-pk.	25057

Trident™ Direct

Easy-to-Use, Low-Dead Volume—The Ultimate Combination of Convenience and Column Protection

Description	qty.	cat.#
High-pressure filter	ea.	25082
1cm guard cartridge holder without filter	ea.	25083
1cm guard cartridge holder with filter	ea.	25084
2cm guard cartridge holder without filter	ea.	25085
2cm guard cartridge holder with filter	ea.	25086
Connection tip for Waters®-style end fittings	ea.	25088
PEEK® tip standard fittings	ea.	25087

Trident™ HPLC Guard Column Cartridges

Guard Column Cartridges	3-pk. (10 x 2.1mm)	3-pk. (10 x 4.0mm)	2-pk. (20 x 2.1mm)	2-pk. (20 x 4.0mm)
Pinnacle II™ C18	921450212	921450210	921450222	921450220
Pinnacle™ DB C18	941450212	941450210	941450222	941450220
Ultra Aqueous C18	917850212	917850210	917850222	917850220
Ultra Phenyl	910550212	910550210	910550222	910550220

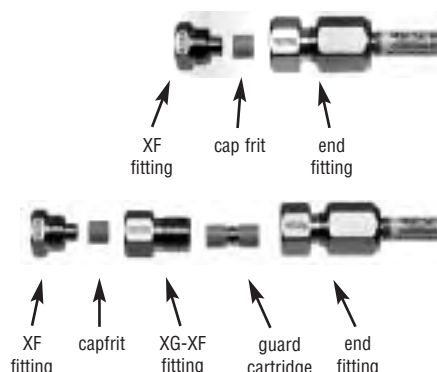
Trident™ HPLC In-Line Guard Cartridge Holders

A Trident™ in-line guard cartridge holder can be used with almost any HPLC column by connecting it with a short piece of 1/16" tubing, appropriate nuts and ferrules, or finger-tight fittings. The system can be used with Restek columns or columns from other manufacturers. Holders are available for either 1 or 2cm guard cartridges. Either size can be purchased with or without a prefilter, which provides added protection against the particles that can shorten the lifetime of the guard cartridge.

Description	qty.	cat.#
Holder for 1cm guard cartridge	ea.	25021
Holder with filter for 1cm guard cartridge	ea.	25040
Holder for 2cm guard cartridge	ea.	25061
Holder with filter for 2cm guard cartridge	ea.	25060
Replacement cap frits: 4mm, 2.0µm	5-pk.	25022
Replacement cap frits: 4mm, 0.5µm	5-pk.	25023
Replacement cap frits: 2mm, 2.0µm	5-pk.	25057

*The standard PEEK® tip in Trident™ Direct systems is compatible with Parker®, Upchurch®, Valco®, and other CPI-style fittings. To use Trident™ Direct systems with Waters®-style end fittings, the tip must be replaced with cat.# 25088.

Guard Column Systems



Trident™ Direct provides three levels of protection



Trident™ Direct high-pressure filter
Protection against particulate matter.



Trident™ Direct 1cm guard cartridge holder with filter
Protection against particulate matter and moderate protection against irreversibly adsorbed compounds.



Trident™ Direct 2cm guard cartridge holder with filter
Protection against particulate matter and maximum protection against irreversibly adsorbed compounds.



Holder for 1cm guard cartridge



Holder with filter for 1cm guard cartridge



Holder for 2cm guard cartridge



Holder with filter for 2cm guard cartridge

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GC Columns



For a complete listing of
our GC columns, request
our annual
Chromatography Products
Guide (lit. cat.# 59473),
or visit our website.

Rtx®-1 Columns

(Crossbond® 100% dimethyl polysiloxane)
temp. limits: -60 to 330/350°C

length	ID (mm)	df (µm)	cat.#
30m	0.25	0.25	10123
30m	0.32	0.25	10124
60m	0.25	0.25	10126
60m	0.32	0.25	10127

Rtx®-5 Columns

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)
temp. limits: -60 to 320/340°C

length	ID (mm)	df (µm)	cat.#
30m	0.25	0.25	10223
30m	0.32	0.25	10224
30m	0.25	0.50	10238
30m	0.32	0.50	10239

XTI®-5 Columns

(Crossbond® 5% phenyl - extended temp. and inertness)
temp. limits: -60 to 330/350°C

length	ID (mm)	df (µm)	cat.#
30m	0.25	0.50	12238
30m	0.32	0.50	12239
30m	0.53	0.50	12240*
30m	0.53	1.0	12255**

* temp. limits: -60 to 330/360°C

** temp. limits: -60 to 325/350°C

Rtx®-5 Amine Columns

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)
temp. limits: -60 to 300/315°C

length	ID (mm)	df (µm)	cat.#
15m	0.25	0.50	12335
30m	0.25	0.50	12338
15m	0.25	1.0	12350
30m	0.25	1.0	12353

Rtx®-50 Columns

(Crossbond® 50% methyl/50% phenyl polysiloxane)
temp. limits: 0 to 300/320°C

length	ID (mm)	df (µm)	cat.#
30m	0.25	0.50	10538
30m	0.32	0.50	10539
30m	0.53	0.50	10540*

*temp. limits: 0 to 270/290°C

Rtx®-200 Columns

(Crossbond® trifluoropropylmethyl polysiloxane)
temp. limits: -20 to 290/310°C

length	ID (mm)	df (µm)	cat.#
30m	0.25	1.0	15053
30m	0.32	1.0	15054
30m	0.53	1.0	15055*

*temp. limits: 0 to 270/290°C

Stabilwax® Columns

(Crossbond® Carbowax® - provides oxidation resistance)
temp. limits: 40 to 250°C

length	ID (mm)	df (µm)	cat.#
30m	0.25	0.25	10623
30m	0.32	0.25	10624
30m	0.53	0.25	10625
30m	0.32	1.0	10654*
30m	0.53	1.0	10655*

*temp. limits: 40 to 240/250°C

Rtx®-WAX Columns

(Crossbond® polyethylene glycol)
temp. limits: 20 to 250°C

length	ID (mm)	df (µm)	cat.#
30m	0.25	0.25	12423
30m	0.32	0.25	12424
30m	0.25	0.50	12438
30m	0.32	0.50	12439

Stabilwax®-DA Columns

(Crossbond® Carbowax® for acidic samples)
temp. limits: 40 to 250°C

length	ID (mm)	df (µm)	cat.#
30m	0.25	0.25	11023
30m	0.32	0.25	11024
30m	0.53	0.25	11025
30m	0.25	0.50	11038
30m	0.32	0.50	11039
30m	0.53	0.50	11040

Rtx®-VMS Columns

temp. limits: -40 to 240/260°C

length	ID (mm)	df (µm)	cat.#
30m	0.25	1.4	19915
30m	0.32	1.8	19919
30m	0.53	3.0	19985
60m	0.25	1.4	19916
60m	0.32	1.8	19920
75m	0.53	3.0	19974

MXT®-1 Columns

Silcosteel®-treated metal column
(Crossbond® 100% dimethyl polysiloxane)
temp. limits: -60 to 360°C

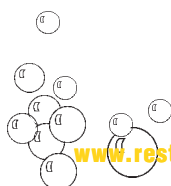
length	ID (mm)	df (µm)	cat.#
15m	0.28	0.1	70106
30m	0.28	0.1	70109
15m	0.28	0.25	70121
30m	0.28	0.25	70124

MXT®-WAX Columns

Silcosteel®-treated metal column
(Crossbond® polyethylene glycol)
temp. limits: 20 to 250°C

length	ID (mm)	df (µm)	cat.#
30m	0.28	0.25	70624
30m	0.28	0.50	70639
30m	0.28	1.0	70654*

*temp limits: 40 to 240°C



Fragrance Materials Association Test Mix

Use this mix in essential oils analysis, to aid in the detection of inlet problems, stationary phase degradation, loss of resolution, changes in sensitivity, and the presence of reactive sites in the sample pathway. The required 5% test solution can be conveniently made by diluting the entire 0.5mL of neat mixture to 10mL with acetone.

benzyl salicylate	362 parts
cinnamic aldehyde	5 parts
cinnamic alcohol	3 parts
cinnamyl acetate	3 parts
ethyl butyrate	362 parts
eucalyptol	5 parts
geraniol	6 parts
hydroxycitronellal	50 parts
d-limonene	200 parts
thymol crystal	3 parts
vanillin	1 part
benzoic acid	1% of mix

Neat, 0.5mL in an amber ampul

Each	5-pk.	10-pk.
31807	31807-510	—

AOCS #1 Mix

Chain Compound	%
16:0 methyl palmitate	6.0
18:0 methyl stearate	3.0
18:1 methyl oleate	35.0
18:2 methyl linoleate	50.0
18:3 methyl linolenate	3.0
20:0 methyl arachidate	3.0

Composition listed as a weight/weight % basis.

Each
35022

FAME #15 Mix

Chain Compound	%
16:0 methyl palmitate	10.0
18:0 methyl stearate	3.0
18:1 methyl oleate	50.0
18:2 methyl linoleate	30.0
20:0 methyl arachidate	1.5
20:1 methyl eicosenoate	1.5
22:0 methyl behenate	3.0
24:0 methyl lignocerate	1.0

Composition listed as a weight/weight % basis.

Each
35036

Ethylene Oxide Standard

ethylene oxide

500µg/mL in dimethylsulfoxide, 1mL/ampul

Each	10-pk.
36005	36105

USP 467 Calibration Mixture #4

Meets guidelines in USP25/NF20, effective January 2002.

benzene	2µg/mL
chloroform	60
1,4-dioxane	380
methylene chloride	600
trichloroethene	80

Prepared in methanol, 1mL/ampul

Each	10-pk.
36006	36106

ASTM D6042-96 Calibration Mix

This mixture contains the common antioxidants and slips listed in American Society for Testing and Materials (ASTM International) Method D6042-96.

BHT	Irganox® 3114
erucamide slip	Irganox® 1010
vitamin E	Irganox® 1076
Irgafos® 168	

50µg/mL each in isopropanol, 1mL/ampul

Each	5-pk.	10-pk.
31628	31628-510	31728

ASTM D6042-96 Internal Standard Mix

Tinuvin® P

51.8µg/mL in isopropanol, 1mL/ampul

Each	5-pk.	10-pk.
31629	31629-510	31729

Analytical Reference Materials



Fruit Juice Organic Acid Standard

citric acid	2000µg/mL
fumaric acid	10
malic acid	2000
quinic acid	2000
tartaric acid	2000

In water, 1mL/ampul

Each	5-pk.	10-pk.
35080	35080-510	—
w/data pack		
35080-500	35080-520	35180

In water, 5mL/ampul

Each	5-pk.	10-pk.
35081	35081-510	—
w/data pack		
35081-500	35081-520	35181



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"The Company Chromatographers Trust"

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- ✓ Mixtures made to your EXACT specifications.
- ✓ Most reference materials shipped within 5-7 days after receipt of your order.*

Restek should be your first choice for custom-made reference materials. Our inventory of over 3,000 pure, characterized, neat compounds ensures you of maximum convenience, maximum value, and minimum time spent blending mixtures in your lab. For our online custom reference material request form, visit <http://www.restekcorp.com/stdreq.htm>

*Availability of raw materials and final product testing required may affect delivery of some mixtures. International orders require additional shipping time.

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Introduction

There is an immense range of analytes and matrices present in food, flavor, and fragrance systems, and many governing organizations have developed requirements for their testing. The Nutritional Labeling and Education Act (NLEA) of 1990 requires that labels containing information about nutritional content be placed on nearly all processed foods. The food analyst is called upon to provide the analytical data for these labels. Testing of the packaging materials or a chemical shelf-life study may be requested for a product. In addition, the food and flavor chemist might need to test the quality of incoming raw materials, determine the flavor profile of a product or ingredient, or quantify trace-level nutrients.

The Association of Official Analytical Chemists (AOAC) International has published many methods for the analysis of foods, broken out by analyte and matrix type. Chromatographic techniques are used in a number of these procedures. Other organizations, such as AOCS (American Oil Chemists Society) and AACC (American Association of Cereal Chemists) also have published chromatographic methods for food and ingredient testing. Because of the complexity of many food, flavor, and fragrance matrices, some type of separation or isolation technique may be needed to ensure accurate results. For example, in the analysis of flavors and fragrances, hundreds of volatile species can contribute to an aroma or flavor. Gas and liquid chromatography (GC and HPLC) are powerful techniques that can greatly reduce the amount of sample preparation needed, as well as provide additional selectivity. By separating the compound(s) of interest from the sample matrix, accurate identification and quantitation becomes much more likely.

GC often is used in the analysis of flavor and fragrance compounds, lipids, and preservatives. To analyze a compound by GC, the compound must be volatile and thermally stable; however, a compound can be derivatized to increase its volatility. The original methods used to analyze foods and flavors were developed for packed GC columns, although capillary columns have surpassed these in popularity. Detectors such as the flame ionization detector (FID) and thermal conductivity detector (TCD) are popular in food analysis. In addition, a GC with a mass spectral detector (MSD) is a powerful tool in the analysis of complex flavor and fragrance samples.

HPLC is used in the analysis of carbohydrates, organic acids, preservatives, and some flavor compounds. HPLC offers a wide variety of column types and mobile phases, making this technique applicable to many compound types. Reversed phase, normal phase, ion exchange, size exclusion, and bioaffinity separation techniques are all useful in food, flavor, and fragrance analysis. Detectors used in HPLC include UV-visible, refractive index, fluorescence, conductivity, and mass spectrometers, depending on the application.

This guide shows example foods, flavors, and fragrances analyses obtained by using Restek GC and HPLC columns. Many additional examples, and much information, can be attained from the reference publications, from our general catalog, and from our website.



Rebecca Wittrig, Ph.D.
HPLC Products
Marketing Manager

Becky has more than 14 years experience in HPLC & GC. Prior to joining Restek as the Foods, Flavors, & Fragrances Innovations Manager, she supervised the chromatography labs at The Pillsbury Company and Ecolab, Inc. Becky has a Ph.D. in Analytical Chemistry from Purdue University and a B.A. in Chemistry from Gustavus Adolphus College (Minnesota). If you have any questions or comments about food, flavor, or fragrance analyses, please contact Becky by e-mail at becky.wittrig@restekcorp.com or by phone at 800-356-1688 or 814-353-1300, ext. 2347.

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List is accurate to the best of our knowledge at the time of printing. Consult individual manufacturers or other sources for specific information.

Fats & Oils

Analyzing Fats & Oils

In foods, fats serve several functions—providing flavor, texture, and serving as a source of essential fatty acids and fat-soluble vitamins. Lipids are substances in foods that are soluble in a non-polar solvent, such as hexane, benzene, or chloroform/methanol. They include compounds such as glycerides, free fatty acids, phospholipids, glycolipids, terpenes, sterols, and waxes. Lipids can be divided into three general groups: 1) simple lipids, which include fats and waxes; 2) compound lipids, which include phospholipids and glycolipids; and 3) derived lipids, which include fatty acids, alcohols, and sterols. Over 90% of the lipids found in food are present as triglycerides—esters of fatty acids and glycerol.

Free Fatty Acids

Free fatty acid molecules consist of carbon chains of varying lengths with an acidic group ($-\text{COOH}$) at one end of the molecule. Fatty acids with chain lengths of 2–20 carbon atoms account for up to 10% of the lipid content in food. In general, these fatty acids are straight chain molecules, either fully hydrogenated or with some degree of unsaturation (i.e., double bonds). Because free fatty acids are adsorptive and the longer chain acids lack volatility, analysis of these compounds can be difficult. The acids can be converted to methyl esters and analyzed by GC, but the additional sample preparation required to do this increases time and cost. The analysis of free fatty acids without derivatization can be accomplished using a **Stabilwax®-DA** column, a bonded Carbowax® column specifically deactivated for acidic compounds. To minimize loss from discrimination in the injection port, direct injection is recommended, although splitless injections can be used. For additional examples of organic acid analysis, see pages 14–15.

for **more** info

Request Applications Note **GC Analysis of Free Fatty Acids on Stabilwax®-DA Columns** (cat.# 59155B).

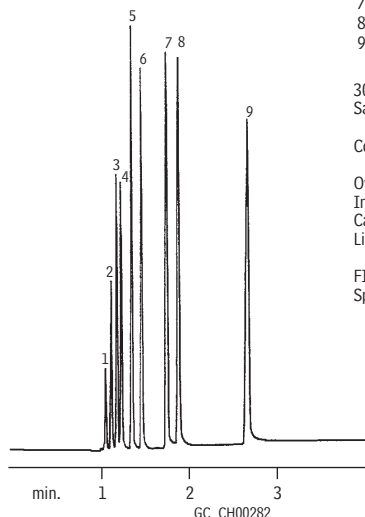
Fatty Acids (Free) Rtx®-200

GC

Peak List:

1. acetic acid
2. propionic acid
3. isobutyric acid
4. *n*-butyric acid
5. isovaleric acid
6. *n*-valeric acid
7. isocaproic acid
8. caproic acid
9. heptanoic acid

Also see page 15!



30m, 0.25mm ID, 0.25 μ m Rtx®-200 (cat.# 15023)
 Sample: 0.8 μ L split injection of a free fatty acid standard.
 Conc.: approximately 10 to 20ng/ μ L.
 Oven temp.: 90°C
 Inj. & det. temp.: 250°C
 Carrier gas: hydrogen
 Linear velocity: 40cm/sec.
 (flow rate: 1.4cc/min.)
 FID sensitivity: 4 x 10⁻¹¹ AFS
 Split vent: 40cc/min.

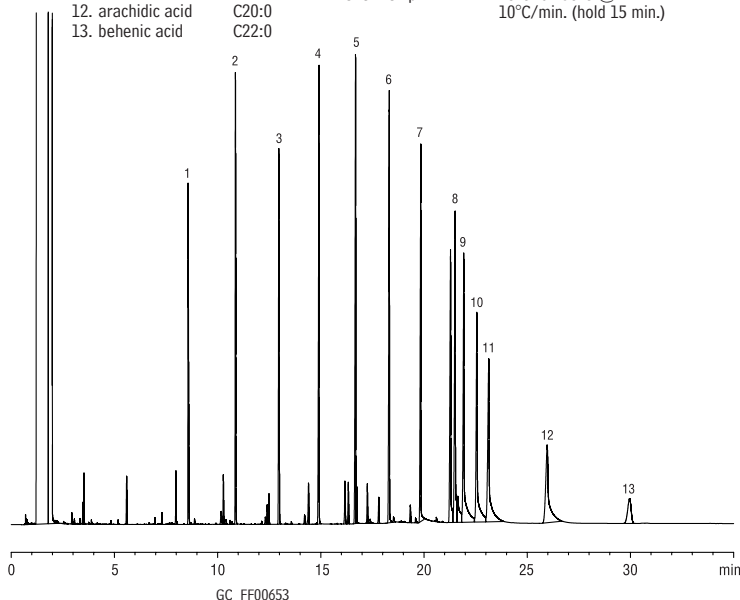
Fatty Acids (Free) Stabilwax®-DA

GC

Peak List:

- | | |
|--------------------|-------|
| 1. butyric acid | C4:0 |
| 2. caproic acid | C6:0 |
| 3. caprylic acid | C8:0 |
| 4. capric acid | C10:0 |
| 5. lauric acid | C12:0 |
| 6. myristic acid | C14:0 |
| 7. palmitic acid | C16:0 |
| 8. stearic acid | C18:0 |
| 9. oleic acid | C18:1 |
| 10. linoleic acid | C18:2 |
| 11. linolenic acid | C18:3 |
| 12. arachidic acid | C20:0 |
| 13. behenic acid | C22:0 |

Stabilwax®-DA 30m, 0.32mm ID, 0.25 μ m (cat.# 11024)
 Sample: 1.0 μ L FFA Mix
 Solvent: water
 Conc.: 5mg/mL in methanol
 Inj.: splitless/250°C
 Splitless hold time: 0.25min.
 Carrier gas: hydrogen (constant flow mode)
 Flow rate: 6.0mL/min.
 Split flow: 75mL/min.
 Det.: FID/250°C
 Inlet liner: laminar cup splitter
 Oven temp.: 40°C to 250°C @ 10°C/min. (hold 15 min.)

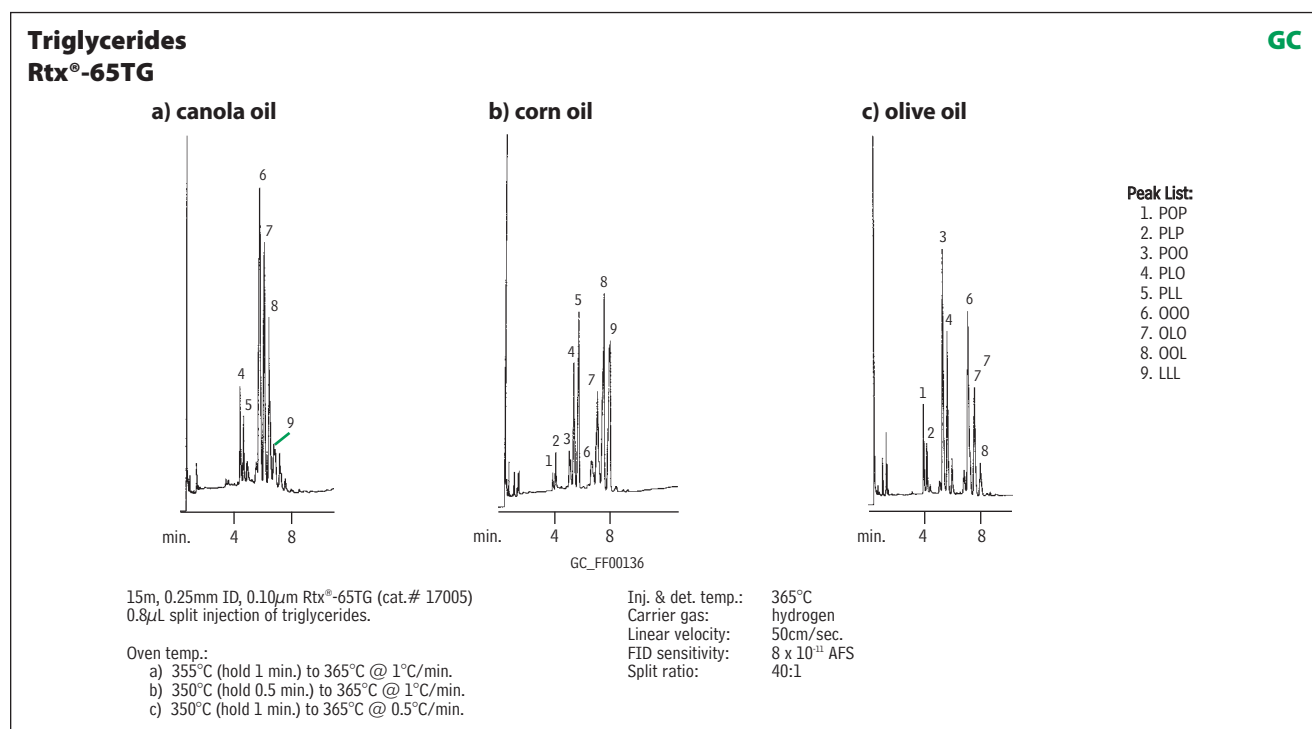


Triglycerides

Triglycerides are naturally-occurring esters of fatty acids and glycerol, and the main component (90-95%) of dietary lipids. Mono- and diglycerides also are esters, but contain one or two fatty acid groups, respectively. Triglycerides are classified according to the nature of their esterified fatty acids. The fatty acid groups in the triglyceride molecule can be classified as saturated or fully hydrogenated (e.g., C14:0), or unsaturated (e.g., C18:1 or C18:2).

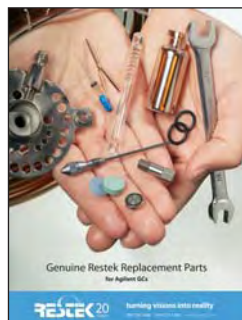
In general, capillary GC columns are the preferred tool for triglyceride analyses, providing shorter analyses times, higher efficiency, and better quantitation than packed column GC, HPLC, or supercritical fluid chromatography (SFC). Sample preparation is minimal, involving liquefying the sample before diluting with a solvent such as dichloromethane or isooctane. Additional sample preparation is necessary if mono- and diglycerides and free fatty acids are present in significant amounts. Because these compounds have relatively high molecular weights and polarities that increase with the degree of unsaturation, high oven temperatures are necessary. An **Rtx®-65TG** (65% phenyl/35% methyl polysiloxane) column is able to resolve triglycerides according to degree of unsaturation, as well as according to carbon number. The extended thermal stability of this column allows the use of a high oven temperature, yielding short analyses times. In addition, the advanced deactivation techniques used to prepare **Rtx®-65TG** columns result in lower bleed and longer column lifetimes than for traditional columns.

For more information, request Applications Note **GC Analysis of Triglycerides** (cat.# 59580A).

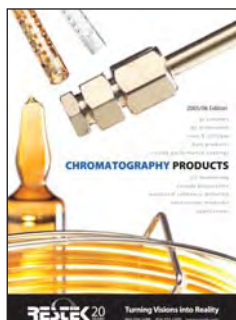


for more info

Restek offers an extensive line of GC and HPLC columns, accessories, and replacement parts. Call to request one of these catalogs for a full listing of products or visit us on the web at www.restek.com



Agilent GC Replacement
Parts, Lit. Cat. #59627E



2005 General Catalog
Lit. Cat. #59065



HPLC Products
Lit. Cat. #59241B

Fats & Oils

Fatty acid methyl esters (FAMES)

FAMES analysis is important in fats and oils characterization and in the determination of total fat content in foods. To prepare the methyl esters, fats are extracted from matrices using non-polar solvents, and saponified to produce free fatty acids. After derivatization to the methyl esters, molecules have increased volatility and decreased activity, which permits more accurate quantitation by GC.

Capillary columns with polyethylene glycol (PEG) or Carbowax® stationary phases are used to analyze saturated and unsaturated FAMES. For the resolution of the *cis* and *trans* isomers, bis-cyanopropyl phases typically are used. **Stabilwax®** and **Rtx®-Wax** columns provide excellent resolution of FAMES derived from both plant and animal sources. **FAMEWAX®** columns offer excellent resolution of polyunsaturated FAMES with significantly reduced analyses times, compared to traditional Carbowax® stationary phases. Individual *cis* and *trans* isomers are resolved on an **Rt-2560** column, making it the column of choice for analyzing partially hydrogenated fats.

for more info

Request Applications Note **Analyzing Fatty Acid Methyl Esters** (cat.# 59584A).

ordering note

To order one of the columns highlighted on pages 1 through 7, please see the following pages:

FAMEWAX™	33
Rtx®-65TG	32
Rtx®-200	35
Rtx®-2560	36
Rtx®-Wax	33
Stabilwax®	34
Stabilwax®-DA	34

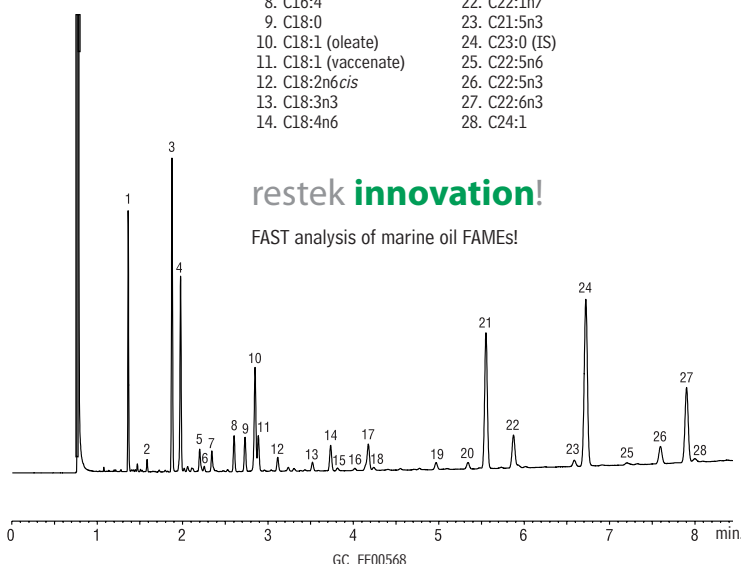
FAMES (Marine Oil) FAMEWAX™

GC

FAMEWAX™ 30m, 0.32mm ID, 0.25µm (cat.# 12498)
 Sample: 12mg/mL total FAMES
 Inj.: 0.5µL, split (150:1), 3mm ID split liner for Trace Series GCs, packed with glass wool (cat.# 20936-202.1)
 Inj. temp.: 250°C
 Carrier gas: hydrogen, constant flow
 Linear velocity: 62cm/sec.
 Oven temp.: 195°C to 240°C @ 5°C/min. (hold 1 min.)
 Det.: FID, 250°C

Peak List:

1. C14:0	15. C18:4n3
2. C15:0	16. C20:0
3. C16:0	17. C20:1n7
4. C16:1	18. C20:1n9
5. C16:2	19. C20:4n6
6. C17:0	20. C20:4n3
7. C17:1	21. C20:5n3
8. C16:4	22. C22:1n7
9. C18:0	23. C21:5n3
10. C18:1 (oleate)	24. C23:0 (IS)
11. C18:1 (vaccenate)	25. C22:5n6
12. C18:2n6cis	26. C22:5n3
13. C18:3n3	27. C22:6n3
14. C18:4n6	28. C24:1



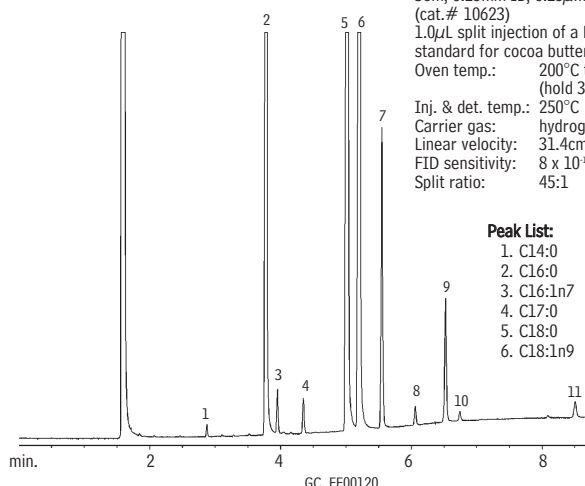
FAMES (Cocoa Butter) Stabilwax®

GC

30m, 0.25mm ID, 0.25µm Stabilwax® (cat.# 10623)
 1.0µL split injection of a FAME reference standard for cocoa butter.
 Oven temp.: 200°C to 250°C @ 8°C/min. (hold 3 min.)
 Inj. & det. temp.: 250°C
 Carrier gas: hydrogen
 Linear velocity: 31.4cm/sec. set @ 200°C
 FID sensitivity: 8 x 10⁻¹¹ AFS
 Split ratio: 45:1

Peak List:

1. C14:0	7. C18:2n6
2. C16:0	8. C18:3n3
3. C16:1n7	9. C20:0
4. C17:0	10. C20:1n9
5. C18:0	11. C22:0
6. C18:1n9	



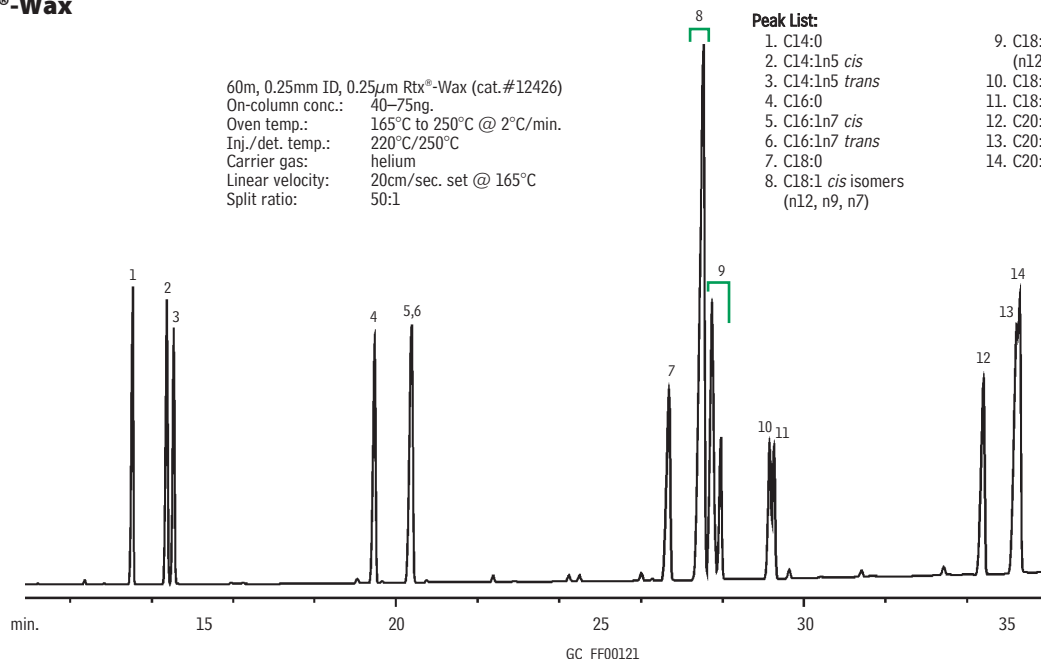
FAMES (cis/trans isomers) Rtx®-Wax

GC

60m, 0.25mm ID, 0.25µm Rtx®-Wax (cat.#12426)
On-column conc.: 40–75ng.
Oven temp.: 165°C to 250°C @ 2°C/min.
Inj./det. temp.: 220°C/250°C
Carrier gas: helium
Linear velocity: 20cm/sec. set @ 165°C
Split ratio: 50:1

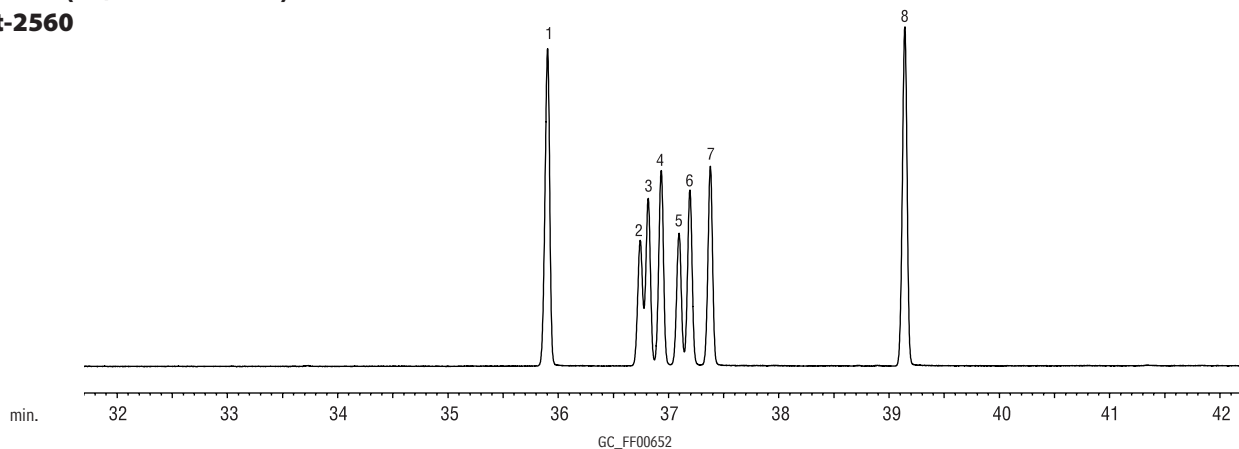
Peak List:

- | | |
|---|---|
| 1. C14:0 | 9. C18:1 <i>trans</i> isomers (n12, n9, n7) |
| 2. C14:1n5 <i>cis</i> | 10. C18:2n6 <i>cis</i> |
| 3. C14:1n5 <i>trans</i> | 11. C18:2n6 <i>trans</i> |
| 4. C16:0 | 12. C20:0 |
| 5. C16:1n7 <i>cis</i> | 13. C20:1n9 <i>cis</i> |
| 6. C16:1n7 <i>trans</i> | 14. C20:1n9 <i>trans</i> |
| 7. C18:0 | |
| 8. C18:1 <i>cis</i> isomers (n12, n9, n7) | |



FAMES (cis/trans isomers) Rt-2560

GC



Rt-2560, 100m, 0.25mm ID, 0.2µm (cat.# 13199)
Sample: *cis/trans* FAME Mix (cat.# 35079), 10mg/mL total FAMES in methylene chloride
Inj.: 1.0µL split (split ratio 20:1), 4mm inlet liner (cat.# 20814)
Inj. temp.: 225°C
Carrier gas: hydrogen, constant flow
Flow rate: 1.2mL/min.
Oven temp.: 100°C (4 min. hold)
to 240°C @ 3°C/min. (10 min. hold)
Det.: FID @ 250°C

Compound

% in Mix

- | | |
|---|------|
| 1. C18:0 methyl stearate | 20.0 |
| 2. C18:1 methyl petroselaidate (<i>trans</i> -6) | 8.0 |
| 3. C18:1 methyl elaidate (<i>trans</i> -9) | 10.0 |
| 4. C18:1 methyl transvacenate (<i>trans</i> -11) | 12.0 |
| 5. C18:1 methyl petroselinate (<i>cis</i> -6) | 8.0 |
| 6. C18:1 methyl oleate (<i>cis</i> -9) | 10.0 |
| 7. C18:1 methyl vacenate (<i>cis</i> -11) | 12.0 |
| 8. C18:2 methyl linoleate (<i>cis</i> -9,12) | 20.0 |

did you know?

Our Technical Service Department is staffed with more than 35 experienced chemists on rotating shifts from various departments. Whether your chromatography problem is simple or complex, call Restek's Technical Service Team at 1-800-356-1688 (ext. 4), or your Restek representative, and we will do everything we can to help you find a solution.

Fats & Oils

The Institute for Nutraceutical Advancement (INA) has published a method for the analysis of the fatty acid content in saw palmetto by GC. The analysis is performed after the triglycerides are transesterified and converted to their methyl esters. An **Rtx®-Wax** column provides the efficiency and selectivity needed to perform this analysis, allowing accurate identification of the FAMES present.

for more info

Request Applications Note **The Institute for Nutraceutical Advancement (INA) Validates GC Methods for Saw Palmetto Using Rtx®-5 and Stabilwax® Columns** (cat.# 59136).

FAMES (Saw Palmetto) Rtx®-Wax

GC

Peak List	Conc. (mg/mL)
1. methyl caproate (C6:0)	0.4
2. methyl caprylate (C8:0)	0.4
3. methyl nonanoate (C9:0)	2.0
4. methyl caprate (C10:0)	0.4
5. methyl laurate (C12:0)	5.0
6. methyl myristate (C14:0)	2.0
7. methyl palmitate (C16:0)	2.0
8. methyl palmitoleate (C16:1)	0.4
9. methyl stearate (C18:0)	0.4
10. methyl oleate (C18:1)	5.0
11. methyl linoleate (C18:2)	1.0
12. methyl linolenate (C18:3)	0.4

30m, 0.25mm, 0.25µm Rtx®-Wax (cat.# 12423)

1µL split injection of saw palmetto standard

Conc.: see peak list

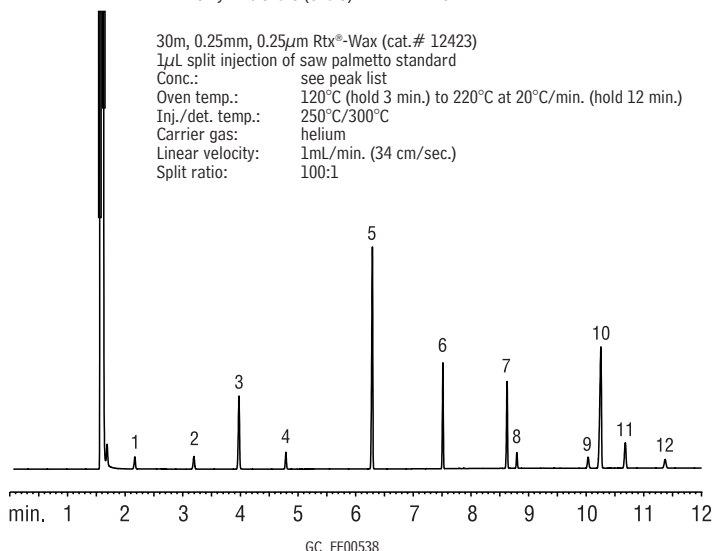
Oven temp.: 120°C (hold 3 min.) to 220°C at 20°C/min. (hold 12 min.)

Inj./det. temp.: 250°C/300°C

Carrier gas: helium

Linear velocity: 1mL/min. (34 cm/sec.)

Split ratio: 100:1



FAMES (NLEA) Rt-2560

GC

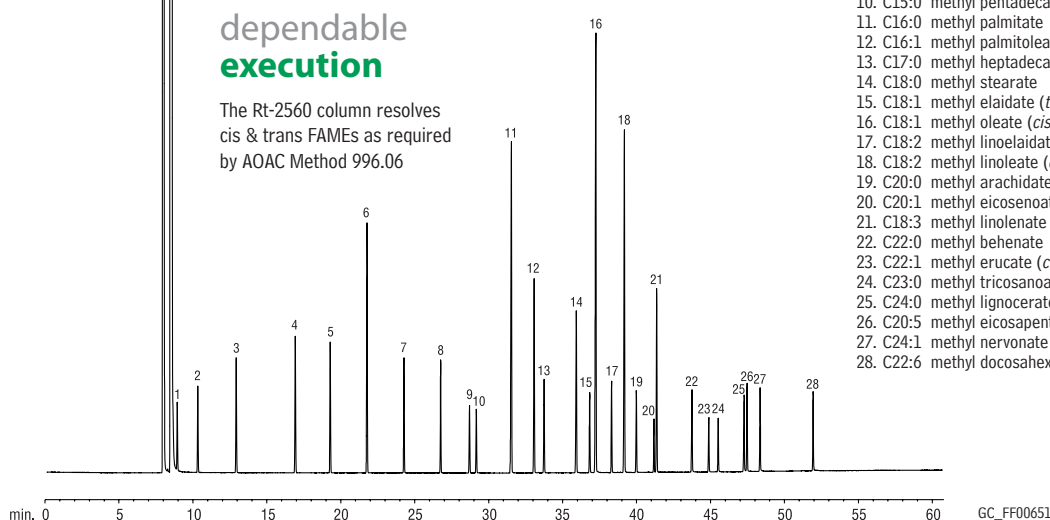
Column: Rt-2560 100m, 0.25mm ID, 0.20µm (cat.# 13199)
 Sample: NLEA FAME Mix (cat.# 35078), 30mg/mL total FAMES in methylene chloride
 Inj.: 1.0µL split (split ratio 100:1), 4mm inlet liner (cat.# 20814)
 Inj. temp.: 225°C
 Carrier gas: hydrogen, constant flow
 Flow rate: 1.2 mL/min.
 Oven temp.: 100°C (4 min. hold) to 240°C @ 3°C/min. (10 min. hold)
 Det.: FID @ 250°C

dependable execution

The Rt-2560 column resolves cis & trans FAMES as required by AOAC Method 996.06

Peak List:

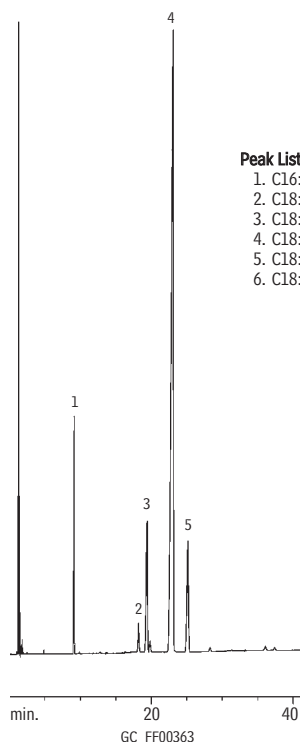
1. C4:0 methyl butyrate
2. C6:0 methyl hexanoate
3. C8:0 methyl octanoate
4. C10:0 methyl decanoate
5. C11:0 methyl undecanoate
6. C12:0 methyl laurate
7. C13:0 methyl tridecanoate
8. C14:0 methyl myristate
9. C14:1 methyl myristoleate (*cis*-9)
10. C15:0 methyl pentadecanoate
11. C16:0 methyl palmitate
12. C16:1 methyl palmitoleate (*cis*-9)
13. C17:0 methyl heptadecanoate
14. C18:0 methyl stearate
15. C18:1 methyl elaidate (*trans*-9)
16. C18:1 methyl oleate (*cis*-9)
17. C18:2 methyl linoelaidate (*trans*-9,12)
18. C18:2 methyl linoleate (*cis*-9,12)
19. C20:0 methyl arachidate
20. C20:1 methyl eicosenoate (*cis*-11)
21. C18:3 methyl linolenate (*cis*-9,12,15)
22. C22:0 methyl behenate
23. C22:1 methyl erucate (*cis*-13)
24. C23:0 methyl tricosanoate
25. C24:0 methyl lignocerate
26. C20:5 methyl eicosapentaenoate (*cis*-5,8,11,14,17)
27. C24:1 methyl nervonate (*cis*-15)
28. C22:6 methyl docosahexaenoate (*cis*-4,7,10,13,16,19)



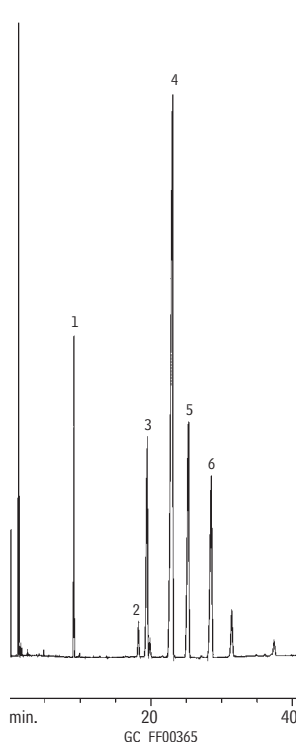
FAMES
FAMEWAX™

GC

Evening Primrose Oil



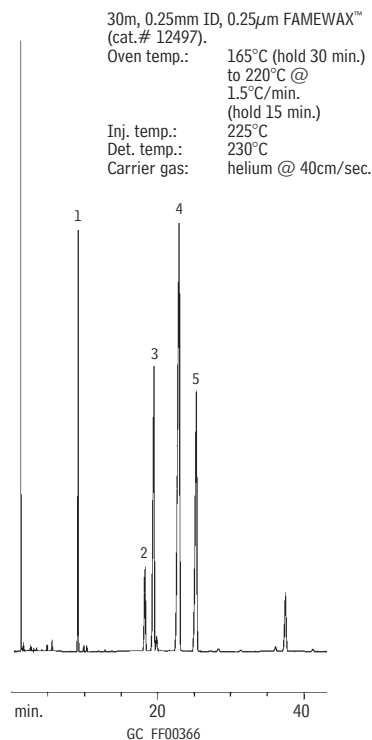
Black Currant Seed Oil



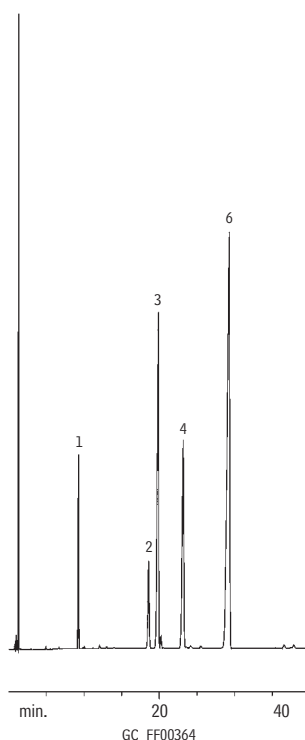
Peak List:

1. C16:0
2. C18:0
3. C18:1n9
4. C18:2n6
5. C18:3n6
6. C18:3n3

Borage Seed Oil



Flax Seed Oil



30m, 0.25mm ID, 0.25µm FAMEWAX™
(cat.# 12497).
Oven temp.: 165°C (hold 30 min.)
to 220°C @
1.5°C/min.
(hold 15 min.)
Inj. temp.: 225°C
Det. temp.: 230°C
Carrier gas: helium @ 40cm/sec.

Essential fatty acids

Essential fatty acids (EFAs) are polyunsaturated fatty acids (PUFAs) that the body needs to perform important functions, including: determining membrane fluidity, reactivity, oxidation rate, and energy production, maintaining body temperature, insulating nerves, and cushioning body tissue. However, the body cannot produce EFAs, they must be obtained through the diet. Two important families of EFAs are the Omega-3 (n-3) series and the Omega-6 (n-6) series. The Omega-3 series includes α -linolenic acid, eicosapentaenoic acid, and docosahexaenoic acid. The Omega-6 series includes linoleic acid, γ -linolenic acid, dihomo- γ -linolenic acid, and arachidonic acid.

A FAMEWAX™ column has excellent selectivity for EFAs. The PEG stationary phase used in the FAMEWAX™ will resolve the Omega-3 and Omega-6 fatty acids, and the isomers of linolenic acid (C18:3n3 and C18:3n6). The samples are saponified and esterified to form their FAMES before injection. Accurate determinations of the fatty acid profiles of oils, such as flax seed oil and evening primrose oil, also are possible with this column.

for more info

Request Applications Note **Determination of Omega-3 and Omega-6 Fatty Acid Composition in Evening Primrose Oil, Flax Seed Oil, and Borage Oil** (cat.# 59128).



Julie Kowalski
Innovation Chemist

Fats & Oils

Cholesterol and Other Dietary Sterols

Cholesterol is a lipid with a completely different structure than a fatty acid. Cholesterol is present only in foods of animal origin. Because cholesterol content must be included on nutritional label panels, accurate quantitation is important for products such as butter, eggs, and baked goods. Capillary GC is recommended in AOAC Methods 970.51E and 976.26 for the determination of cholesterol content. Cholesterol, and other sterols, must be recovered from the unsaponified fraction of an ether extract. The sterols can be converted to the trimethylsilyl (TMS) or butyl ester derivatives and analyzed on an **Rtx®-5** capillary column, or they can be analyzed underivatized on a highly inert **XTI®-5** column. An **XTI®-5** column offers low reactivity and high thermal stability for accurate quantitation with very short analyses times. For more complex mixtures of sterols, including coprostanone and cholesterol, a more polar **Rtx®-225** column should be used (page 9).

for more info

Request Applications Note **Analysis of Cholesterol and Other Dietary Sterols** (cat.# 59581).

ordering note

To order one of the columns highlighted on **page 8 or 9**, please see the following pages:

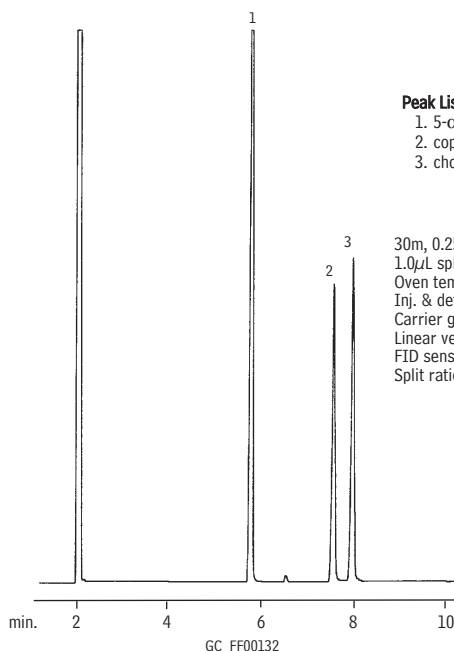
Rtx®-5	31
Rtx®-225	33
XTI®-5	31

also available

Custom lengths and film thicknesses are available. Call technical service at **800-356-1688 (ext. 4)**, or contact your Restek representative.

Sterols (Cholesterol) XTI®-5

GC



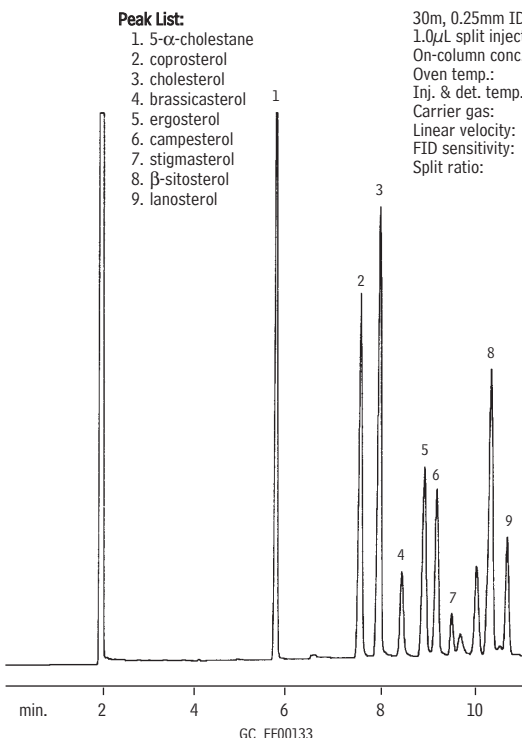
Peak List:

1. 5- α -cholestane
2. coprosterol
3. cholesterol

30m, 0.25mm ID, 0.50 μ m XTI®-5 (cat.# 12238)
 1.0 μ L split injection, 250ng on-column
 Oven temp.: 330°C
 Inj. & det. temp.: 300°C
 Carrier gas: helium
 Linear velocity: 30cm/sec. set @ 40°C
 FID sensitivity: 8 x 10⁻¹¹ AFS
 Split ratio: 100:1

Sterols XTI®-5

GC



Peak List:

1. 5- α -cholestane
2. coprosterol
3. cholesterol
4. brassicasterol
5. ergosterol
6. campesterol
7. stigmasterol
8. β -sitosterol
9. lanosterol

30m, 0.25mm ID, 0.50 μ m XTI®-5 (cat.# 12238)
 1.0 μ L split injection.
 On-column conc.: 250ng
 Oven temp.: 330°C
 Inj. & det. temp.: 300°C
 Carrier gas: helium
 Linear velocity: 30cm/sec. set @ 40°C
 FID sensitivity: 8 x 10⁻¹¹ AFS
 Split ratio: 100:1

Sterols (Cholesterol) Rtx®-225

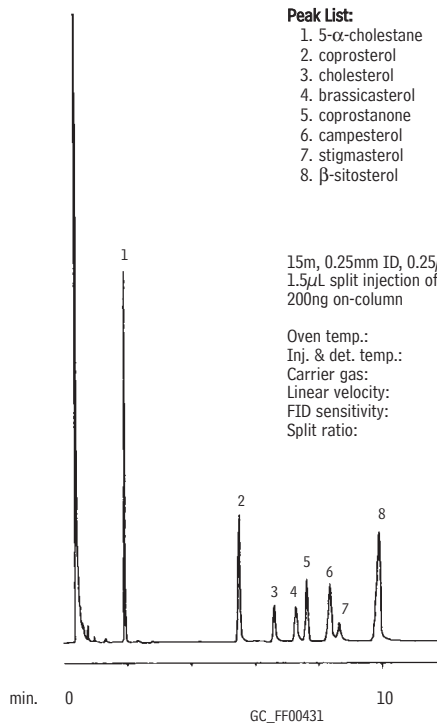
GC

Peak List:

1. 5- α -cholestane
2. coprosterol
3. cholesterol
4. brassicasterol
5. coprostanone
6. campesterol
7. stigmasterol
8. β -sitosterol

15m, 0.25mm ID, 0.25 μ m Rtx®-225 (cat.# 14020)
1.5 μ L split injection of neutral sterols and phytosterols,
200ng on-column

Oven temp.: 260°C
Inj. & det. temp.: 260°C
Carrier gas: helium
Linear velocity: 45cm/sec. set @ 240°C
FID sensitivity: 8 x 10⁻¹¹ AFS
Split ratio: 30:1



In addition to the method for fatty acid content in saw palmetto (page 6), the INA has published a method for the determination of sterols in saw palmetto by GC. This assay can be applied to stigmasterol, campesterol, brassicasterol, and β -sitosterol in saw palmetto fruit, oil extract, and blended powders. The sample is analyzed after hydrolysis, saponification, and derivatization of the sterols. For this assay, an **Rtx®-5** column is used. This column features the thermal stability needed to provide accurate quantitation of the phytosterols (340°C).

for more info

Request Applications Note **The Institute for Nutraceutical Advancement (INA) Validates GC Methods for Saw Palmetto Using Rtx®-5 and Stabilwax Columns** (cat.# 59136).

Phytosterols (Saw Palmetto) Rtx®-5

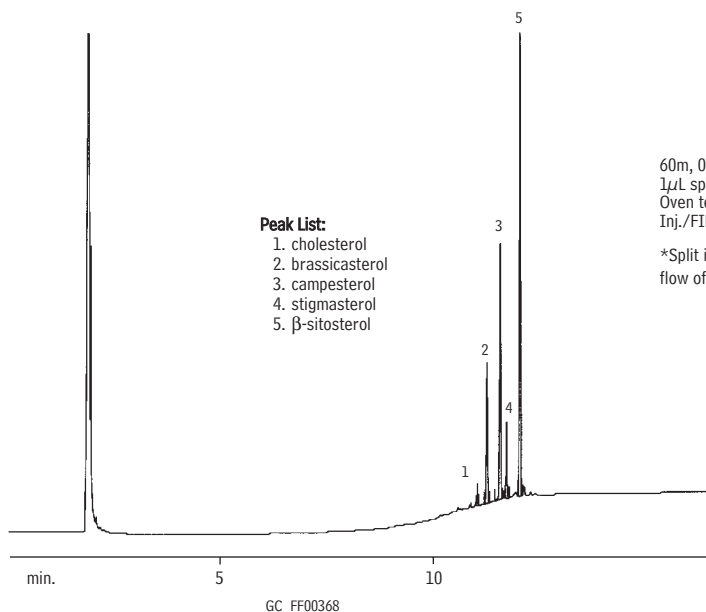
GC

Peak List:

1. cholesterol
2. brassicasterol
3. campesterol
4. stigmasterol
5. β -sitosterol

60m, 0.25mm ID, 0.25 μ m Rtx®-5 (cat.# 10226).
1 μ L splitless injection*
Oven temp.: 200°C (hold 1 min.) to 340°C @ 15°C/min. (hold 10 min.)
Inj./FID temp.: 345°C/355°C.

*Split injection may be used, but results can have greater variability. A split flow of 112mL/min. is suggested.



Chromatogram provided by the Institute for Nutraceutical Advancement (INA)

Carbohydrates

Carbohydrates

Carbohydrates are important constituents of food and beverage products and include simple sugars, oligosaccharides, sugar alcohols, and polysaccharides. Sugar analysis is needed for the generation of nutritional panels, as both the sugar content and the total carbohydrate content must be included. In addition, sugar alcohols are gaining popularity in dietetic foods, and often must be monitored in the presence of other sugars.

Simple sugars include mono- and disaccharides such as fructose, glucose, sucrose, maltose, and lactose. In foods and beverages, they provide sweetness, texture, and color development. The perceived sweetness of each sugar is different and is evaluated on the basis of character, intensity, and duration. Therefore, the ability to profile the individual mono- and disaccharides is important to food chemists. Methods exist for the quantitation of individual sugar species, as well as for the determination of total sugar content.

An oligosaccharide is a polymer of 2–10 simple sugars, including compounds such as lactose, maltose, and maltotriose. A polysaccharide is defined as a polymer of greater than 10 simple sugars, which includes starches and gums. In food systems, they serve as bulking agents, emulsifiers, stabilizers, free-flowing agents, and water binders. The total starch content in foods can be determined by enzymatically digesting the starch, followed by the quantitative measurement of the resulting glucose and maltose.

Sugars: Maple Flavored Syrup Ultra Amino

HPLC

Peak List:

1. fructose
2. glucose
3. sucrose

Sample:

Inj.: 20 μ L
Conc.: 10% solution of maple syrup
Solvent: acetonitrile:water (70:30, v/v)

Column: Ultra Amino

Cat. #: 9107365
Dimensions: 150 x 4.6mm
Particle size: 3 μ m
Pore size: 100Å

Conditions:

Mobile phase: acetonitrile:water (75:25, v/v)
Flow: 0.8mL/min.
Temp.: 35°C
Det.: refractive index

min.

LC_0159

Sugars Test Mix Pinnacle II™ Amino (3 μ m)

HPLC

Peak List:

- | Peak List: | Conc. (μ g/mL) |
|-------------|---------------------|
| 1. fructose | 2.0 |
| 2. glucose | 2.1 |
| 3. sucrose | 4.0 |
| 4. maltose | 4.5 |
| 5. lactose | 4.4 |

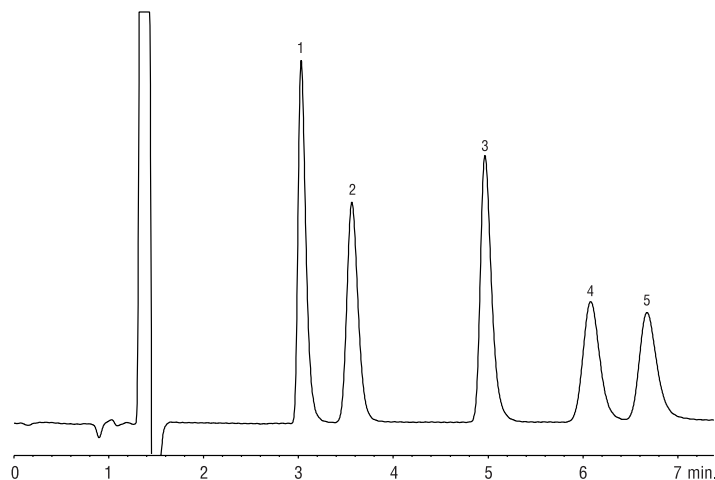
Column:

Pinnacle II™ Amino
Cat. #: 9217365
Dimensions: 150 x 4.6mm
Particle size: 3 μ m
Pore size: 110Å

Conditions:

Mobile phase: water:acetonitrile (25:75, v/v)
Flow: 1.5 mL/min.
Temp.: 35°C
Det.: refractive index @ 35°C

Sample:
Inj.: 5 μ L
Solvent: mobile phase



LC_022323

Sugars (as alditol acetates) Rtx®-225

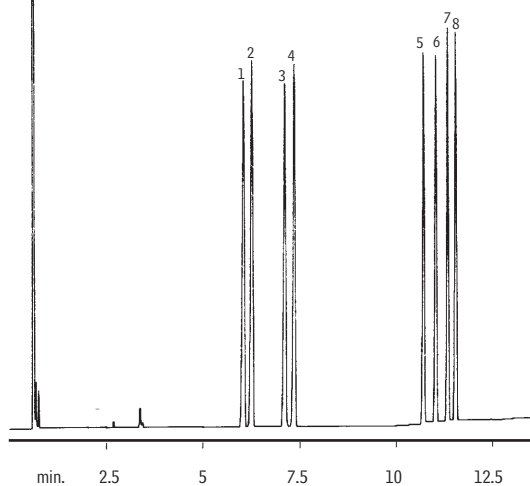
GC

Peak List:

1. rhamnitol
2. fucitol
3. ribitol
4. arabinitol
5. mannitol
6. galactitol
7. glucitol
8. inositol

15m, 0.25mm ID, 0.25µm Rtx®-225 (cat.# 14020)
0.5µL split injection

Oven temp.: 190°C (hold 5 min.) to 250°C
@ 8°C/min. (hold 5 min.)
Inj. & det. temp.: 260°C
Carrier gas: hydrogen
Linear velocity: 42cm/sec. set @ 40°C
FID sensitivity: 16 x 10⁻¹¹ AFS
Split ratio: 50:1



GC_FF00128

Either GC or HPLC, depending on the needs of the analyst and the equipment available, can be used to analyze carbohydrates. Typically, greater sensitivity is possible using GC techniques, although sample preparation will become more involved. HPLC analysis using an amino-based stationary phase is the most popular technique for the routine analysis of simple sugars. This analysis involves isocratic elution (e.g., acetonitrile:water, 75:25) and a refractive index detector (RID). An **Ultra Amino** or **Pinnacle II™ Amino** column can be used to separate fructose, glucose, sucrose, maltose, and lactose. Good resolution can be achieved in less than 15 minutes using a flow rate of 0.8mL/min. This method is applicable to a wide range of food and beverage matrices.

Because sensitivity can be limited when using a refractive index detector, GC often is used for trace-level analyses of simple sugars and sugar alcohols after derivatization, which makes the compounds more volatile and thermally stable. Alditol acetate derivatives of sugars can be analyzed using an **Rtx®-225** capillary column, a cyanopropyl-containing siloxane phase. The TMS derivatives of simple sugars, complex sugars, and sugar alcohols can be analyzed using a bonded packed column, such as **3% Rt-101 on 100/120 Silcoport™** packing.

Sugar Alcohols (TMS derivatives) 3% Rt-101 on 100/120 Silcoport™

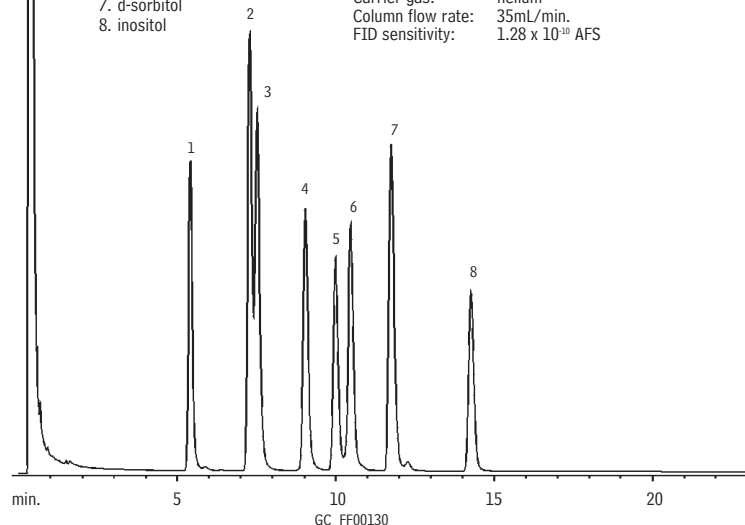
PACKED GC

Peak List:

1. l-arabinose
2. xylitol
3. d-arabinitol
4. d-mannose
5. l-sorbose
6. d-glucose
7. d-sorbitol
8. inositol

3% Rt-101 on 100/120 Silcoport™ (cat.# 80400)
2m, 1/8" OD x 2mm ID

Oven temp.: 140°C to 230°C @ 5°C/min.
(hold 5 min.)
Inj./det. temp.: 250°C / 300°C
Det.: FID
Carrier gas: helium
Column flow rate: 35mL/min.
FID sensitivity: 1.28 x 10⁻¹⁰ AFS



GC_FF00130

Vitamins

Vitamins play vital roles in the human body, and include a wide range of organic compounds. The quantitative analysis of vitamins has become a necessity in both the mainstream food and dietary supplement markets. However, because the body needs vitamins in very small quantities, the analyst often must perform trace-level analyses, which can be challenging. Vitamin assays are used to ensure product quality and to verify nutritional label claims. In addition, assays are important in developing manufacturing and storage processes because many vitamins are light- and/or air-sensitive.

Vitamins can be classified broadly into two groups—water-soluble and fat-soluble. Fat-soluble vitamins include A (retinol), E (alpha-tocopherol), D, and K. They are quite hydrophobic and must be dissolved in an organic solvent. The **Ultra C18** HPLC column features a retentive, high-purity packing that is ideal for separating a range of fat-soluble vitamins. The fully end-capped silica eliminates unwanted analyte-silanol interactions and improves column-to-column reproducibility.

ordering note

Please see pages 40–45 for HPLC columns featured in this catalog.

Vitamins (Fat Soluble) Ultra C18

HPLC

Peak List:	Conc.: (mg/mL)
1. solvent front	n/a
2. menadione (vitamin K ₃)	0.45
3. all- <i>trans</i> -retinol (vitamin A)	0.34
4. vitamin D ₃	0.4
5. unknown	n/a
6. alpha tocopherol (vitamin E)	2.4
7. alpha tocopherol acetate (vitamin E acetate)	2.4
8. unknown	n/a
9. phytylquinone (vitamin K ₁)	0.84

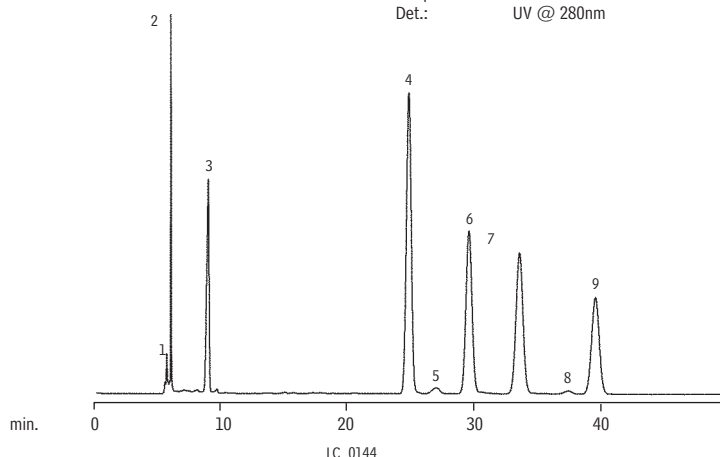
Sample:
Solvent: diethyl ether

Column: **Ultra C18**
Cat. #: 9174575
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:
Mobile phase: acetonitrile:methanol (90:10, v/v)

Time (min.)	Flow (mL/min.)
1.00	1.00
5.00	1.00
5.01	2.00
50.0	2.00

Temp.: 30°C
Det.: UV @ 280nm



Vitamins (Water Soluble) Ultra Aqueous C18

HPLC

Peak List:	Conc.: (mg/mL)
1. thiamin (B ₁)	250
2. ascorbic acid (C)	1000
3. unknown	n/a
4. nicotinic acid (B ₃)	1000
5. unknown	n/a
6. pantothenic acid (B ₅)	1000
7. folic acid (B ₉)	500
8. riboflavin (B ₂)	250
9. methyl paraben	0.2

Sample:
Initial dilutions of B₁ and B₂ basified with ammonium hydroxide (to promote solubility)

Column: **Ultra Aqueous C18**
Cat. #: 9178575
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 100Å

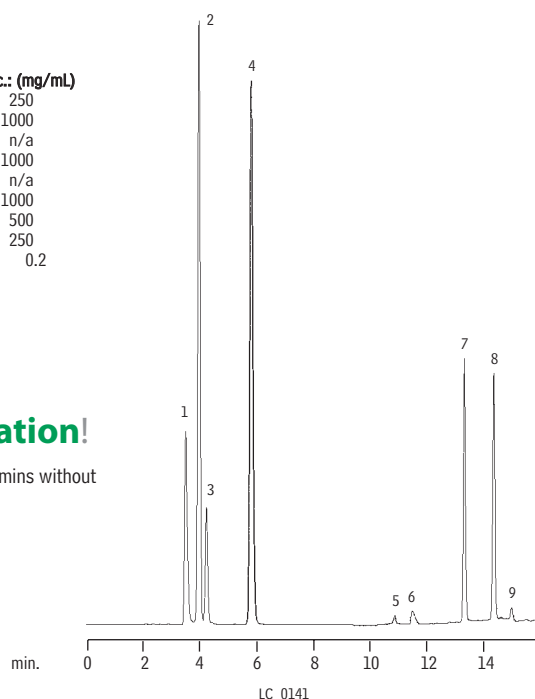
Conditions:
Mobile Phase: A: 25mM potassium phosphate, pH 2.00:methanol (95:5, v/v)
B: methanol:25mM potassium phosphate, pH 3.5 (60:40, v/v)

Time (min.)	% B
0-6	0
6.01	25
6.01-11	25-100
11-16	100

Flow: 1.0mL/min.
Temp.: 27°C
Det.: UV @ 254nm

restek **innovation!**

Monitor water-soluble vitamins without ion pairing reagents.



Cabbage Extract Ultra Aqueous C18

HPLC

Peak List:
1. phenethyl glucosinolate

Sample:
Inj.: 20 µL
Solvent: water

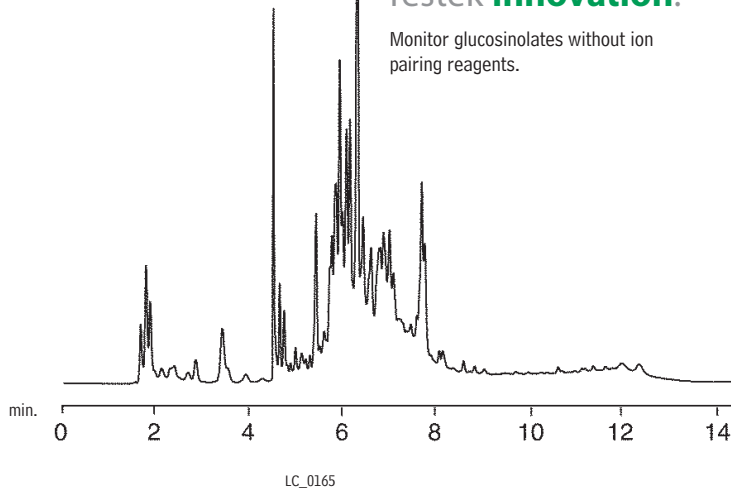
Column: Ultra Aqueous C18
Cat. #: 9178565
Dimensions: 150 x 4.6mm
Particle size: 5 µm
Pore size: 100 Å

Conditions:
Mobile phase: A: 50mM potassium phosphate, pH 2.5
B: acetonitrile
Time (min.): % B
0.0 0
10 75
11 0
16 0

Flow: 1.0mL/min.
Temp.: ambient
Det.: UV @ 210nm

restek innovation!

Monitor glucosinolates without ion pairing reagents.



Water-soluble vitamins include both acidic and basic compounds, as well as some highly polar molecules, e.g., ascorbic acid (vitamin C), thiamin (B1), riboflavin (B2), nicotinic acid (B3), pyridoxine (B6), and folic acid. The very polar compounds are difficult to retain by reversed phase HPLC, and many methods call for ion-pairing reagents to improve retention. **Ultra Aqueous C18** HPLC columns resolve six water-soluble vitamins using a gradient elution program, without the need for ion pairing reagents.

Consumption of glucosinolates (β -thioglucoside N-hydroxysulfate precursors of isothiocyanates) is associated with a significantly reduced risk for a variety of cancers. Because glucosinolates are highly polar, ion pairing reagents are sometimes used to retain them by reversed phase HPLC.

Due to enhanced retention for polar compounds and compatibility with 100% aqueous mobile phases, an **Ultra Aqueous C18** column can separate glucosinolates by reversed phase HPLC without ion pairing reagents.

for more info

Request Applications Note **Analyze Polar Compounds by Reversed Phase HPLC Using Ultra Aqueous C18 Column** (cat. # 59177).

pinnacle II™ hplc columns

Developed using Restek silica. We strictly control the quality of raw material, phase bonding, and column packing. You'll be impressed with our column-to-column reproducibility!

For more information and applications, request the **Pinnacle II™ Column** flyer. (lit. cat. #59281)



Amino Acids

Proteins are polymeric materials with molecular weights greater than 5,000. The basic building blocks of proteins are amino acids, which have the nutritional properties, but not the functional properties, of proteins. There are 20 common amino acids in food systems, categorized as essential or nonessential amino acids. Derivatization often is used to provide adequate retention of amino acids, especially the more hydrophilic compounds. An **Ultra Aqueous C18** reversed phase HPLC column can separate many amino acids without derivatization or ion pairing reagents. To maximize retention, the **Ultra Aqueous C18** column can be used with a 100% aqueous mobile phase without compromising the reproducibility of the analysis.

Organic Acids

Organic acids play several important roles in food and beverage systems. For example, they are important flavor compounds and indicators of product quality. In some fruit juices, the organic acid profile is monitored to determine the purity of the fruit juice. Malic acid and citric acid can be found in fruits, oxalic acid can be found in spinach and rhubarb, and tartaric acid is present in grapes. In food systems, organic acids may be added as acidulants, to control the pH of the product. Certain organic acids also can be used as antimicrobial agents; for example, propionic acid can be used to inhibit mold growth.

The analysis of polar organic acids can be difficult using conventional reversed phase HPLC columns, even with highly aqueous mobile phases. The **Ultra Aqueous C18** column was designed for challenging applications such as this, and provides enhanced retention and selectivity for organic acids.



Cathy Gross
HPLC Products
Marketing Manager

Amino Acids Ultra Aqueous C18

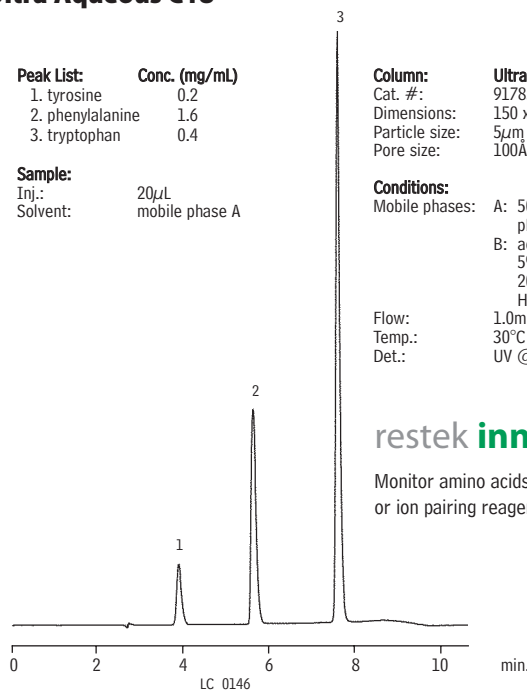
HPLC

Peak List:	Conc. (mg/mL)
1. tyrosine	0.2
2. phenylalanine	1.6
3. tryptophan	0.4

Sample:
Inj.: 20 µL
Solvent: mobile phase A

Column: **Ultra Aqueous C18**
Cat. #: 9178565
Dimensions: 150 x 4.6mm
Particle size: 5 µm
Pore size: 100 Å

Conditions:
Mobile phases: A: 50mM potassium phosphate, pH 2.5
B: acetonitrile
5% - 20% B: 0-5 min.
20% - 5% B: 5-6 min.
Hold at 5% B: 6-13 min.
Flow: 1.0 mL/min.
Temp.: 30°C
Det.: UV @ 254nm



restek **innovation!**

Monitor amino acids without derivatization or ion pairing reagents.

Organic Acids Ultra Aqueous C18

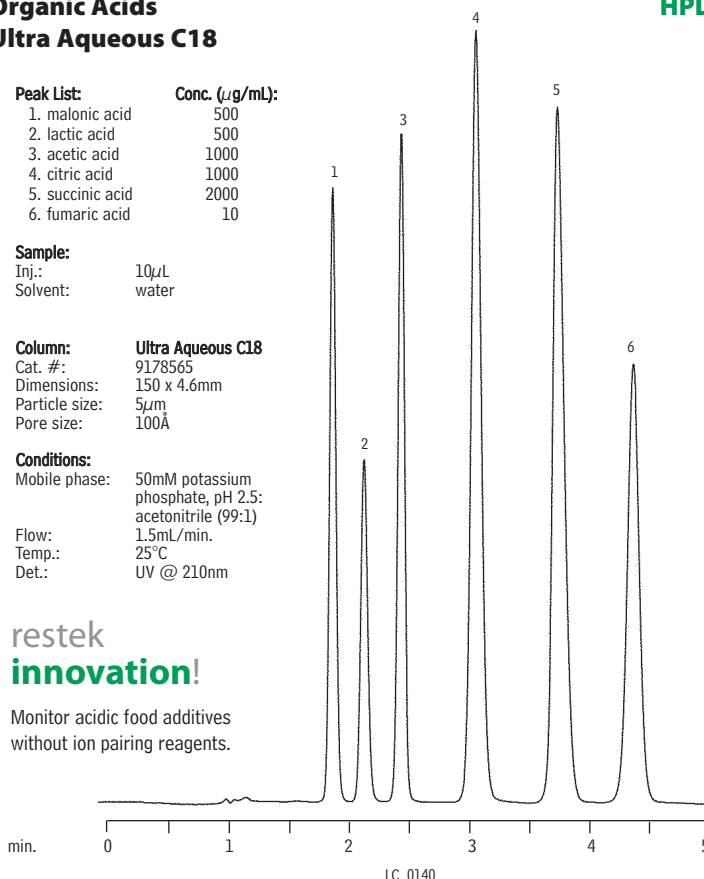
HPLC

Peak List:	Conc. (µg/mL):
1. malonic acid	500
2. lactic acid	500
3. acetic acid	1000
4. citric acid	1000
5. succinic acid	2000
6. fumaric acid	10

Sample:
Inj.: 10 µL
Solvent: water

Column: **Ultra Aqueous C18**
Cat. #: 9178565
Dimensions: 150 x 4.6mm
Particle size: 5 µm
Pore size: 100 Å

Conditions:
Mobile phase: 50mM potassium phosphate, pH 2.5: acetonitrile (99:1)
Flow: 1.5 mL/min.
Temp.: 25°C
Det.: UV @ 210nm



restek **innovation!**

Monitor acidic food additives without ion pairing reagents.

Organic Acids in Fruit Juice

Allure™ Organic Acids

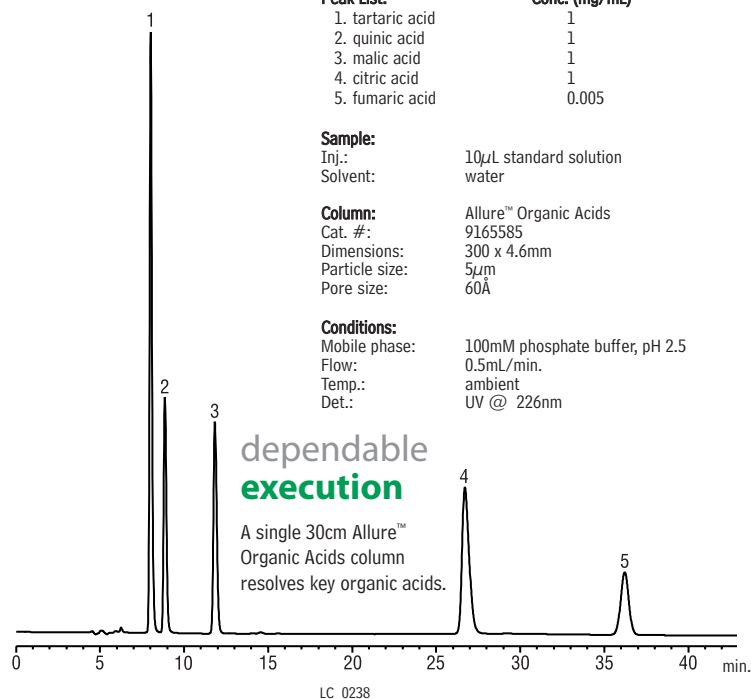
HPLC

Peak List:	Conc. (mg/mL)
1. tartaric acid	1
2. quinic acid	1
3. malic acid	1
4. citric acid	1
5. fumaric acid	0.005

Sample:
Inj.: 10µL standard solution
Solvent: water

Column: Allure™ Organic Acids
Cat. #: 9165585
Dimensions: 300 x 4.6mm
Particle size: 5µm
Pore size: 60Å

Conditions:
Mobile phase: 100mM phosphate buffer, pH 2.5
Flow: 0.5mL/min.
Temp.: ambient
Det.: UV @ 226nm



The organic acid content of fruit juices, such as cranberry and grape, can be determined using AOAC method 986.13. Because several of the acids are extremely difficult to resolve, this procedure calls for two reversed phase C18 columns in series, on a 100% aqueous mobile phase.

A single 30cm Allure™ Organic Acids column effectively resolves key organic acids, such as tartaric and quinic, using the chromatographic conditions specified in AOAC method 986.13!

Organic acids also can be analyzed by GC. Shorter chain, volatile free fatty acids such as acetic, propionic, butyric, and valeric acids can be analyzed using a **Stabilwax®-DA** column, a bonded Carbowax® column specifically deactivated for acidic compounds. Direct injection generally is recommended, to avoid losing volatile low molecular weight free fatty acids through the split vent, thus improving reproducibility.

Less polar columns, such as Rtx®-1 and Rtx®-200 (page 2), can be used to separate short chain acids. However, thicker films are required to improve separation and increase sample capacity for polar compounds.

Short Chain Acids

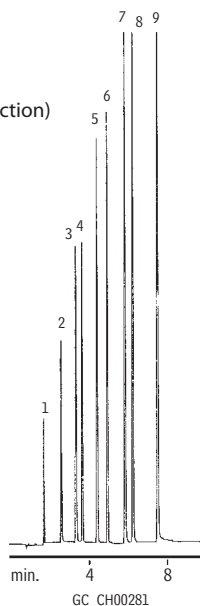
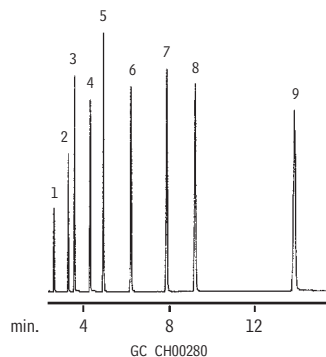
GC

Stabilwax®-DA
(split injection)

Rtx®-1
(direct injection)

Peak List:

1. acetic acid
2. propionic acid
3. isobutyric acid
4. n-butyric acid
5. isovaleric acid
6. n-valeric acid
7. isocaproic acid
8. caproic acid
9. heptanoic acid



30m, 0.25mm ID, 0.25µm Stabilwax®-DA (cat.# 11023)
1.0µL split injection of a free acid standard.
Concentration approximately 10 to 20ng/µL.

Oven temp.: 145°C
Inj. & det. temp.: 250°C
Carrier gas: hydrogen
Linear velocity: 40cm/sec.
FID sensitivity: 2 x 10⁻¹¹ AFS
Split ratio: 50:1

30m, 0.53mm ID, 5.0µm Rtx®-1 (cat.# 10179)
0.2µL injection of a 10–20ng/µL free fatty acid standard in water. Direct injection using a Uniliner® liner.

Oven temp.: 60°C to 180°C @ 15°C/min.
Inj. & det. temp.: 250°C
Carrier gas: hydrogen
Linear velocity: 50cm/sec.
(flow rate: 6cc/min.)
FID sensitivity: 4 x 10⁻¹¹ AFS

for more info

Request Application Notes **Analyze Polar Compounds by Reversed Phase HPLC, Using Ultra Aqueous C18 Column** (cat.# 59177) and **Single Column Method for HPLC Analysis of Organic Acids in Fruit Juices, Using an Allure™ Organic Acids Column** (cat.# 59530).

Preservatives

Preservation techniques are used in foods and beverages to maintain the quality of the product. Food preservation can be done by both physical and chemical means. Physical techniques might involve drying, heating, freezing, pasteurization, or irradiation; chemical techniques include adding sugar, salt, or preservatives. Several common chemicals, such as acetic acid and citric acid, can be used to prevent the growth of food-spoiling microorganisms. Calcium propionate can be used to prevent mold growth. In addition, benzoate and sorbate salts can be used as mold inhibitors in a range of food and beverage products.

Benzoate and sorbate salts can be analyzed in their protonated form (i.e., as benzoic acid and sorbic acid) by reversed phase HPLC using an **Ultra Phenyl** column and acidified water:methanol (80:20, v/v) as the mobile phase. By monitoring the UV absorbance at 245nm, sensitive detection of benzoic and sorbic acids can be achieved. For optimum sensitivity, monitor benzoic acid at 230nm and sorbic acid at 254nm.

Analyze phenolic antioxidants by reversed phase HPLC using a **Pinnacle II™ C18** column and an acidic mobile phase.

Sorbic Acid and Benzoic Acid
Ultra Phenyl

HPLC

Peak List:

1. sorbic acid
2. benzoic acid

Sample:

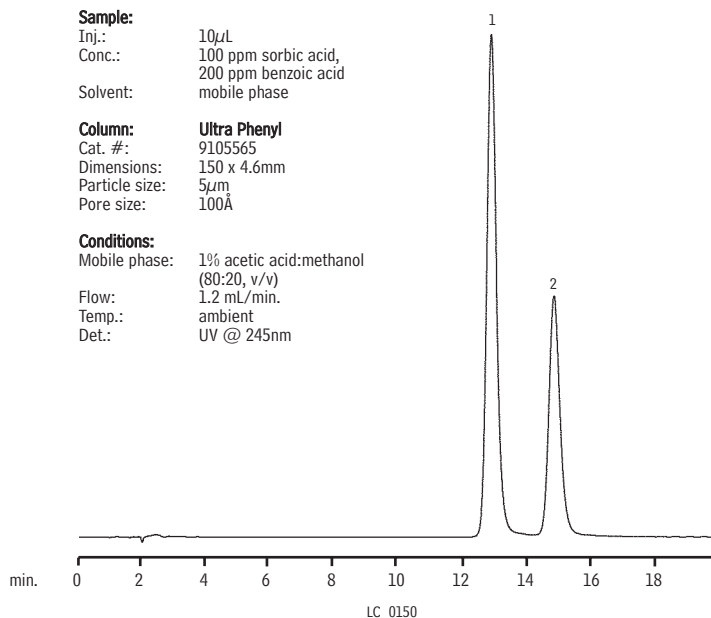
Inj.: 10µL
Conc.: 100 ppm sorbic acid,
200 ppm benzoic acid
Solvent: mobile phase

Column:

Ultra Phenyl
Cat. #: 9105565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:

Mobile phase: 1% acetic acid:methanol
(80:20, v/v)
Flow: 1.2 mL/min.
Temp.: ambient
Det.: UV @ 245nm

Phenolic Antioxidants
Pinnacle II™ C18

HPLC

Peak List:

- | Peak List: | conc.: (ppm) |
|-------------------|--------------|
| 1. propyl gallate | 168 |
| 2. TBHQ | 182 |
| 3. 2-BHA + 3-BHA | 197 |
| 4. BHT | 193 |

Sample:

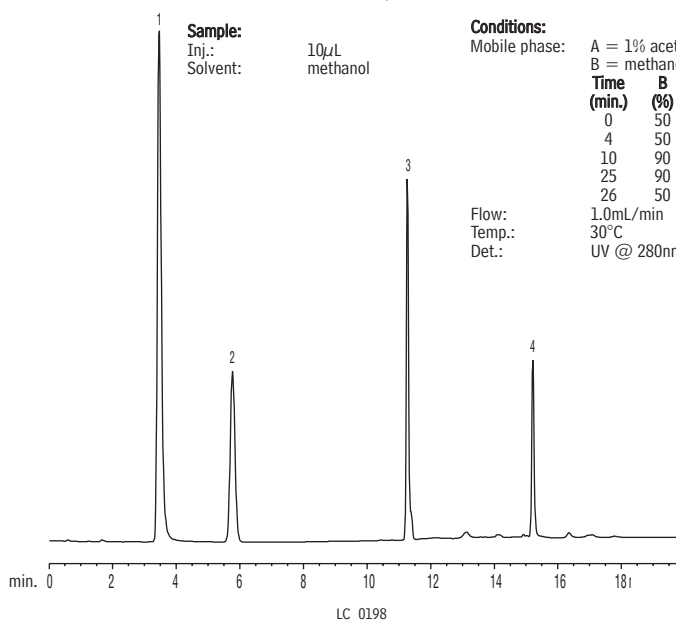
Inj.: 10µL
Solvent: methanol

Column:

Pinnacle II™ C18
Cat. #: 9214565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 110Å

Conditions:

Mobile phase: A = 1% acetic acid
B = methanol
Time (min.) B (%)
0 50
4 50
10 90
25 90
26 50
Flow: 1.0mL/min
Temp.: 30°C
Det.: UV @ 280nm



for more info

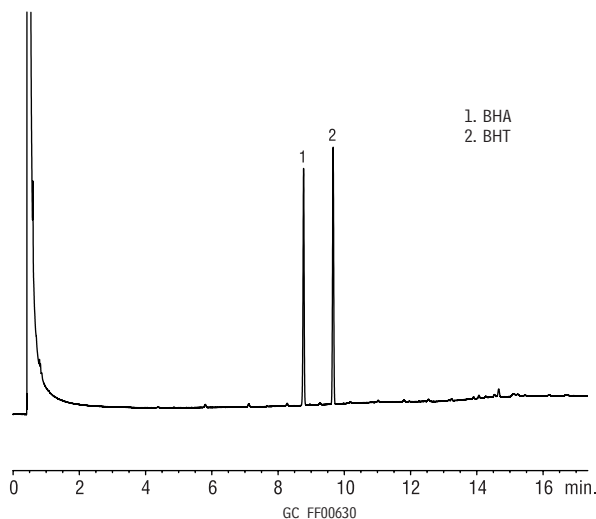
Request Flyer **High Performance Silica Products** (cat.# 59901).

Foods containing fats and oils are prone to lipid oxidation, which can promote off-flavors and limit shelf-life. To inhibit lipid oxidation, antioxidants can be added to the product. Phenolic antioxidants, including butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), propyl gallate (PG), and tert-butyl hydroquinone (TBHQ), are used in a variety of products. Phenolic antioxidants are regulated by the FDA and can be added to the product at levels up to 200ppm based on the fat content. Another approach is to use “natural” antioxidants, such as tocopherols and tocotrienols. These compounds inhibit lipid oxidation and promote general health in the consumer.

Phenolic antioxidants can be analyzed by GC using intermediate polarity **Rtx®-50** or **Rtx®-20** capillary columns. Coelutions that can occur with less polar columns can be avoided. Using direct injection and a flame ionization detector, BHA and BHT can be separated in less than 10 minutes. Using an **Rtx®-20** column, tocopherols from the unsaponified fraction of animal and vegetable fats and oils can be analyzed in their free form without derivatization. Baseline resolution is possible, with analyses times of less than 10 minutes.

BHA and BHT Rtx®-50

GC



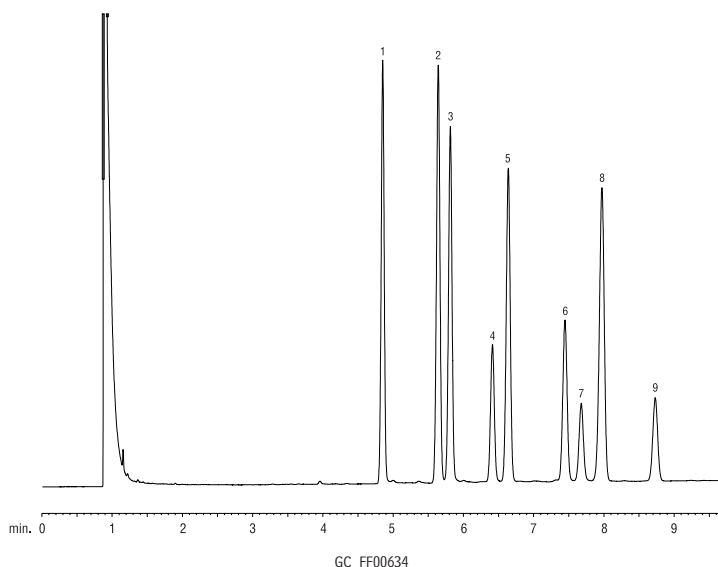
Rtx®-50, 30m, 0.53mm ID, 0.50 μ m (cat.# 10540)
 Sample: 50ppm each in methanol
 Inj.: 1.0 μ L direct injection, gooseneck splitless inlet liner, 4mm (cat.# 20798)
 Inj. temp.: 280°C
 Carrier gas: helium, constant pressure
 Linear velocity: 60cm/sec. @ 50°C
 Oven temp.: 50°C to 240°C @15°C/min. (hold 3 min.)
 Det.: FID @280°C

Tocopherols and Tocotrienols Rtx®-20

GC

1. δ -tocopherol
2. β -tocopherol
3. γ -tocopherol
4. dl- δ -tocotrienol
5. α -tocopherol
6. dl- β -tocotrienol
7. dl- γ -tocotrienol
8. hexadecyl hexadecanoate
9. dl- α -tocotrienol

Rtx®-20, 30m, 0.53mm ID, 0.5 μ m (cat.# 10340)
 Sample: 1mg/mL each component in isooctane
 Inj.: 1.0 μ L split (split ratio 20:1), 4mm inlet liner (cat.# 20814)
 Inj. temp.: 320°C
 Carrier gas: hydrogen, constant flow
 Flow rate: 5.2mL/min.
 Oven temp.: 270°C to 290°C @ 2°C/min.
 290°C to 320°C @ 10°C/min. (1 min. hold)
 Det.: FID @ 320°C



for more info

Request Application Note **HPLC Analysis of Preservatives Using Ultra Aqueous and Pinnacle II™ Columns** (cat. # 59398).

Flavors & Fragrances

Flavor consists of the taste, the aroma, and the trigeminal response to a compound. The aroma of a compound can be exceedingly complex, with several hundred volatiles playing a role. Because the nose can be extremely sensitive to some odorants, trace-level analyses may be necessary. Off-flavors can result from chemical changes in foods, microbial growth, or contamination. Chemical changes include lipid oxidation, nonenzymatic browning, and enzymatic action in the food.

Vanilla Extracts and Flavorings

One example of flavor analysis is the determination of the compounds present in vanilla extracts and flavorings. Vanilla extracts and flavorings are used in a wide range of food products, including dairy products, beverages, baked goods, and confections. In AOAC Method 990.25, flavor compounds in vanilla extract and artificial vanilla flavor are analyzed using HPLC. The analytes are separated on a C8 column and quantified by comparing their UV absorbance at 254nm to an external standard. An efficient separation can be performed using an **Ultra C8** reversed phase HPLC column and a gradient elution program, with acidified water:methanol as the mobile phase. By using a gradient program and flow rate of 1mL/min., the analysis time can be reduced to 25 minutes.

for more info

Request Application Note **Analysis of Vanillin and Ethyl Vanillin in Vanilla Flavors Using Ultra C8 Column** (cat. # 59186).

Vanillin and Ethyl Vanillin Ultra C8

HPLC

Sample:
Inj.: 10mL
Solvent: 40% ethanol

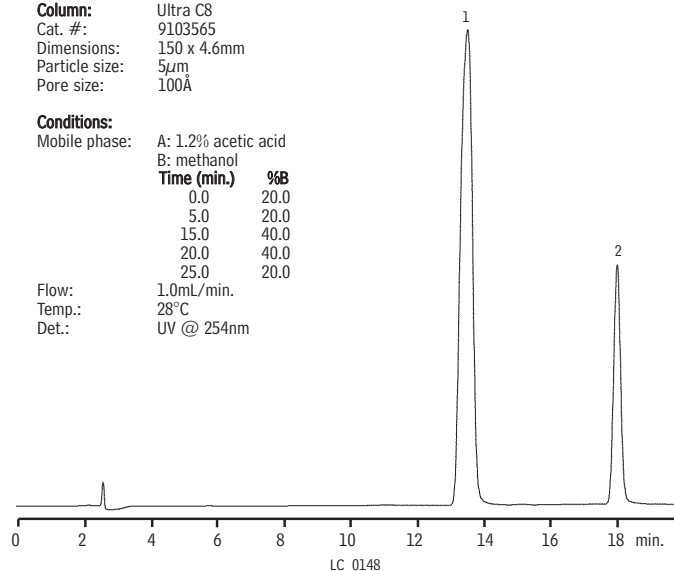
Column: Ultra C8
Cat. #: 9103565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:
Mobile phase: A: 1.2% acetic acid
B: methanol

Time (min.)	%B
0.0	20.0
5.0	20.0
15.0	40.0
20.0	40.0
25.0	20.0

Flow: 1.0mL/min.
Temp.: 28°C
Det.: UV @ 254nm

Peak List:
1. vanillin 0.12
2. ethyl vanillin 0.04



Vanilla Extract Ultra C8

HPLC

Peak List:
1. vanillin

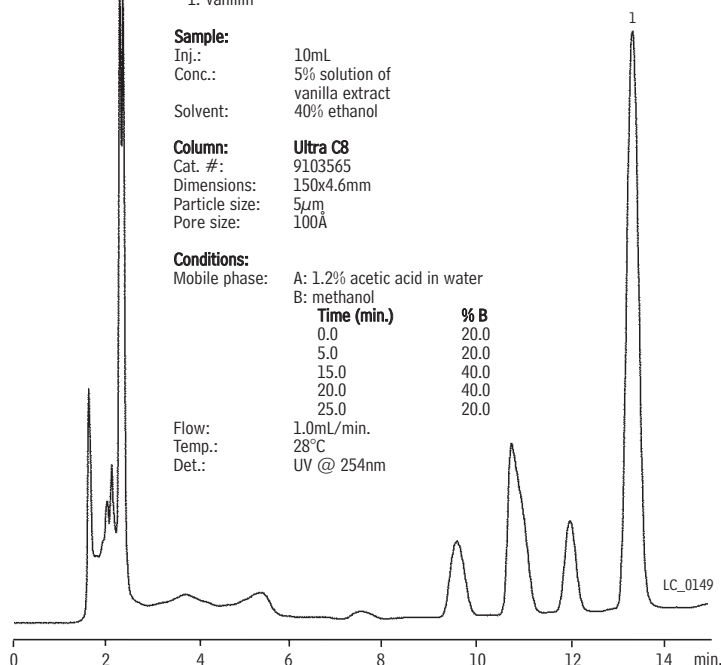
Sample:
Inj.: 10mL
Conc.: 5% solution of vanilla extract
Solvent: 40% ethanol

Column: Ultra C8
Cat. #: 9103565
Dimensions: 150x4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:
Mobile phase: A: 1.2% acetic acid in water
B: methanol

Time (min.)	% B
0.0	20.0
5.0	20.0
15.0	40.0
20.0	40.0
25.0	20.0

Flow: 1.0mL/min.
Temp.: 28°C
Det.: UV @ 254nm



Capsaicinoids: Heat Level Assay Ultra C18

HPLC

Column: Ultra C18
Cat. #: 9174565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 100Å

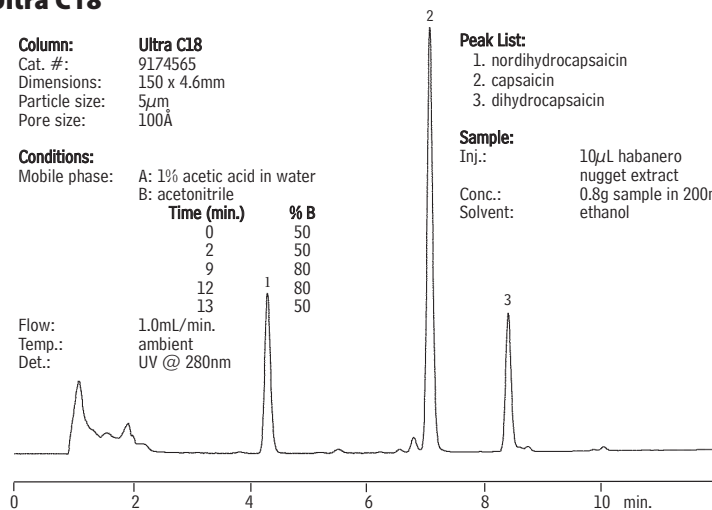
Conditions:
Mobile phase: A: 1% acetic acid in water
B: acetonitrile

Time (min.)	% B
0	50
2	50
9	80
12	80
13	50

Flow: 1.0mL/min.
Temp.: ambient
Det.: UV @ 280nm

Peak List:
1. nordihydrocapsaicin
2. capsaicin
3. dihydrocapsaicin

Sample:
Inj.: 10µL habanero
nugget extract
Conc.: 0.8g sample in 200mL
Solvent: ethanol

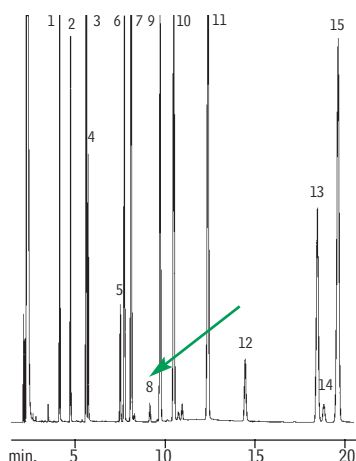


LC_0156

Flavor & Fragrance Compounds Rt-CW20M™ F&F

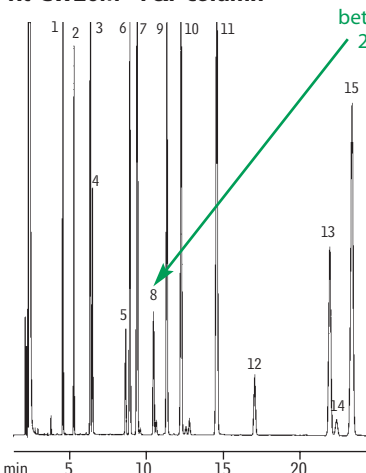
GC

Traditional Carbowax® column



1. methyl heptanoate
2. hexanol
3. methyl octanoate
4. nonanal
5. menthone
6. citronellal
7. methyl nonanoate
8. 2,3-butanediol
9. linalool
10. linalyl acetate
11. methyl decanoate
12. menthol
13. α-terpineol
14. γ-terpineol
15. methyl undecanoate

Rt-CW20M™ F&F column



GC_FF00433

50m, 0.32mm ID, 0.33µm, Carbowax®
20M or Rt-CW20M™ F&F (cat. # 12539)
On-column injection of 5ng to 150ng each
compound in methylene chloride, split 10:1

Carrier gas: hydrogen, 40cm/sec.
Inj. & det. temp.: 220°C
Oven temp.: 110°C

for more info

Request Application Note **Analyzing the Heat Level of Spicy Foods**
Using an Ultra C18 HPLC column (cat. # 59199).

Alcoholic Beverages

The chromatographic profile of alcoholic beverages consists of a wide range of compounds, including acids, alcohols, and aldehydes. GC can be used to analyze these compounds without the need for preliminary extractions.

An **Rtx®-1301** or **MXT®-1301** capillary column provides efficient separation of the volatile organic compounds in alcoholic beverages. Packed columns, such as **CarboBlack™ B** with a **5% Carbowax® 20M** phase, are an excellent alternative for these compounds. **CarboBlack™** columns are made using SilcoSmooth™ stainless steel tubing with a deactivated silica inner layer. This improves inertness, durability, and flexibility over traditional glass packed columns.

for more info

Request Technical Guide **Analyzing Alcoholic Beverages by Gas Chromatography** (cat.# 59462).

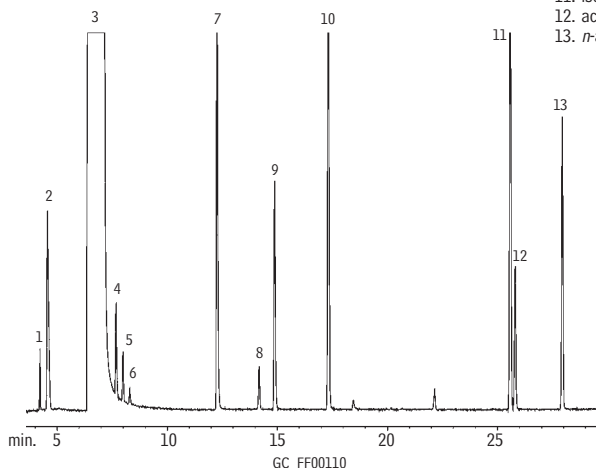
Rum
Rtx®-1301

GC

60m, 0.25mm ID, 1.4µm Rtx®-1301 (cat.# 16016)
1.0µL split injection using a Cycloplitter® liner (cat.# 20706).
Conc.: neat

Oven temp.: 35°C (hold 5 min.) to 100°C @ 1°C/min.
Inj./det. temp.: 150°C / 200°C
Carrier gas: hydrogen @ 40cm/sec.
Split ratio: 100:1

1. acetaldehyde
2. methanol
3. ethanol
4. acetone
5. ethyl formate
6. isopropanol
7. *n*-propanol
8. ethyl acetate
9. *sec*-butanol
10. isobutanol
11. isoamyl alcohol
12. active amyl alcohol
13. *n*-amyl alcohol

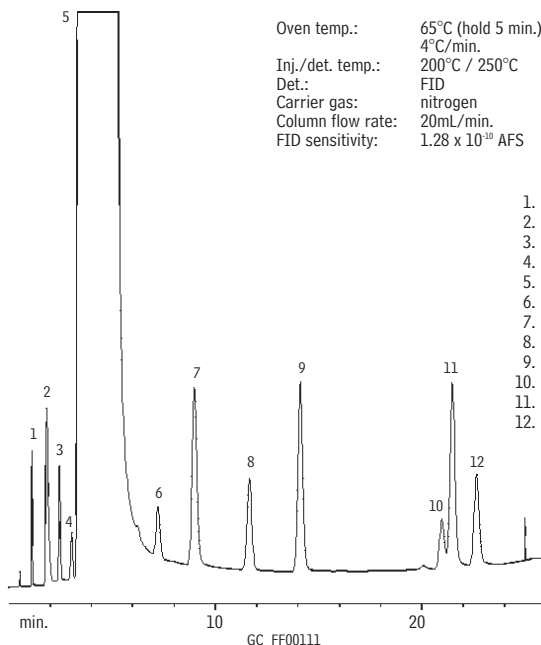
Rum
CarboBlack™ B

PACKED GC

5% Carbowax® 20M 80/120 CarboBlack™ B (cat.# 80105)
2m, 1/8" OD x 2mm ID SilcoSmooth™ tubing
0.5µL on-column injection
Conc.: neat

Oven temp.: 65°C (hold 5 min.) to 150°C @ 4°C/min.
Inj./det. temp.: 200°C / 250°C
Det.: FID
Carrier gas: nitrogen
Column flow rate: 20mL/min.
FID sensitivity: 1.28 x 10⁻¹⁰ AFS

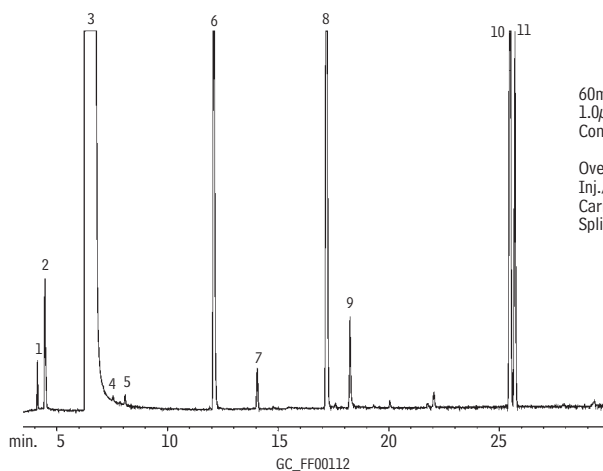
1. acetaldehyde
2. methanol
3. acetone
4. ethyl formate
5. ethanol
6. ethyl acetate
7. *n*-propanol
8. *sec*-butanol
9. isobutanol
10. active amyl alcohol
11. isoamyl alcohol
12. *n*-amyl alcohol



Scotch Rtx®-1301

GC

1. acetaldehyde
2. methanol
3. ethanol
4. acetone
5. isopropanol
6. *n*-propanol
7. ethyl acetate
8. isobutanol
9. acetic acid
10. isoamyl alcohol
11. active amyl alcohol



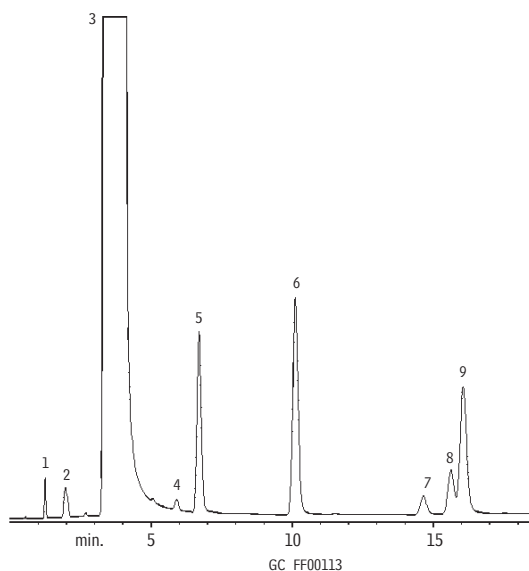
60m, 0.25mm ID, 1.4µm Rtx®-1301 (cat.# 16016)
1.0µL split injection using a Cyclosplitter® inlet liner (cat.# 20706).
Conc.: neat

Oven temp.: 35°C (hold 5 min.) to 100°C @ 1°C/min.
Inj./det. temp.: 150°C / 200°C
Carrier gas: hydrogen @ 40cm/sec.
Split ratio: 100:1

Scotch CarboBlack™ B

PACKED GC

1. acetaldehyde
2. methanol
3. ethanol
4. ethyl acetate
5. *n*-propanol
6. isobutanol
7. acetic acid
8. active amyl alcohol
9. isoamyl alcohol



5% Carbowax® 20M 80/120 CarboBlack™ B (cat.# 80105)
2m, 1/8" OD x 2mm ID Silcosmooth™ tubing
0.5µL on-column injection
Conc.: neat

Oven temp.: 70°C to 150°C @ 4°C/min.
Inj./det. temp.: 200°C / 250°C
Det.: FID
Carrier gas: nitrogen
Column flow rate: 20mL/min.
FID sensitivity: 1.28 x 10⁻¹⁰ AFS

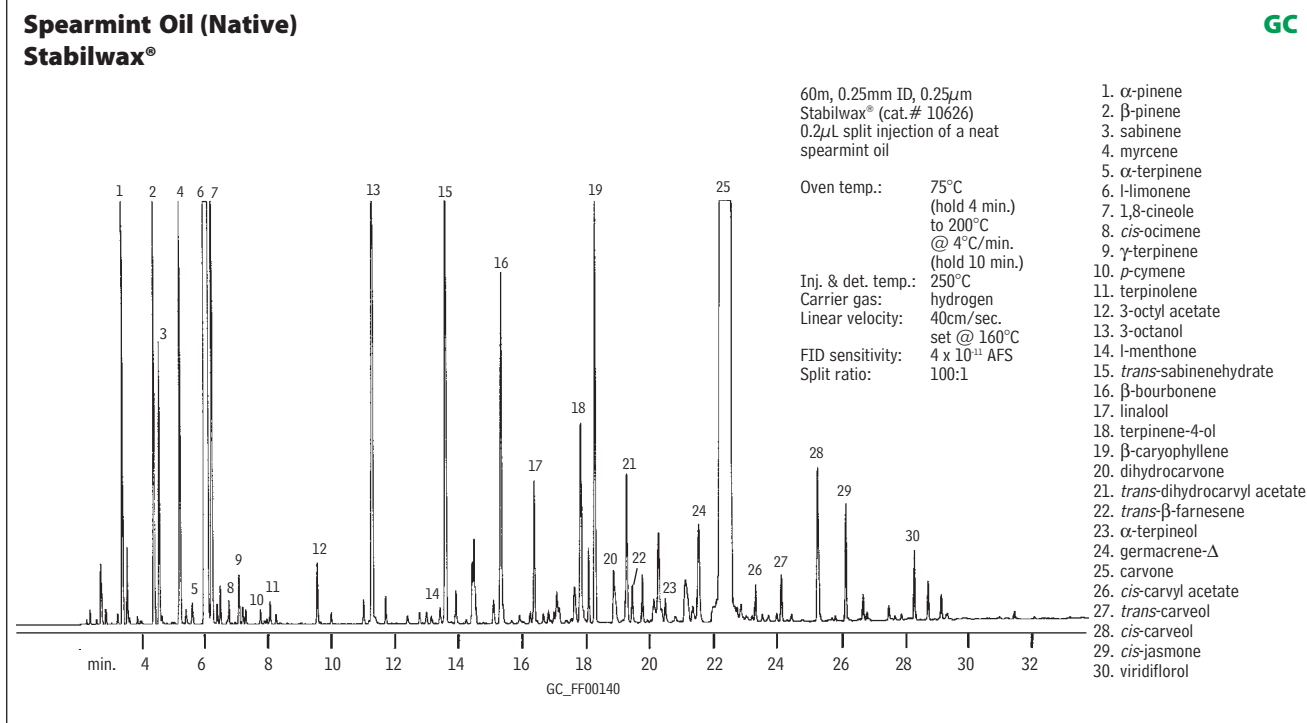
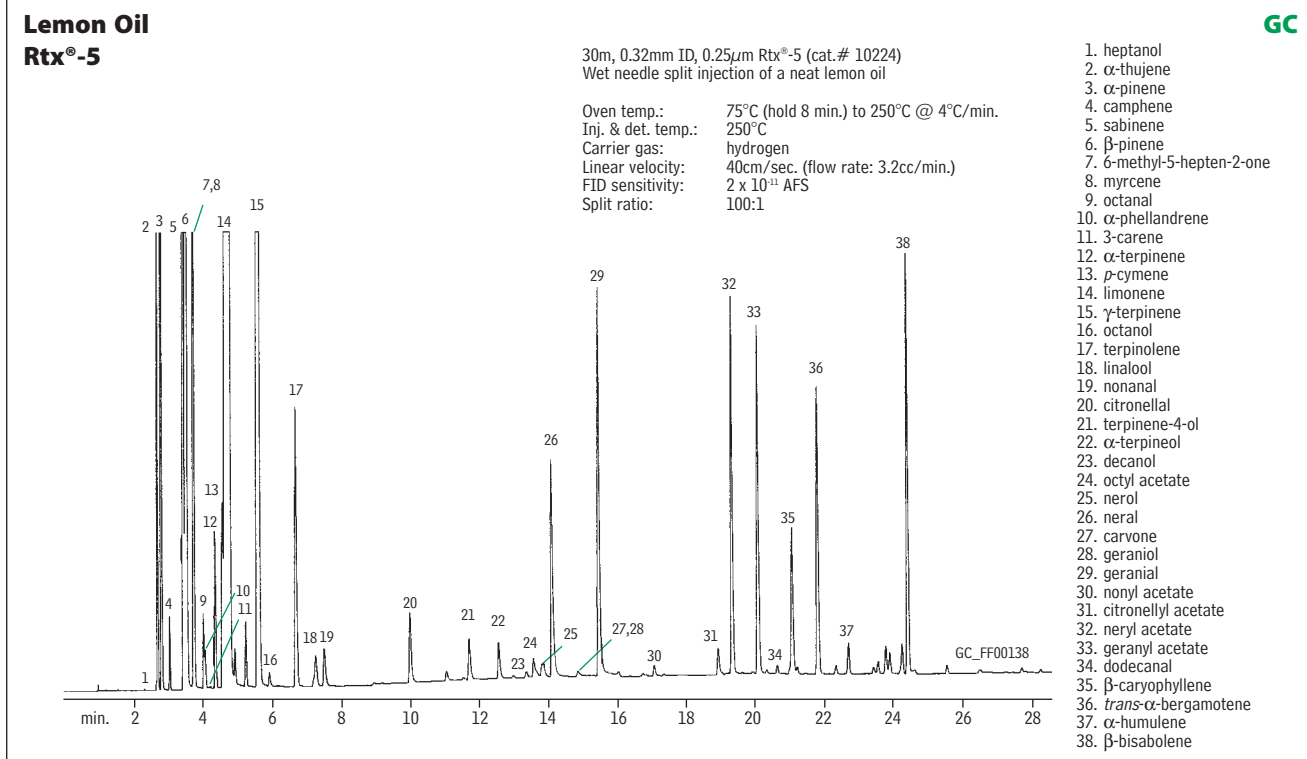
for **more** info

Request Technical Guide **Analyzing Alcoholic Beverages by Gas Chromatography** (cat.# 59462).

Essential Oils

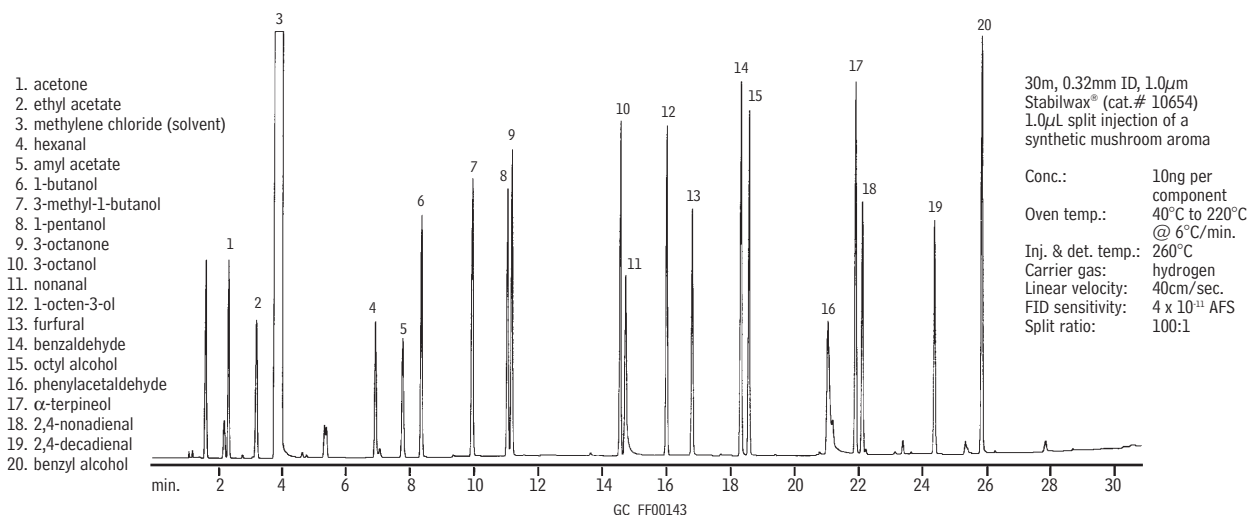
Essential oil samples are very complex; hundreds of components can be present and some are present at ppm levels. **Rtx®-1**, **Rtx®-5** and **Stabilwax®** capillary GC columns are very effective for these analyses. A comprehensive list of retention times for flavor & fragrance compounds on **Rtx®-1** and **Stabilwax®** columns is on pages 28–29.

To determine the enantiomeric ratios of volatile components in essential oils, see Chiral Separations (pages 23–26).



Synthetic Mushroom Aroma Stabilwax®

GC



Chiral Separations

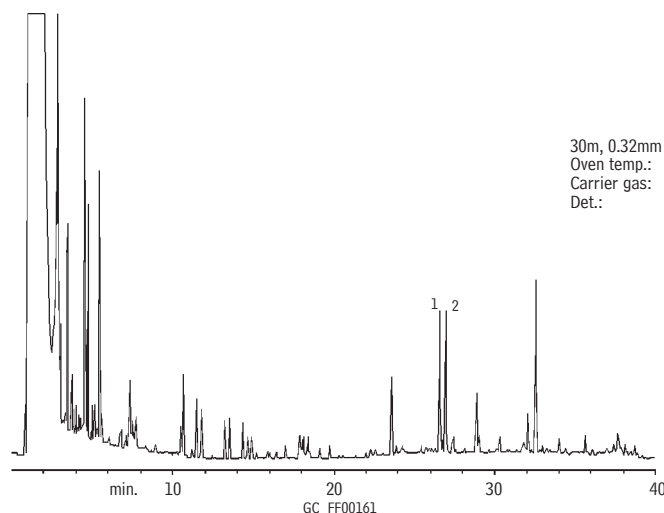
Chiral chromatography is the separation of enantiomeric compounds, which are mirror images of each other that are not superimposable. Common liquid stationary phases used in GC typically are not able to resolve enantiomeric compounds. However, the addition of derivatized cyclodextrin molecules to common stationary phases makes such separations possible. Restek's five chiral capillary columns incorporate various combinations of alkylated β -cyclodextrins into a cyanopropyl-dimethyl siloxane liquid stationary phase. The unique combinations of cyclodextrins allow analysis of a wide range of enantiomeric compounds.

Chiral capillary chromatography is a relatively new technique for determining the enantiomeric ratios of volatile components in essential oils. Enantiomeric ratios can be used for determining the authenticity of an essential oil or for characterizing regional differences among oils. The separation of enantiomeric compounds in flavor and fragrance samples can be optimized through column selection. Each of the five Restek chiral columns offers a different selectivity. The **Rt- β DEXsp™** column is optimized for menthol analysis, while the **Rt- β DEXsa™** column provides the best separation for 1-octen-3-ol, carvone, camphor, 1-phenylethanol, β -citronellol, and rose oxides. **Rt- β DEXsm™** and **Rt- β DEXse™** columns, used in combination, provide the best resolution for *cis*- and *trans*-linalool oxides, linalool, and linalyl acetate. The **Rt- β DEXcst™** column is ideal for semivolatile chiral compounds, including the irone isomers and γ - and δ -lactones.

Raspberry Flavor Rt- β DEXsa™

GC

1. (R)- α -ionone
2. (S)- α -ionone



Chiral Separations

Flavor chemists can use chiral chromatography to monitor the ratios of various enantiomeric compounds. γ -lactones, for example, can be monitored to determine if a peach flavor has been adulterated. Ethyl-2-methylbutyrate and 2-methylbutyrate are important contributors to apple flavor, and both are naturally present in predominantly the (S) form in apple juices. The enantiomers of these two compounds can be resolved on an **Rt- β DEXsm™** column.

for **more** info

Request **Chiral Column Technical Guide** (cat.# 59889).

tech tip

To optimize chiral separations, use:

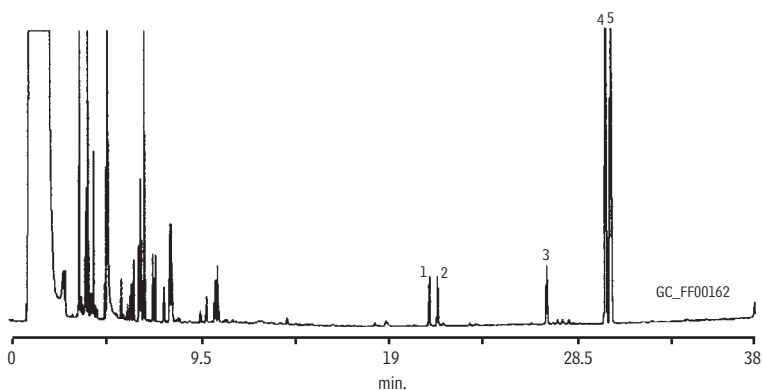
- 1) Faster linear velocities (80cm/sec.) with hydrogen carrier gas.
- 2) Slower temperature ramp rates (1–2°C/min.).
- 3) Appropriate minimum operating temperature (40 or 60°C).
- 4) On-column concentrations of 50ng or less.

Peach/Vanilla Flavor Rt- β DEXsa™

GC

1. (R)- γ -octalactone
2. (S)- γ -octalactone
3. (R)- γ -decalactone
4. (R)- γ -undecalactone
5. (S)- γ -undecalactone

30m, 0.32mm ID, 0.25 μ m Rt- β DEXsa™ (cat.# 13108)
Oven temp.: 60°C (hold 2 min.) to 100°C @ 15°C/min.
to 220°C @ 3°C/min.
Carrier gas: helium, 60cm/sec. set @ 60°C
Det.: MS @ 220°C

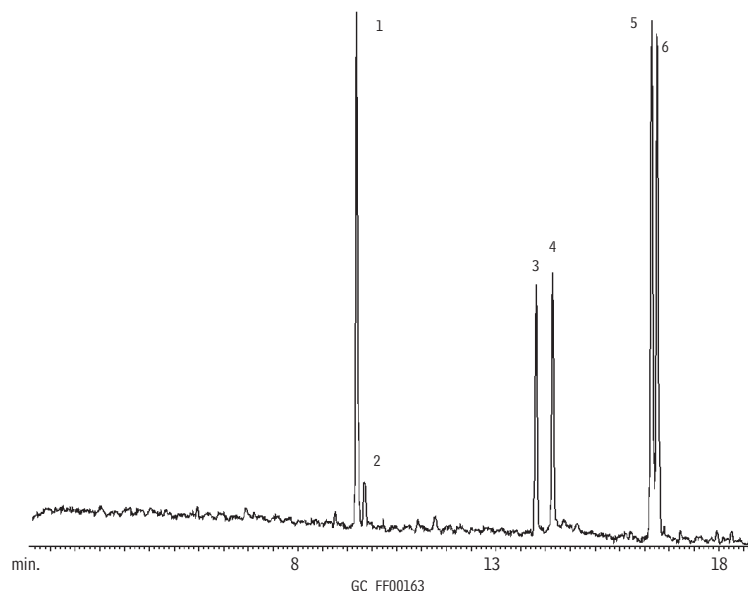


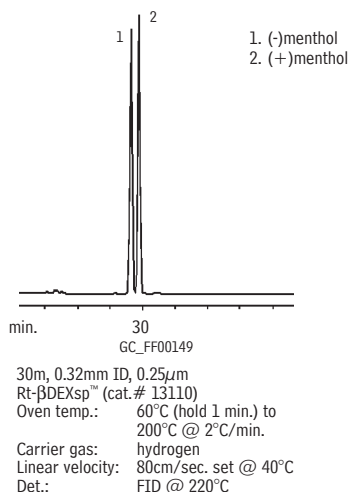
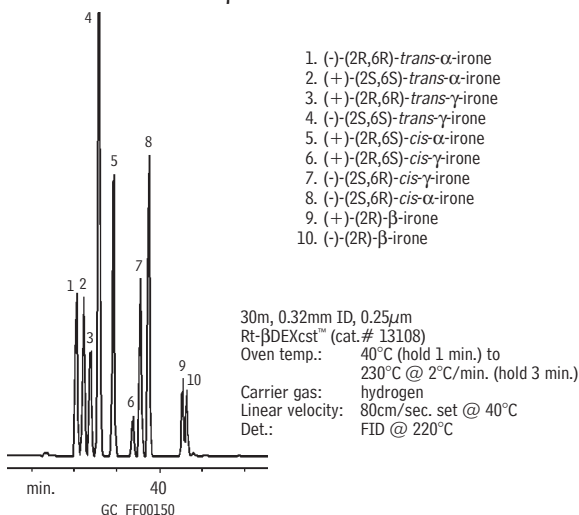
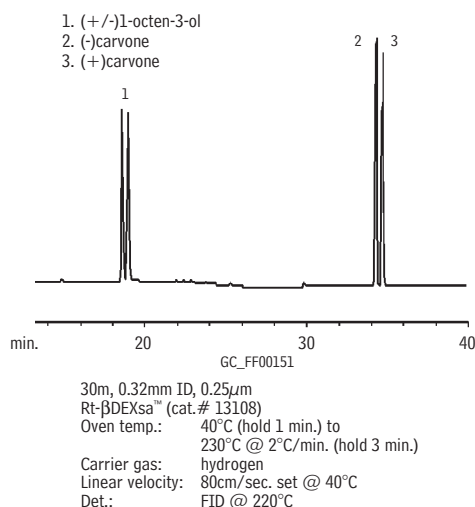
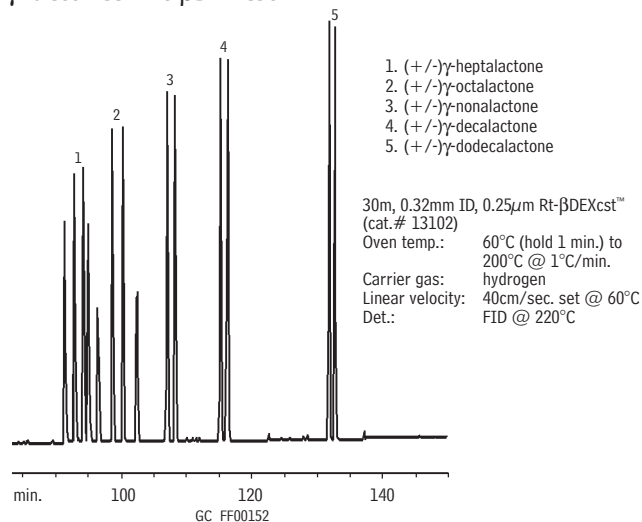
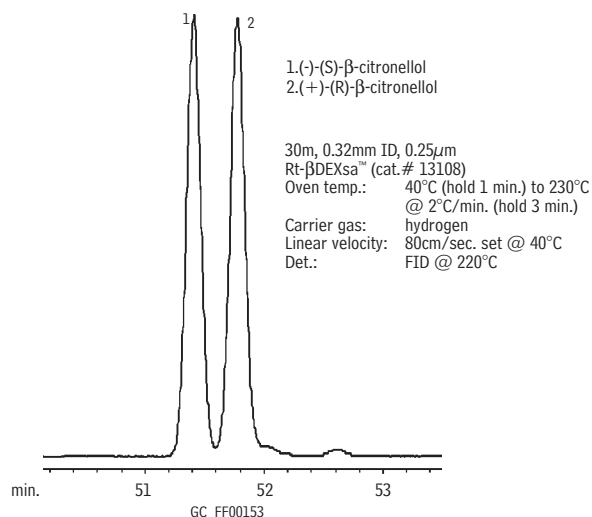
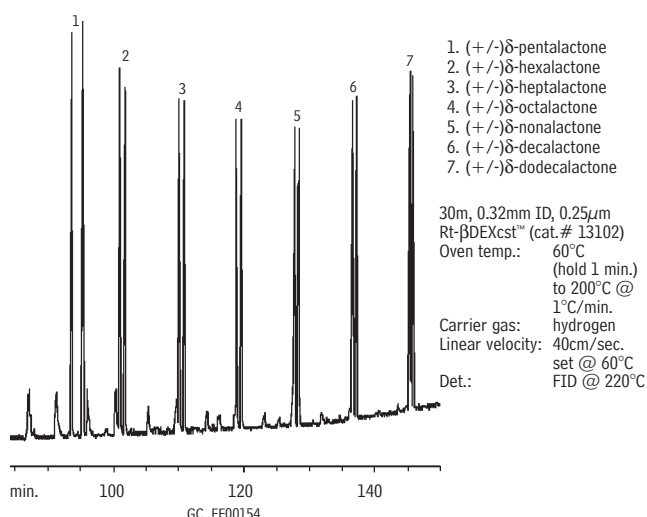
Bergamot Flavor Rt- β DEXse™

GC

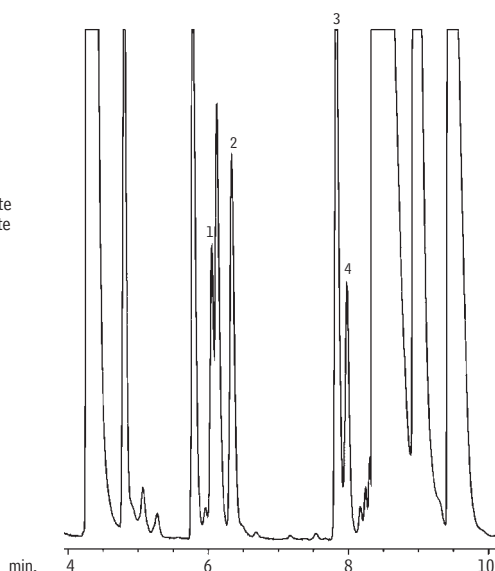
1. (S)-limonene
2. (R)-limonene
3. (R)-linalool
4. (S)-linalool
5. (R)-linalyl acetate
6. (S)-linalyl acetate

30m, 0.32mm ID, 0.25 μ m Rt- β DEXse™ (cat.# 13106)
Oven temp.: 40°C (hold 1 min.) to 200°C @ 4°C/min.
Carrier gas: helium, 60cm/sec. set @ 40°C
Det.: MS @ 220°C



Menthol - Rt- β DEXsp™Irene Isomers - Rt- β DEXcst™1-octen-3-ol and carvone - Rt- β DEXsa™ γ -lactones - Rt- β DEXcst™ β -citronellol - Rt- β DEXsa™ δ -lactones - Rt- δ DEXcst™

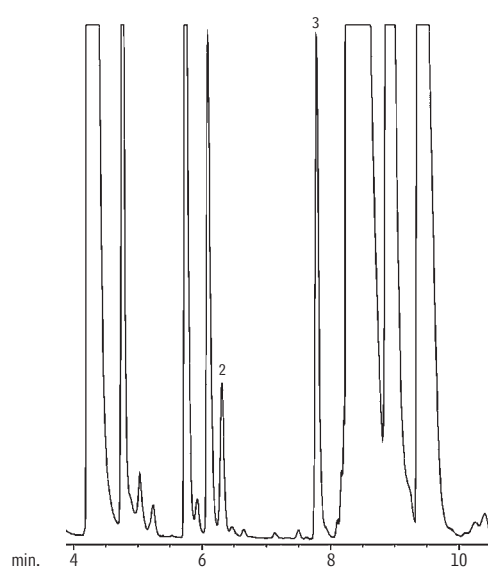
Apple Juice with Added Standards
Rt- β DEXsm™



1. (R)-ethyl 2-methylbutyrate
2. (S)-ethyl 2-methylbutyrate
3. (R)-2-methylbutyrate
4. (S)-2-methylbutyrate

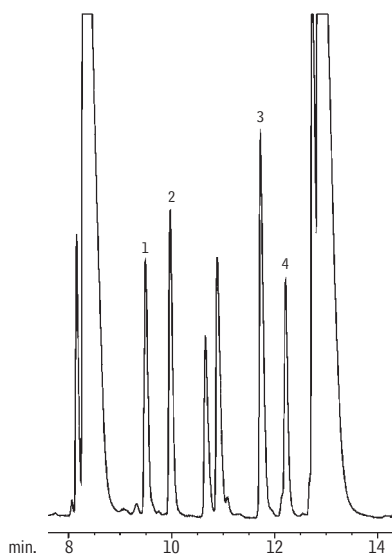
GC_FF00155
30m, 0.32mm ID, 0.25 μ m Rt- β DEXsm™ (cat.# 13104)
On-column conc. (standards): ~50ng

Apple Juice
Rt- β DEXsm™



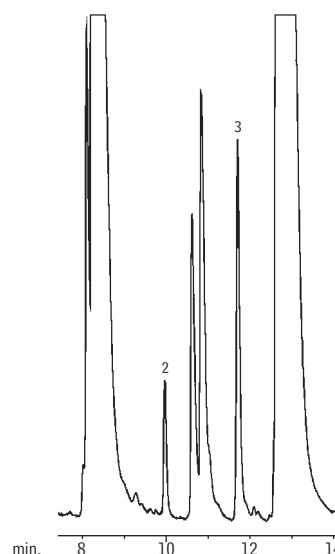
GC_FF00156
30m, 0.32mm ID, 0.25 μ m Rt- β DEXsm™ (cat.# 13104)

Apple Juice with Added Standards
Rt- β DEXse™



GC_FF00157
30m, 0.32mm ID, 0.25 μ m Rt- β DEXse™ (cat.# 13106)
On-column conc. (standards): ~50ng

Apple Juice
Rt- β DEXse™

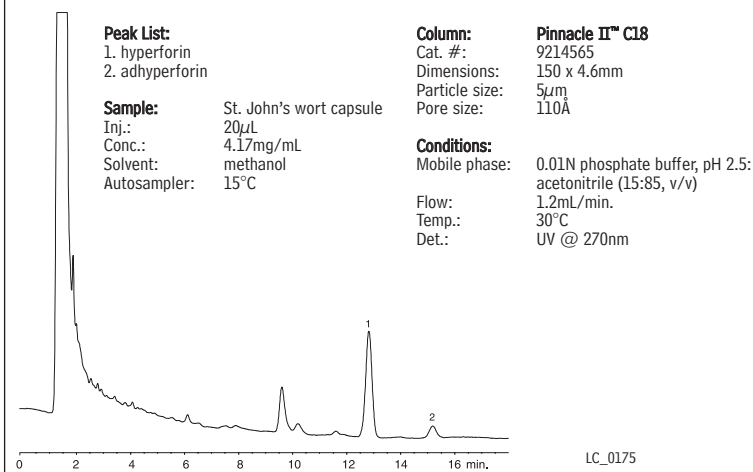


GC_FF00158
30m, 0.32mm ID, 0.25 μ m Rt- β DEXse™ (cat.# 13106)

1.0 μ L split injection.
Oven temp.: 40°C (hold 1 min.) to 220°C @ 2°C/min.
Inj. & det. temp.: 220°C
Carrier gas: hydrogen
Linear velocity: 80cm/sec.

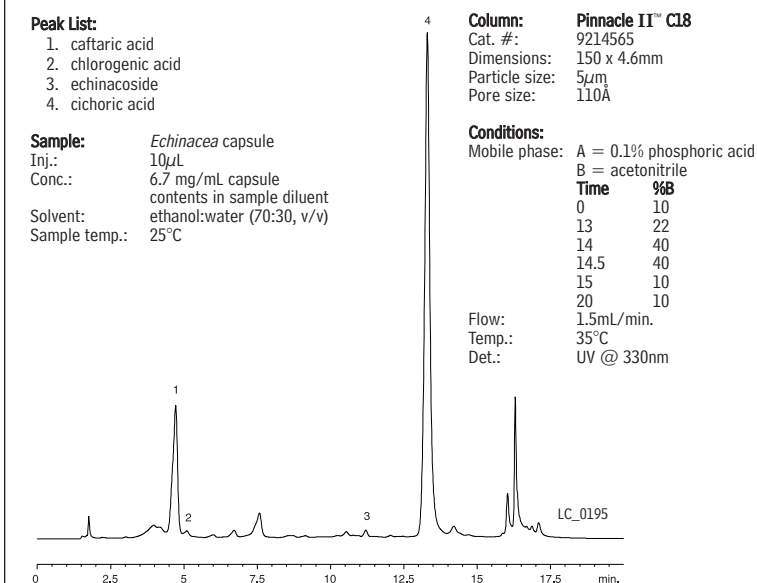
Hyperforin in St. John's Wort Pinnacle II™ C18

HPLC



Phenolics in Echinacea Pinnacle II™ C18

HPLC



Dietary Supplements

Over the past decade, the nutraceutical industry has seen rapid growth as more people add flow-ers, leaves, roots, and fruits of botanicals to their diets in hope of gaining health benefits.

Herbal products are very complex, often containing hundreds of compounds, and it is not always clear which compounds are responsible for the beneficial properties. Marker compounds—phytochemicals that have been identified and are known to have some relationship to the reported health benefit—can be evaluated qualitatively to identify a raw material or to verify purity. To determine the concentration or strength of a material, quantitative analysis is necessary.

Hyperforin in St. John's Wort

According to the Institute for Nutraceutical Advancement (INA), INA Method 112.001—the HPLC analysis of hyperforin and adhyperforin in St. John's wort—the samples are extracted with methanol in an ultrasonic bath. Chromatographic separation is performed on a C18 reversed phase column with acetonitrile and phosphate buffer as the mobile phase. Analysis of encapsulated St. John's wort using a Pinnacle II™ C18 column shows excellent peak shapes for the active ingredients in this herbal product.

Phenolics in Echinacea

The active compounds in *echinacea* are thought to be caffeic acid derivatives such as caftaric acid, cichoric acid, chlorogenic acid, and echinacoside.

Conclusion

Gas and liquid chromatography are powerful tools for the analysis of foods, flavors, and fragrances. This technical guide describes columns and analytical conditions that will help you achieve more accurate identification and quantitation of target analytes. However, if you have questions, please don't hesitate to contact Restek's technical service by e-mail (support@restekcorp.com) or by phone (extension 4), or contact your Restek representative. We will do everything we can to help you find a solution.

References

1. Fennema, O. R. Food Chemistry (1996), 3rd edition.
2. Bensinger, M. "How Hot is that 'Devil' Sauce?" in Fiery Foods Magazine (1997), Sept/Oct.
3. Brandt, Laura. "The Creation and Use of Vanilla", Food Product Design (1996), editorial archives.
4. AOAC Official Methods of Analysis (2000), 17th edition, AOAC International.
5. Official Methods and Recommended Practices (1998), 5th edition, American Oil Chemists' Society.
6. AACC Approved Methods (2000), 10th edition, American Association of Cereal Chemists.

References are not available from Restek.

for more info

Request Applications Note **Analyzing Nutraceutical Products by Liquid and Gas Chromatography** (cat.# 59364).

Flavor and Fragrance Compounds Retention Time Index

Retention time data collected using 60m, 0.25mm ID, 0.25µm Rtx®-1 and Stabilwax® columns.

Oven temp.: 100°C to 260°C @ 4°C/min.; **Carrier gas:** helium; **Linear velocity:** 27.2cm/sec. @ 100°C; **Dead time:** 3.68 min. @ 100°C.

Component	Retention Time (min.)	
	Rtx®-1	Stabilwax®
isopropyl alcohol	3.66	3.93
allyl alcohol	3.67	4.05
<i>tert</i> -butyl alcohol	3.68	3.89
1-propanol	3.70	4.13
3-buten-2-ol	3.71	4.12
ethyl formate	3.71	3.84
acetone	3.72	3.86
methyl acetate	3.75	3.86
2-butanol	3.76	4.03
propyn-1-ol	3.77	6.26
<i>tert</i> -amyl alcohol	3.79	4.14
isobutyraldehyde	3.79	3.84
2-methyl-3-buten-2-ol	3.82	4.12
methyl ethyl ketone	3.84	3.83
<i>cis</i> -2-buten-1-ol	3.84	5.01
ethyl acetate	3.86	3.90
propyl formate	3.87	3.83
2-methylfuran	3.88	3.87
isobutyl alcohol	3.89	4.72
methyl propanoate	3.89	4.03
3-buten-1-ol	3.91	4.76
3-methyl-2-butanol	3.92	4.25
2-butenal	3.94	4.26
2-pentanol	3.95	4.42
isopropyl acetate	3.96	4.03
1-butanol	3.96	4.57
neopentanol	3.99	4.60
methyl isobutyrate	4.03	4.08
2-pentanone	4.03	3.95
isoamyl alcohol	4.04	4.96
allyl acetate	4.05	4.14
ethyl acrylate	4.06	4.08
3-pentanone	4.07	4.08
pentanal	4.07	4.08
<i>tert</i> -butyl acetate	4.09	3.93
pinacolone	4.09	4.00
propyl acetate	4.10	4.04
2-ethylfuran	4.10	3.99
2,5-dimethylfuran	4.12	3.98
3-methyl-1-butanol	4.18	4.88
3-penten-2-one	4.18	4.45
2-methyl-1-butanol	4.20	4.88
pinacol alcohol	4.20	4.38
thiophene	4.21	4.16
methylisobutylketone (MIBK)	4.22	4.15
methyl butanoate	4.22	4.09
2-methyl-3-pentanone	4.27	4.15
ethyl isobutyrate	4.28	4.03
<i>cis</i> -2-penten-1-ol	4.29	5.85
3-methyl-3-pentanol	4.29	4.37
1-pentanol	4.30	5.24
3-hexanone	4.30	4.28
3-methyl-2-buten-1-ol	4.32	5.81
isobutyl acetate	4.34	4.13
butyrolactone	4.34	7.07
2-methyl-3-pentanol	4.36	4.59

Component	Retention Time (min.)	
	Rtx®-1	Stabilwax®
2-hexanone	4.41	4.11
ethyl butanoate	4.41	4.18
cyclopentanol	4.42	5.74
cyclopentanone	4.46	4.96
2,4-dimethyl-2-pentanol	4.47	4.45
3-hexanol	4.47	4.84
2-hexanol	4.48	5.01
4-methyl-3-penten-2-one	4.48	4.61
hexanal	4.48	4.41
2,4-dimethyl-3-pentanone	4.49	3.95
pyridine	4.50	4.91
propyl propanoate	4.51	4.22
<i>a</i> -angelicalactone	4.51	7.26
butyl acetate	4.54	4.29
methyl pentanoate	4.61	4.36
furfural	4.64	7.65
2,2-dimethyl-3-pentanol	4.65	4.63
2-methyl-1-pentanol	4.65	5.63
4-hexen-3-one	4.67	4.88
isopropyl butyrate	4.72	4.19
furfuryl alcohol	4.74	11.07
2,4-dimethyl-3-pentanol	4.75	4.76
<i>trans</i> -2-hexenal	4.78	5.08
pinacol	4.79	7.91
ethyl-2-methyl butanoate	4.80	4.26
2-ethyl-1-butanol	4.80	5.72
<i>trans</i> -2-hexenol	4.82	6.56
5-methyl-2-hexanone	4.84	4.54
1-hexanol	4.92	6.16
3-ethyl-3-pentanol	4.94	4.94
isoamyl acetate	4.98	4.49
<i>cis</i> -3-hexen-1-ol	4.98	6.58
4-heptanone	4.99	4.45
<i>trans</i> -2-hexen-1-ol	5.01	6.81
anisole	5.06	6.13
3-heptanone	5.08	4.62
1,3-xylene	5.09	4.67
1,4-xylene	5.09	4.64
2-heptanone	5.10	4.85
4-heptanol	5.14	5.45
propyl butyrate	5.16	4.49
ethyl pentanoate	5.17	4.54
cyclohexanone	5.19	5.85
2-heptanol	5.20	5.63
heptanal	5.21	4.35
butyl propionate	5.24	4.56
amyl acetate	5.29	4.73
1,2-xylene	5.30	4.93
nonane	5.32	3.80
isobutyl isobutyrate	5.34	4.39
methyl hexanoate	5.41	3.81
tetrahydro-2-fufanmethanol	5.50	8.53
d-valerolactone	5.51	9.57
cumene	5.62	4.84
5-methyl-3-heptanone	5.65	4.94
ethyl amyl ketone	5.65	4.94

Component	Retention Time (min.)	
	Rtx®-1	Stabilwax®
5-methylfurfural	5.73	9.64
<i>a</i> -pinene	5.81	4.84
benzaldehyde	5.90	8.76
tricyclene	5.91	4.16
1-heptanol	5.95	7.54
camphene	6.02	4.24
2,6-dimethyl-4-heptanone	6.03	4.81
1-octen-3-ol	6.09	7.33
furfuryl acetate	6.09	8.77
3-octanone	6.16	5.35
2-octanone	6.20	6.35
<i>sec</i> -butylbenzene	6.28	5.37
<i>tert</i> -butylbenzene	6.32	5.15
myrcene	6.32	4.53
butyl butyrate	6.34	5.02
<i>b</i> -pinene	6.38	4.40
octanal	6.39	5.73
2-octanol	6.39	7.00
hexyl acetate	6.49	5.41
decane	6.58	4.08
2-methylanisole	6.60	6.97
<i>a</i> -phellandrene	6.69	6.13
3-methylanisole	6.74	7.39
4-methylanisole	6.78	7.42
2-ethyl-1-hexanol	6.85	8.10
benzyl alcohol	6.86	16.48
3-carene	6.89	4.67
<i>p</i> -cymene	6.91	5.38
<i>a</i> -terpinene	6.93	12.38
limonene	7.09	4.84
salicylaldehyde	7.09	11.93
camphor	7.11	8.77
<i>trans</i> -ocimene	7.13	4.72
1,8-cineole	7.16	5.06
eucalyptol	7.16	5.06
<i>cis</i> -ocimene	7.21	5.06
<i>a</i> -methylbenzylalcohol	7.34	18.34
<i>p</i> -cresol	7.45	21.40
<i>g</i> -terpinene	7.54	5.26
1-octanol	7.55	9.37
2,6-dimethylanisole	7.55	7.16
5-nonanone	7.57	6.39
tetrahydrofurfuryl acetate	7.60	9.84
fenchone	7.68	7.08
linalool oxide	7.81	7.56
3-nonanone	7.82	6.38
2-nonanone	7.89	7.17
methyl benzoate	8.03	10.72
linalool	8.13	9.02
2-nonanol	8.15	8.65
nonanal	8.16	6.86
terpinyl acetate	8.18	12.51
maltol	8.23	18.48

Retention time data collected using 60m, 0.25mm ID, 0.25µm Rtx®-1 and Stabilwax® columns.

Oven temp.: 100°C to 260°C @ 4°C/min.; **Carrier gas:** helium; **Linear velocity:** 27.2cm/sec. @ 100°C; **Dead time:** 3.68 min. @ 100°C.

Component	Retention Time (min.)		Component	Retention Time (min.)		Component	Retention Time (min.)	
	Rtx®-1	Stabilwax®		Rtx®-1	Stabilwax®		Rtx®-1	Stabilwax®
<i>trans</i> -sabinene hydrate	8.37	9.06	r-carvone	11.48	13.28	carvone hydrate	16.25	27.44
2,4-dimethylanisole	8.39	8.45	s-carvone	11.51	13.36	tetradecane	16.38	6.95
2,5-dimethylanisole	8.39	8.42	geraniol	11.74	15.84	coumarin	16.54	29.90
undecane	8.41	4.38	<i>trans</i> -cinnamaldehyde	11.97	20.50	<i>cis</i> -carvyl propionate	16.63	14.68
a-thujone	8.46	15.01	citral b	12.12	13.25	a-ionone	16.88	16.23
methyl octanoate	8.57	6.69	neral	12.12	13.25	<i>trans</i> -cinnamyl acetate	16.88	23.12
b-thujone	8.68	7.63	propyl benzoate	12.16	13.82	ethyl vanillin	17.08	31.44
2,3-dimethylanisole	8.87	9.30	1-decanol	12.36	13.99	isoeugenol	17.09	27.57
citronellal	9.19	7.99	perillaldehyde	12.37	14.52	3-methyl- <i>p</i> -anisaldehyde	17.23	20.08
benzyl acetate	9.31	13.02	citronellyl formate	12.38	10.69	b-caryophyllene	17.32	10.56
menthone	9.42	7.97	<i>trans</i> -menthyl acetate	12.56	9.01	<i>trans</i> -carvyl propionate	17.65	15.76
borneol	9.53	12.52	indole	12.57	29.33	a-methylcinnamic acid	18.15	4.64
ethyl benzoate	9.62	11.74	<i>trans</i> -anethole	12.67	15.41	a-humulene	18.29	12.05
1-nonanol	9.70	11.56	cumin alcohol	12.68	21.88	2,3-dimethylanisaldehyde	18.46	25.74
isoborneol	9.72	11.69	thymol	12.71	23.82	b-ionone	18.57	18.37
menthofuran	9.73	8.14	2-undecanone	12.81	10.45	vanillin acetate	18.93	30.85
isomenthone	9.75	8.57	carvacrol	12.87	24.56	pentadecane	19.34	8.60
neomenthol	9.85	10.22	bornyl acetate	12.88	10.09	valencene	19.40	13.15
a-terpineol	9.91	5.58	<i>trans</i> -cinnamyl alcohol	12.94	26.11	2,5-dimethylanisaldehyde	19.50	21.72
menthol	9.95	11.16	<i>cis</i> -menthyl acetate	13.02	9.62	6-methylcoumarin	20.22	32.66
dihydrocarveol	10.09	13.64	perillyl alcohol	13.02	19.36	carvone acetate	20.50	26.09
terpinen-4-ol	10.11	10.31	tridecane	13.47	5.72	7-methylcoumarin	20.65	32.52
2-decanone	10.14	8.41	2-methylcinnamaldehyde	13.59	19.52	ethyl laurate	21.69	16.17
a-terpinolene	10.32	7.36	triacetin	13.67	21.33	caryophyllene oxide	21.88	19.47
4-allylanisole	10.35	11.75	methyl decanoate	13.69	10.26	hexadecane	22.27	10.64
estragole	10.35	11.75	<i>cis</i> -carvyl acetate	14.09	13.23	cinnamide	22.36	45.53
decanal	10.48	8.51	cumic acid	14.40	34.95	amyl cinnamaldehyde	23.08	25.95
<i>trans</i> -dihydrocarvone	10.59	23.12	g-valerolactone	14.48	10.55	<i>cis</i> - <i>trans</i> -farnesol	24.61	27.48
verbenone	10.67	12.88	citronellyl acetate	14.49	11.81	heptadecane	25.10	12.94
dodecane	10.75	4.90	eugenol	14.55	23.53	<i>trans</i> - <i>trans</i> -farnesol	25.28	28.09
<i>cis</i> -dihydrocarvone	10.80	10.80	thiazole	14.67	5.23	guaiazulene	27.04	28.98
linalyl acetate	10.93	9.36	neryl acetate	14.76	13.08	nootketone	27.69	31.89
b-citronellol	11.06	13.93	<i>trans</i> -carvyl acetate	14.88	14.05	octadecane	27.83	15.40
<i>cis</i> -nerol	11.14	13.82	dihydrocoumarin	14.91	26.02	nonadecane	30.44	17.92
carveol	11.24	16.29	geranyl acetate	15.26	13.08	eicosane	32.94	20.44
benzyl acetone	11.27	16.25	dihydrojasnone	15.34	16.24	heneicosane	35.32	22.91
citral a	11.39	12.13	vanillin	15.34	32.21	docosane	37.62	25.35
geraniol	11.39	12.13	ethyl decanoate	15.69	11.22	tricosane	39.79	27.68
cuminaldehyde	11.43	14.40	2-dodecanone	15.72	12.83	tetracosane	42.02	29.95
pulegone	11.43	11.46	<i>cis</i> -jasnone	15.75	18.34	hexacosane	47.40	34.26
<i>p</i> -anisaldehyde	11.47	20.15	<i>trans</i> -cinnamyl acid	16.07	37.16			

rtx®-1 F&F gc columns

for flavor and fragrance compounds

- Specifically tailored to meet the demanding selectivity criteria of the flavor and fragrance industry.
- Excellent thermal stability and column lifetimes.
- Stringent QA ensures column-to-column reproducibility.

See **page 30** for product listing.

Rtx®-1 (Crossbond® 100% dimethyl polysiloxane)

- General-purpose non-polar phase, ideal for flavor and fragrance compounds.
- Thermally stable to 350°C.
- Polarity similar to DB-1, SPB-1, HP-1, Ultra-1 phases.
- Equivalent to USP G1, G2, G38 phases.

Rtx®-1 (fused silica)

(Crossbond® 100% dimethyl polysiloxane)

ID	df (μm)	temp. limits	15-Meter	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	0.10	-60 to 330/350°C	10105	10108	10111		10114
	0.25	-60 to 330/350°C	10120	10123	10126		10129
	0.50	-60 to 330/350°C	10135	10138	10141		10144
	1.00	-60 to 320/340°C	10150	10153	10156		10159
0.32mm	0.10	-60 to 330/350°C	10106	10109	10112		10115
	0.25	-60 to 330/350°C	10121	10124	10127		10130
	0.50	-60 to 330/350°C	10136	10139	10142		10145
	1.00	-60 to 320/340°C	10151	10154	10157		10160
	1.50	-60 to 310/330°C	10166	10169	10172		10175
	3.00	-60 to 280/300°C	10181	10184	10187		10190
	4.00	-60 to 280/300°C		10198			
	5.00	-60 to 260/280°C	10176	10178	10180		
0.45mm	2.55	-60 to 270/290°C				10992	
0.53mm	0.10	-60 to 320/340°C	10107	10110	10113		
	0.25	-60 to 320/340°C	10122	10125	10128		
	0.50	-60 to 310/330°C	10137	10140	10143		
	1.00	-60 to 310/330°C	10152	10155	10158		
	1.50	-60 to 310/330°C	10167	10170	10173		
	3.00	-60 to 270/290°C	10182	10185	10188		10189
	5.00	-60 to 270/290°C	10177	10179	10183		10194
	7.00	-60 to 240/260°C	10191	10192	10193		
ID	df (μm)	temp. limits	10-Meter	20-Meter	40-Meter		
0.10mm	0.10	-60 to 330/350°C	41101	41102			
	0.40	-60 to 320/340°C	41103	41104			
0.18mm	0.20	-60 to 330/350°C	40101	40102	40103		
	0.40	-60 to 320/340°C	40110	40111	40112		

Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

Rtx®-1 F&F (Crossbond® 100% dimethyl polysiloxane)

- Application-specific non-polar phase for flavor and fragrance compounds.
- Thermally stable to 350°C.
- Polarity similar to HP-1 phase.

Rtx®-1 F&F (fused silica)

(Crossbond® 100% dimethyl polysiloxane)

ID	df (μm)	temp. limits	15-Meter	30-Meter	50-Meter	60-Meter
0.25mm	0.25	-60 to 330/350°C		18023		18026
	0.50	-60 to 330/350°C		18038		18041
	1.00	-60 to 320/340°C		18053		18056
0.32mm	0.25	-60 to 330/350°C		18024		18027
	0.50	-60 to 330/350°C		18039	18010	18042
	1.00	-60 to 320/340°C		18054		18057
0.53mm	0.50	-60 to 310/330°C	18037	18040		18043
	1.00	-60 to 310/330°C	18052	18055		18058
	1.50	-60 to 310/330°C	18067	18070		18073

for **more info**

Rtx®-5 (Crossbond® 5% diphenyl / 95% dimethyl polysiloxane)

- General-purpose low polarity phase.
- Thermally stable to 350°C.
- Polarity similar to DB-5, SPB-5, HP-5, Ultra-2 phases.
- Equivalent to USP G27, G36 phases.

Rtx®-5 (fused silica)

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

ID	df (μm)	temp. limits*	15-Meter	30-Meter	60-Meter	105-Meter
0.25mm	0.10	-60 to 330/350°C	10205	10208	10211	10214
	0.25	-60 to 330/350°C	10220	10223	10226	10229
	0.50	-60 to 330/350°C	10235	10238	10241	10244
	1.00	-60 to 320/340°C	10250	10253	10256	10259
0.32mm	0.10	-60 to 330/350°C	10206	10209	10212	10215
	0.25	-60 to 330/350°C	10221	10224	10227	10230
	0.50	-60 to 330/350°C	10236	10239	10242	10245
	1.00	-60 to 330/350°C	10251	10254	10257	10260
	1.50	-60 to 310/330°C	10266	10269	10272	10275
	3.00	-60 to 280/300°C	10281	10284	10287	10290
0.53mm	0.10	-60 to 320/340°C	10207	10210	10213	
	0.25	-60 to 320/340°C	10222	10225	10228	
	0.50	-60 to 310/330°C	10237	10240	10243	
	1.00	-60 to 310/330°C	10252	10255	10258	
	1.50	-60 to 310/330°C	10267	10270	10273	
	3.00	-60 to 270/290°C	10282	10285	10288	
	5.00	-60 to 270/290°C	10277	10279	10283	
ID	df (μm)	temp. limits	10-Meter	20-Meter	40-Meter	
0.10mm	0.10	-60 to 330/350°C	41201	41202		
	0.40	-60 to 320/340°C	41203	41204		
0.18mm	0.20	-60 to 325/340°C	40201	40202	40203	
	0.40	-60 to 315/330°C	40210	40211	40212	

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

XTI®-5 (Crossbond® 5% diphenyl / 95% dimethyl polysiloxane)

- High temperature, ultra-low bleed, low polarity phase, ideal for sterols.
- Thermally stable to 360°C.
- Polarity similar to DB-5HT, DB-5XLT, PTE-5 phases.
- Equivalent to USP G27, G36 phases.

XTI®-5 (fused silica)

(Crossbond® 5% phenyl/95% dimethyl polysiloxane - extended temperature and inertness)

ID	df (μm)	temp. limits	15-Meter	30-Meter
0.25mm	0.25	-60 to 360°C	12220	12223
	0.50	-60 to 330/350°C	12235	12238
	1.00	-60 to 325/350°C	12250	12253
0.32mm	0.25	-60 to 360°C	12221	12224
	0.50	-60 to 330/350°C	12236	12239
	1.00	-60 to 325/350°C	12251	12254
0.53mm	0.50	-60 to 330/360°C	12237	12240
	1.00	-60 to 325/350°C	12252	12255
	1.50	-60 to 310/330°C	12267	12270

a **plus 1™** story

Going above and beyond her normal job responsibilities, Santina has helped collect production data for new columns, closely inspects columns during various steps of production and has developed a system of identifying problems in "wax-phase" columns. Because of her keen attention to detail, we have resolved several persistent problems with wax-phase columns.

Santina Newlen, GC Column Manufacturing Technician

**Rtx®-50 (Crossbond® 50% methyl / 50% phenyl polysiloxane)**

- General-purpose mid-polarity phase, ideal for antioxidants.
- Thermally stable to 320°C.
- Polarity similar to DB-17, DB-608, HP-17, SPB-50, SP-2250 phases.
- Equivalent to USP G3 phase.

Rtx®-50 (fused silica)

(Crossbond® 50% methyl/50% phenyl polysiloxane)

ID	df (μm)	temp. limits*	15-Meter	30-Meter	60-Meter
0.53mm	0.25	0 to 280/300°C	10522		
	0.50	0 to 270/290°C	10537	10540	10543
	0.83	0 to 270/290°C		10569	
	1.00	0 to 260/280°C	10552	10555	10558
1.50	0 to 250/270°C	10567	10570	10573	

also available

Other ID's available—for more information refer to our general catalog.

Rtx®-65TG / MXT®-65TG (Crossbond® 65% diphenyl / 35% dimethyl polysiloxane)

- Application-specific column, designed for triglycerides.
- Specially tested with triglyceride mixture.
- Thermally stable to 370°C.

Rtx®-65TG (fused silica)

(Crossbond® 65% diphenyl/35% dimethyl polysiloxane)

ID	df (μm)	temp. limits	15-Meter	30-Meter
0.25mm	0.10	40 to 370°C	17005	17008
0.32mm	0.10	40 to 370°C	17006	17009
0.53mm	0.10	40 to 360/370°C	17007	17010

did you know?

Restek's MXT® columns—rugged, flexible, inert
Silcosteel®-treated stainless steel

MXT®-65TG (Silcosteel®-treated stainless steel)

(Crossbond® 65% diphenyl/35% dimethyl polysiloxane)

ID	df (μm)	temp. limits	15-Meter	30-Meter
0.25mm	0.10	20 to 370°C	77005	77008
0.53mm	0.10	20 to 370°C	77007	77010

Rtx®-20 (Crossbond® 80% dimethyl / 20% diphenyl polysiloxane)

- General-purpose low to mid-polarity phase, ideal for flavor compounds, alcoholic beverage analysis.
- Thermally stable to 320°C.
- Polarity similar to SPB-20, VOCOL phases.
- Equivalent to USP G28, G32 phases.

Rtx®-20 (fused silica)

(Crossbond® 80% dimethyl/20% diphenyl polysiloxane)

ID	df (μm)	temp. limits*	15-Meter	30-Meter	60-Meter	105-Meter
0.25mm	0.10	-20 to 300/320°C	10305	10308	10311	10314
	0.25	-20 to 300/320°C	10320	10323	10326	10329
	0.50	-20 to 290/310°C	10335	10338	10341	10344
	1.00	-20 to 280/300°C	10350	10353	10356	10359
0.32mm	0.10	-20 to 300/320°C	10306	10309	10312	10315
	0.25	-20 to 300/320°C	10321	10324	10327	10330
	0.50	-20 to 290/310°C	10336	10339	10342	10345
	1.00	-20 to 280/300°C	10351	10354	10357	10360
0.53mm	1.50	-20 to 270/290°C	10366	10369	10372	10375
	3.00	-20 to 250/270°C	10381	10384	10387	10390
	0.10	-20 to 260/280°C	10307	10310	10313	
	0.25	-20 to 260/280°C	10322	10325	10328	
	0.50	-20 to 260/280°C	10337	10340	10343	
	1.00	-20 to 260/280°C	10352	10355	10358	
	1.50	-20 to 250/270°C	10367	10370	10373	
	3.00	-20 to 240/260°C	10382	10385	10388	
ID	df (μm)	temp. limits	10-Meter	20-Meter	40-Meter	
0.18mm	0.20	-20 to 300/320°C	40301	40302	40303	
	0.40	-20 to 300/320°C	40310	40311	40312	

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

Rtx®-225 (Crossbond® 50% cyanopropylmethyl / 50% phenylmethyl polysiloxane)

- General-purpose polar phase, ideal for FAMES, carbohydrates, sterols, flavor compounds.
- Thermally stable to 240°C.
- Polarity similar to DB-225, HP-225 phases.
- Equivalent to USP G7, G19 phases.

Rtx®-225 (fused silica)

(Crossbond® 50% cyanopropylmethyl/50% phenylmethyl polysiloxane)

ID	df (μm)	temp. limits*	15-Meter	30-Meter	60-Meter
0.25mm	0.10	40 to 220/240°C	14005	14008	
	0.25	40 to 220/240°C	14020	14023	14026
	0.50	40 to 220/240°C	14035	14038	14041
0.32mm	0.10	40 to 220/240°C	14006	14009	
	0.25	40 to 220/240°C	14021	14024	14027
	0.50	40 to 220/240°C	14036	14039	14042
0.53mm	1.00	40 to 200/220°C	14051	14054	14057
	0.10	40 to 200/220°C	14007	14010	
	0.25	40 to 200/220°C	14022	14025	
	0.50	40 to 200/220°C	14037	14040	14043
	1.00	40 to 200/220°C	14052	14055	14058

FAMEWAX™ (Crossbond® polyethylene glycol)

- Application-specific column, designed for FAMES.
- Specially tested with FAME mixture.
- Thermally stable to 250°C.
- Polarity similar to Omegawax phase.

FAMEWAX™ (fused silica)

(Crossbond® polyethylene glycol)

ID	df (μm)	temp. limits	30-Meter
0.25mm	0.25	20 to 250°C	12497
0.32mm	0.25	20 to 250°C	12498
0.53mm	0.50	20 to 250°C	12499

Rtx®-Wax (Crossbond® Carbowax® polyethylene glycol)

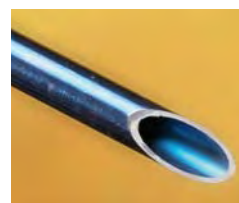
- General-purpose polar phase, ideal for FAMES, flavor compounds.
- 20°C minimum operating temperature.
- Thermally stable to 250°C.
- Polarity similar to DB-WAX, HP-Wax phases.
- Equivalent to USP G14, G15, G16, G20, G39 phases.

Rtx®-Wax (fused silica)

(Crossbond® Carbowax® polyethylene glycol)

ID	df (μm)	temp. limits*	15-Meter	30-Meter	60-Meter
0.25mm	0.10	20 to 250°C	12405	12408	
	0.25	20 to 250°C	12420	12423	12426
	0.50	20 to 250°C	12435	12438	12441
0.32mm	0.10	20 to 250°C	12406	12409	
	0.25	20 to 250°C	12421	12424	12427
	0.50	20 to 250°C	12436	12439	12442
0.53mm	1.00	20 to 240/250°C	12451	12454	12457
	0.25	20 to 250°C	12422	12425	
	0.50	20 to 250°C	12437	12440	12443
	1.00	20 to 240/250°C	12452	12455	12458
ID	df (μm)	temp. limits	10-Meter	20-Meter	
0.10mm	0.10	20 to 250°C	41601	41602	
	0.20	20 to 240/250°C	41603	41604	

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

did you **know?**

Siltek®/Sulfinert® and Silcosteel® tubing & fittings are ideal for transporting active compounds such as polar organics and sulfur compounds.

Stabilwax® / MXT®-WAX (Crossbond® Carbowax® polyethylene glycol)

- General-purpose polar phase, ideal for FAMES, flavor compounds.
- Resistant to oxidative damage.
- Thermally stable to 250°C.
- Polarity similar to DB-WAXetr, HP-Innowax, Supelcowax 10 phases.
- Equivalent to USP G14, G15, G16, G20, G39 phases.

Stabilwax® (fused silica)

(Crossbond® Carbowax® polyethylene glycol—provides oxidation resistance)

ID	df (μm)	temp. limits	15-Meter	30-Meter	30-Meter 6/pk.	60-Meter
0.25mm	0.10	40 to 250°C	10605	10608		10611
	0.25	40 to 250°C	10620	10623		10626
	0.50	40 to 250°C	10635	10638		10641
0.32mm	0.10	40 to 250°C	10606	10609		10612
	0.25	40 to 250°C	10621	10624		10627
	0.50	40 to 250°C	10636	10639		10642
0.53mm	1.00	40 to 240/250°C	10651	10654	10654-600	10657
	0.10	40 to 250°C	10607	10610		10613
	0.25	40 to 250°C	10622	10625		10628
	0.50	40 to 250°C	10637	10640		10643
	1.00	40 to 240/250°C	10652	10655	10655-600	10658
	1.50	40 to 230/240°C	10666	10669		10672
	2.00	40 to 220/230°C	10667	10670		

did you know?

We have over 2,000 pure, characterized, neat compounds in our inventory! If you do not see the EXACT mixture you need listed on any of these pages, call us.

See **page 48** for our Custom Reference Materials Request Form.

Rt-CW20M™ F&F (Carbowax® polyethylene glycol)

- Application-specific column, designed for flavor and fragrance compounds.
- True non-bonded Carbowax® 20M polarity.
- Thermally stable to 220°C.
- Polarity similar to HP-20M, Carbowax® 20M phases.

Rt-CW20M™ F&F (fused silica)

(nonbonded Carbowax® polyethylene glycol)

ID	df (μm)	temp. limits	30-Meter	50-Meter
0.25mm	0.25	60 to 220°C	12523	
0.32mm	0.33	60 to 220°C		12539

Stabilwax®-DA (Crossbond® acid-deactivated Carbowax® polyethylene glycol)

- Application-specific column, designed for underivatized free acids.
- No need for sample derivatization.
- Resistant to oxidative damage.
- Thermally stable to 250°C.
- Polarity similar to DB-FFAP, HP-FFAP, NUKOL, OV-351 phases.
- Equivalent to USP G25, G35 phases.

Stabilwax®-DA (fused silica)

(Crossbond® Carbowax® polyethylene glycol for acidic compounds)

ID	df (μm)	temp. limits	15-Meter	30-Meter	60-Meter
0.25mm	0.10	40 to 250°C	11005	11008	11011
	0.25	40 to 250°C	11020	11023	11026
	0.50	40 to 250°C	11035	11038	11041
0.32mm	0.10	40 to 250°C	11006	11009	11012
	0.25	40 to 250°C	11021	11024	11027
	0.50	40 to 250°C	11036	11039	11042
0.53mm	1.00	40 to 240/250°C	11051	11054	11057
	0.10	40 to 250°C	11007	11010	11013
	0.25	40 to 250°C	11022	11025	11028
	0.50	40 to 250°C	11037	11040	11043
	1.00	40 to 240/250°C	11052	11055	11058
	1.50	40 to 230/240°C	11062	11065	11068

Rtx®-200 (Crossbond® trifluoropropylmethyl polysiloxane)

- General-purpose mid-polarity phase, ideal for alcohols, ketones, glycols.
- Thermally stable to 340°C.
- Polarity similar to DB-200, DB-210 phases.
- Equivalent to USP G6 phase.

Rtx®-200 (fused silica)

(Crossbond® trifluoropropylmethyl polysiloxane)

ID	df (μm)	temp. limits*	15-Meter	30-Meter	60-Meter	105-Meter
0.25mm	0.10	-20 to 320/340°C	15005	15008	15011	
	0.25	-20 to 320/340°C	15020	15023	15026	15029
	0.50	-20 to 310/330°C	15035	15038	15041	15044
0.32mm	1.00	-20 to 290/310°C	15050	15053	15056	15059
	0.10	-20 to 320/340°C	15006	15009	15012	
	0.25	-20 to 320/340°C	15021	15024	15027	15030
0.53mm	0.50	-20 to 310/330°C	15036	15039	15042	15045
	1.00	-20 to 290/310°C	15051	15054	15057	15060
	1.50	-20 to 280/300°C	15066	15069	15072	15075
0.53mm	0.10	-20 to 310/330°C	15007	15010	15013	
	0.25	-20 to 310/330°C	15022	15025	15028	
	0.50	-20 to 300/320°C	15037	15040	15043	
0.53mm	1.00	-20 to 290/310°C	15052	15055	15058	
	1.50	-20 to 280/300°C	15067	15070	15073	
	3.00	-20 to 260/280°C	15082	15085	15088	15091
ID	df (μm)	temp. limits	10-Meter	20-Meter	40-Meter	
0.18mm	0.20	-20 to 310/330°C	45001	45002	45003	
	0.40	-20 to 310/330°C	45010	45011	45012	

Rtx®-1301 (G43) (Crossbond® 6% cyanopropylphenyl / 94% dimethyl polysiloxane)

- General-purpose low to mid-polarity phase, ideal for alcohols, flavor compounds.
- Thermally stable to 280°C.
- Polarity similar to DB-1301, DB-624, SPB-1301, SPB-624 phases.
- Equivalent to USP G43 phase.

Rtx®-1301 (G43) (fused silica)

(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (μm)	temp. limits*	15-Meter	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	0.10	-20 to 280°C	16005	16008	16011		16014
	0.25	-20 to 280°C	16020	16023	16026		16029
	0.50	-20 to 270°C	16035	16038	16041		16044
	1.00	-20 to 260°C	16050	16053	16056		16059
	1.40	-20 to 240°C			16016		
0.32mm	0.10	-20 to 280°C	16006	16009	16012		16015
	0.25	-20 to 280°C	16021	16024	16027		16030
	0.50	-20 to 270°C	16036	16039	16042		16045
	1.00	-20 to 260°C	16051	16054	16057		16060
	1.50	-20 to 250°C	16066	16069	16072		16075
0.53mm	0.10	-20 to 280°C	16007	16010	16013		
	0.25	-20 to 280°C	16022	16025	16028		
	0.50	-20 to 270°C	16037	16040	16043		
	1.00	-20 to 260°C	16052	16055	16058		
	1.50	-20 to 250°C	16067	16070	16073		
	3.00	-20 to 240°C	16082	16085	16088	16076	16091

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

for **more** infoRequest the Fast Facts for
Rtx®/MXT®-1301 columns
(lit. cat. #59317).

Rt-2560 (biscyanopropyl polysiloxane)

- Application-specific column, designed for separating *cis* and *trans* FAMES.
- Thermally stable to 250°C.
- Polarity similar to SP-2560 phase.

Rt-2560

(biscyanopropyl polysiloxane)

ID	df (μm)	temp. limits	100-Meter
0.25mm	0.20	20 to 250°C	13199

Rt-βDEXse™ (fused silica)(2,3-di-O-ethyl-6-O-*tert*-butyl dimethylsilyl beta cyclodextrin doped into 14% cyanopropylphenyl/86% dimethyl polysiloxane)

ID	df (μm)	temp. limits	30-Meter
0.25mm	0.25	40 to 230°C	13107
0.32mm	0.25	40 to 230°C	13106

Uses: Similar in performance to Rt-βDEXsm™ but provides better resolution for limonene, linalool, linalyl acetate, ethyl-2-methylbutyrate, 2,3-butane diol.

Rt-βDEXsp™ (fused silica)(2,3-di-O-propyl-6-O-*tert*-butyl dimethylsilyl beta cyclodextrin doped into 14% cyanopropylphenyl/86% dimethyl polysiloxane)

ID	df (μm)	temp. limits	30-Meter
0.25mm	0.25	40 to 230°C	13111
0.32mm	0.25	40 to 230°C	13110

Uses: Often useful in dual-column configurations, with the Rt-βDEXsm™ column, for complex enantiomeric separations.

Rt-βDEXsa™ (fused silica)(2,3-di-acetoxy-6-O-*tert*-butyl dimethylsilyl beta cyclodextrin doped into 14% cyanopropylphenyl/86% dimethyl polysiloxane)

ID	df (μm)	temp. limits	30-Meter
0.25mm	0.25	40 to 230°C	13109
0.32mm	0.25	40 to 230°C	13108

Uses: Unique selectivity for esters and lactones, and other fruit flavor components.

Rt-βDEXcst™ (fused silica)

(Proprietary cyclodextrin material doped into 14% cyanopropylphenyl/86% dimethyl polysiloxane)

ID	df (μm)	temp. limits	30-Meter
0.25mm	0.25	40 to 230°C	13103
0.32mm	0.25	40 to 230°C	13102

Uses: This proprietary stationary phase was developed specifically for the fragrance industry, and also has been used for pharmaceutical applications.

Rt-βDEXsm™ (fused silica)(2,3-di-O-methyl-6-O-*tert*-butyl dimethylsilyl beta cyclodextrin doped into 14% cyanopropylphenyl/86% dimethyl polysiloxane)

ID	df (μm)	temp. limits	30-Meter
0.25mm	0.25	40 to 230°C	13105
0.32mm	0.25	40 to 230°C	13104

Uses: Excellent column for most components of essential oils.

free literature

FREE applications notes to assist you with your analysis. Request your copies today!

• Grape Flavor Analysis, Using an Rt-γDEXsa™ GC Column (lit. cat. # 59553)

• GC Analysis of Chiral Flavor Compounds in Apple Juices, Using Rt-βDEXsm™ and Rt-βDEXse™ Columns (lit. cat. # 59546)

Related Literature

All of the following publications are free on request.

All are Restek application notes, unless otherwise indicated.

Lit. cat.# Title

- | | |
|--------|---|
| 59128 | Determination of Omega-3 (n-3) and Omega-6 (n-6) Fatty Acid Composition in Evening Primrose Oil, Flax Seed Oil, Black Currant Oil, and Borage Oil |
| 59136 | The Institute for Nutraceutical Advancement (INA) Validates GC Methods for Saw Palmetto, Using Rtx®-5 and Stabilwax® Columns |
| 59155B | GC Analysis of Volatile Free Fatty Acids on the Stabilwax®-DA Column |
| 59177 | Analyze Polar Compounds by Reversed Phase HPLC, Using Ultra Aqueous C18 Columns |
| 59181 | HPLC Analysis of Vitamins |
| 59186 | Analysis of Vanillin and Ethyl Vanillin in Vanilla Flavors, Using Ultra C8 Column |
| 59199 | Analyzing the Heat Level of Spicy Foods, Using an Ultra C18 HPLC Column |
| 59348 | Monitoring Volatile Compounds in Food Contact Packaging, Using Purge and Trap GC/MS and an Rtx®-5MS Capillary Column |
| 59364 | Analyzing Nutraceutical Products by Liquid and Gas Chromatography |
| 59398 | Analysis of Preservatives, Using HPLC |
| 59462 | Analyzing Alcoholic Beverages by Gas Chromatography (technical guide) |
| 59530 | Single-Column Method for HPLC Analysis of Organic Acids in Fruit Juices, Using an Allure™ Organic Acids Column |
| 59546 | GC Analysis of Chiral Flavor Compounds in Apple Juices, Using the Rt-βDEXsm™ and Rt-βDEXse™ Columns |
| 59553 | Grape Flavor Analysis, Using an Rt-βDEXsa™ GC Column |
| 59580A | Fast, Selective Triglyceride Analysis |
| 59581 | Analysis of Cholesterol and Other Dietary Sterols |
| 59584A | High-Resolution Analyses of Fatty Acid Methyl Esters (FAMES) by Gas Chromatography |
| 59889 | A Guide to the Analysis of Chiral Compounds by GC (technical guide) |
| 59890 | Selection Guide for Polar WAX GC Column Phases (technical guide) |
| 59901 | High Performance Silica Products |
| 59012 | Genuine Restek Replacement Parts for HPLC Systems (flyer) |
| 59627E | Genuine Restek Replacement Parts for Agilent GCs (brochure) |
| 59241B | HPLC Columns and Accessories (HPLC catalog) |
| 59065 | Restek Chromatography Supplies Catalog (current edition) |

free literature

Many example chromatograms in our 24-page chiral analysis guide will help you find the best chiral column, or columns, for your application.

Request **A Guide to the Analysis of Chiral Compounds by GC** (lit. cat. # 59889) for more information about chiral separations.

Call Restek at **800-356-1688** or **814-353-1300, ext. 5**, or contact your Restek representative, to request your free copy.

View these electronic publications on our website: www.restek.com

HPLC Analysis of Preservatives

Using Ultra Aqueous and Pinnacle II™ Columns

Restek Advantage 2002 vol. 2

High-Resolution Analysis of Fatty Acid Methyl Esters (FAMES)

Using an Rt-2560 Capillary GC Column to Resolve *cis* and *trans* Isomers

Restek Advantage 2003 vol. 1

Analyzing Fatty Acid Methyl Esters (FAMES) by GC

Using Restek Capillary Columns and Analytical Reference Materials

Restek Advantage 2002 vol. 4

tech tip

To optimize chiral separations, use:

- 1) Faster linear velocities (80cm/sec.) with hydrogen carrier gas.
- 2) Slower temperature ramp rates (1–2°C/min.).
- 3) Appropriate minimum operating temperature (40 or 60°C).
- 4) On-column concentrations of 50ng or less.

Packed GC Columns

please note

All stock CarboBlack™ columns are PRE-CONDITIONED

CarboBlack™ Solid Supports

Graphitized carbon black offers unique selectivity and very little adsorption for alcohol analyses. Two types of CarboBlack™ supports are available, CarboBlack™ B and CarboBlack™ C. CarboBlack™ B support, with its higher surface area, can hold up to a 10% loading of a non-silicone liquid phase. CarboBlack™ C support can hold up to a 1% loading of a non-silicone liquid phase. Many Carbowax® 20M-loaded CarboBlack™ packings are available. CarboBlack™ packings are treated with KOH or picric acid for basic or acidic compounds, and special alcoholic beverage loadings are available. CarboBlack™ supports provide resolution and retention similar to Carbowax™ and Carbograph™ supports.

Column Configurations



General Configuration
Suffix -800



Agilent 5880, 5890, 5987, 6890:
Suffix -810



Varian 3700, Vista Series, FID:
Suffix -820



PE 900-3920 Sigma 1,2,3:
Suffix -830



PE Auto System 8300, 8400, 8700 (Not On-Column):
Suffix -840

See page 103 for custom configurations

Note: Initial 2" of column will be empty, to accommodate a needle. For a completely filled column add suffix -901.

On CarboBlack™ B	Mesh	Stainless Steel Tubing				SilcoSmooth™ Tubing**			
		L (ft.)	OD (in.)	ID (mm)	cat.#*	L (m)	OD (in.)	ID (mm)	cat.#*
5% Carbowax® 20M	80/120	—	—	—	—	2	1/8	2	80105-
5% Carbowax® 20M	60/80	6	1/8	2.1	88012-	1.8	1/8	2	80106-
6.6% Carbowax® 20M	80/120	6	1/8	2.1	80451-	2	1/8	2	80107-
4% Carbowax® 20M/ 0.8% KOH	60/80	—	—	—	—	2	1/8	2	80116-
1% Rt-1000	60/80	8	1/8	2.1	88013-	2.4	1/8	2	80206-
1% Rt-1000	60/80	6	1/8	2.1	80452-	2	1/8	2	80207-
3% Rt-1500	80/120	10	1/8	2.1	80453-	3.05	1/8	2	80211-
1% Rt-1510	60/80	10	1/8	2.1	80454-	3.05	1/8	2	80216-
1.5% XE-60/1% H ₃ PO ₄	60/80	6	1/8	2.1	80455-	1.8	1/8	2	80305-

Nickel 200 Tubing

On CarboBlack™ B	Mesh	L (m)	OD (in.)	ID (mm)	cat.#*
5% Krytox (Ni 200 tubing)	60/80	3.05	1/8	2.1	80127-

On CarboBlack™ C	Mesh	Stainless Steel Tubing				SilcoSmooth™ Tubing**			
		L (ft.)	OD (in.)	ID (mm)	cat.#*	L (m)	OD (in.)	ID (mm)	cat.#*
0.2% Carbowax® 1500	60/80	6	1/8	2.1	80456-	2	1/8	2	80121-
0.2% Carbowax® 1500	80/100	6	1/8	2.1	80457-	2	1/8	2	80122-
0.1% Rt-1000	80/100	6	1/8	2.1	80458-	1.8	1/8	2	80205-
0.19% picric acid	80/100	6	1/8	2.1	80459-	2	1/8	2	80311-
0.3% Carbowax® 20M/0.1% H ₃ PO ₄	60/80	2.5	3/16	3.2	80460-	0.75	3/16	3.2	80111-

*Please add configuration suffix number to cat.# when ordering.

**Silcosteel®-deactivated stainless steel.

Chromosorb® Diatomaceous Earth Supports

Restek offers the full line of Chromosorb® solid supports that are specially sieved to remove fines and ensure tight particle distribution. Choosing the appropriate support will depend on your application. Need assistance? Call Technical Service at 800-356-1688 or 814-353-1300, ext. 4, or contact your local Restek representative for more information.

Chromosorb® P (used to prepare Silcoport™ P)

Chromosorb® P support is manufactured from hard firebrick, making it a rugged material. This support is available acid washed (AW), non-acid washed (NAW), and traditional dimethyldichlorosilane (DMDCS) treated. Chromosorb® P support can hold up to 30 weight% of liquid stationary phase, making it the highest loading support available.

Chromosorb® W (used to prepare Silcoport™ W and Silcoport™ BW)

Chromosorb® W support is a flux-calcinated diatomite. This solid support is very fragile but offers the highest inertness of all diatomaceous earth supports. It can be prepared with up to 25 weight% of liquid stationary phase. Chromosorb® W support is available in AW, NAW, and DMDCS, or treated with Restek's proprietary (Silcoport™) deactivation. Chromosorb® W-HP is an acid washed, silanized version of Chromosorb® W.

Chromosorb® G

Chromosorb® G support is the hardest support available and has the lowest surface area of all the diatomaceous earth supports. Chromosorb® G support is available as AW, NAW, and DMDCS-treated. It can hold up to 10 weight% of liquid stationary phase.

Chromosorb® T

Chromosorb® T support is made from Teflon® and is an extremely inert solid support.

Call Restek at 800-356-1688 or 814-353-1300, ext. 3, or contact your local Restek representative for quotes on any Chromosorb® material. Some of the popular Chromosorb®-based stock columns and packings available are:

Chromosorb®-Based Packed Columns

		Stainless Steel Tubing				SilcoSmooth™ Tubing**			
		L (ft.)	OD (in.)	ID (mm)	cat.#*	L (m)	OD (in.)	ID (mm)	cat.#*
On 100/120 Silcoport™ W***									
3% Rt-101		6	1/8	2.1	80461-	2	1/8	2	80400-
3% Rt-2100		6	1/8	2.1	80462-	2	1/8	2	80420-
5% Rt-1200/1.75% Bentone 34		6	1/8	2.1	80463-	2	1/8	2	80125-
5% Rt-1200/5% Bentone 34		6	1/8	2.1	80464-	2	1/8	2	80129-
		Stainless Steel Tubing				SilcoSmooth™ Tubing**			
		L (ft.)	OD (in.)	ID (mm)	cat.#*	L (m)	OD (in.)	ID (mm)	cat.#*
On Chromosorb® PAW									
10% TCEP	100/120	8	1/8	2.1	80465-	2.5	1/8	2	80126-
23% Rt-1700	80/100	30	1/8	2.1	80466-	9.2	1/8	2	80128-

*Please add configuration suffix number to cat.# when ordering.

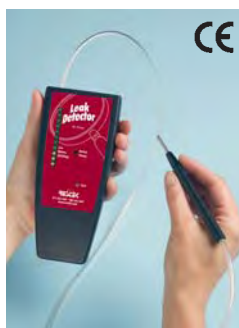
**Silcosteel®-deactivated stainless steel.

***Modified version of Chromosorb® W; highest inertness, most consistent performance.

Restek's packed columns deliver the

1-2-3 PUNCH!

1. Bonded stationary phases mean short conditioning times, low bleed, and unsurpassed column lifetimes.
2. SilcoSmooth™ tubing provides the inertness of glass and the durability of stainless steel.
3. Silcoport™ diatomaceous earth provides unsurpassed inertness for trace analysis.

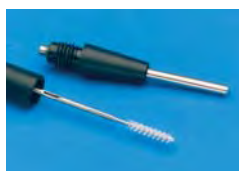


Leak Detector

- Affordable thermal conductivity leak detector - every analyst should have one.
- Compact, portable, ergonomic design is easy to hold and operate.
- Sensitive - detects helium, hydrogen*, or nitrogen at 1×10^{-4} cc/sec
- Fast results - responds to leaks in less than 2 seconds.
- Autozeroing with the touch of a button.
- Battery-operated, for portability.
- Built-in rechargeable battery—charging adaptor included.

In continuing our efforts to provide chromatographers with the best available columns, tools, and accessories, we have enhanced our popular Restek Electronic Leak Detector. New features include internal battery charge capability, a low battery indicator, a battery charge indicator light, yellow lights to signal a nitrogen leak, a repositioned on/off switch, to eliminate accidentally powering on the unit, and a new probe tip design that prevents debris from entering the unit. The new leak detector maintains the microchip technology that enables high sensitivity in a compact unit, the autozero feature that allows instantaneous zeroing with the touch of a button, and the ergonomic design that puts all controls at your fingertips, for maximum ease of use.

The new Restek Electronic Leak Detector is the affordable solution for GC leak detection. Leaks can cause detector noise and baseline instability, waste carrier gas, and shorten column lifetimes. The leak detector responds in less than 2 seconds to leaks of gases with thermal conductivities different from air, indicating leaks with both an audible alarm and an LED readout. The leak detector detects minute gas leaks that can go undetected by liquid leak detectors. And, remember - you should never use liquid leak detectors on a capillary system, because liquids can be drawn into the column through the leaks.



Easy-to-clean probe assembly.

Description	qty.	cat.#
Leak Detector with 110Volt Battery Charger	ea.	22451
Leak Detector with 220Volt European Battery Charger	ea.	22451-EUR
Leak Detector with 220Volt UK Battery Charger	ea.	22451-UK

*Caution: The Restek Electronic Leak Detector is NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possible hazardous conditions.



Each kit is sealed in a factory-clean Mylar® bag.

FastPack™ Inlet Kits for Agilent GCs

- Convenient: the parts you use are all in one package—no hunting for individual parts.
- Economical: costs less than the sum of the individual parts.
- Clean: Mylar® bag is factory sealed; no contamination of the products from weeks in the lab.

FastPack™ Inlet Kits are a great way to make routine maintenance easy. Each kit includes one:

- Inlet liner—choose from four popular styles.
- Viton® O-ring.
- 0.8mm ID gold-plated inlet seal.
- Inlet seal washer.
- 11mm Thermolite® septum.



FastPack™ Inlet Kits make routine injection port maintenance easy!

		1 pack includes 5 maintenance kits		
Deactivated Liner	cat.#	pack of 5 kits	5 or more packs	20 or more packs
4mm Splitless*	21101			
4mm Splitless Gooseneck*	21102			
4mm Splitless Double Gooseneck*	21103			
4mm Split with Fused Silica Wool**	21104			

*Liner dimensions: 4mm ID, 6.5mm OD, 78.5mm long.

**Liner dimensions: 4mm ID, 6.3mm OD, 78.5mm Long.

Thermolite® Septa

- Usable to 340°C inlet temperature.
- Each batch tested with FIDs, ECDs, and MSDs to ensure lowest bleed.
- Excellent puncturability.
- Preconditioned and ready to use.
- Do not adhere to hot metal surfaces.
- Packaged in non-contaminating glass jars.



Septum Diameter	25-pk.	50-pk.	100-pk.
5mm (1/16")	20351	20352	20353
6mm (1/4")	20355	20356	20357
7mm	20381	20382	20383
8mm	20370	20371	—
9mm	20354	20358	20362
9.5mm (3/16")	20359	20360	20361
10mm	20378	20379	20380
11mm (7/16")	20363	20364	20365
11.5mm	22385	22386	22387
12.5mm (1/2")	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Plug	20372	20373	20374

InfraRed™ Septa

- Usable to 325°C inlet temperature.
- Preconditioned and ready to use.
- Excellent puncturability.
- Do not adhere to hot metal surfaces.
- Low bleed.
- Packaged in non-contaminating glass jars.



Septum Diameter	25-pk.	50-pk.	100-pk.
9mm	21417	21418	21419
9.5mm (3/16")	21421	21422	21423
10mm	21424	21425	21426
11mm (7/16")	21427	21428	21429
11.5mm	21430	21431	21432
12.5mm (1/2")	21433	21434	21435
17mm	21436	21437	21438
Shimadzu Plug	21439	21440	21441

IceBlue™ Septa

- Usable to 250°C inlet temperature.
- General-purpose septa.
- Excellent puncturability.
- Preconditioned and ready to use.
- Do not adhere to hot metal surfaces.
- Packaged in non-contaminating glass jars.
- Ideal for SPME.



Septum Diameter	50-pk.	100-pk.
9mm	22381	22382
9.5mm (3/16")	22388	22389
10mm	22390	22391
11mm (7/16")	22392	22393
11.5mm	22383	22384
12.5mm (1/2")	22394	22395
17mm	22396	22397
Shimadzu Plug	22398	22399

septum sizes**Reference Chart**

Instrument	Septum Size (mm)
Agilent (HP)	
5880A, 5890, 6890, 6850, PTV	11
5700, 5880	9.5/10
On-Column Injection	5
CE Instruments (TMQ)	
TRACE™ GC	17
Finnigan (TMQ)	
GC 9001	9.5
GCQ	9.5
GCQ w/TRACE™, PTV	17
QCQ™	9.5
TRACE™ 2000	9.5
Fisons/Carlo Erba (TMQ)	
8000 series	17
Gow-Mac	
6890 series	11
All other models	9.5
PerkinElmer	
Sigma series	11
900,990	11
8000 series	11
Auto SYS	11
Auto SYS XL	11
Pye/Unicam	
All models	7
Shimadzu	
All models	Plug
SRI	
All models	Plug
Tracor	
540	11.5
550,560	9.5
220,222	12.5
Varian	
Injector type:	
Packed column	9.5/10
Split/splitless	
1078/1079	10/11
1177	9
1075/1077	11

Measure

your old
septum here
(size in mm)

5

7

9

9.5

10

11

11.5

12.5

17

GC Accessories

Hole in Drilled Uniliner® makes direct injection possible with EPC-equipped 6890 GCs!

DI Liners for Agilent 5890 & 6890 GCs

For 0.25/0.32/0.53mm ID Columns

Benefits/Uses	ID*/OD & Length (mm)	Similar to Agilent part #	ea.	cat.# 5-pk.	25-pk.
trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	—	21054	21055	20998
trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	—	21054-214.1	21055-214.5	20998-214.25
trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	G1544-80730	20756	20771	—
trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	—	20508	20509	—
trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	G1544-80700	20954	20989	—
trace, active samples, high recovery & linearity	1.0 ID 6.3 OD x 78.5	—	21390-214.1	21391-214.5	—

DI Liners for Varian 1177 GCs

For 0.25/0.32/0.53mm ID Columns

Benefits/Uses	ID*/OD & Length (mm)	Similar to Varian part #	cat.# ea.	cat.# 5-pk.
trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	—	21470	21471
trace, active samples, high recovery & linearity	4.0 ID 6.3 OD x 78.5	—	21468	21469

DI Liners for Shimadzu GCs

For 0.32/0.53mm ID Columns

Benefits/Uses	ID*/OD & Length (mm)	Similar to Shimadzu part #	ea.	cat.# 5-pk.	25-pk.
trace, active samples, high recovery & linearity	3.5 ID 5.0 OD x 95	—	21285	21286	—
trace, active samples, high recovery & linearity	3.5 ID 5.0 OD x 95	—	21287	21288	—
trace, active samples, high recovery & linearity	3.5 ID 5.0 OD x 95	—	21289	21290	—
trace, active samples, high recovery & linearity	3.5 ID 5.0 OD x 95	—	21291	21292	—

DI Liners for PerkinElmer GCs

For 0.32/0.53mm ID Columns

Benefits/Uses	ID*/OD & Length (mm)	Similar to PE part #	cat.# ea.	cat.# 5-pk.
trace, active samples, high recovery & linearity	4.0 ID 6.2 OD x 92.1	—	20819	20822
trace, active samples, high recovery & linearity	4.0 ID 6.2 OD x 92.1	—	21293	21294
trace, active samples, high recovery & linearity	4.0 ID 5.0 OD x 92.1	—	21295	21296
trace, active samples, high recovery & linearity	4.0 ID 6.2 OD x 92.1	—	21297	21298

Direct Injection Liners for Thermo Finnigan 8000 & TRACE™ Series GCs

0.32 & 0.53mm ID columns

Benefits/Uses	ID*/OD & Length (mm)	Similar to TF part #	ea.	cat.#	25-pk.
trace, active samples, high recovery, & linearity	5.0 ID 8.0 OD x 105	—	22411	22412	—
trace, active samples, high recovery, & linearity	5.0 ID 8.0 OD x 105	—	22413	22414	—

All liners are **100% deactivated**. All liners are shipped intermediate polarity (IP) deactivated unless otherwise requested.

Viton® O-Rings for Agilent GCs

- Fit split (6.3mm OD) or splitless (6.5mm OD) liners.

Description	Max. temp.	Similar to Agilent part #	qty.	cat.#
Viton® O-Rings for Agilent GCs	250°C	5180-4182	25-pk.	20377

**Graphite O-Rings for Agilent and Varian 1177 GCs**

- Excellent thermal stability at injection port temperatures up to 450°C!

Description	Max. temp.	Similar to Agilent part #	Restek cat.#	
			10-pk.	50-pk.
Graphite O-rings for split liners (6.35mm ID)	450°C	5180-4168	20296	20297
Graphite O-rings for splitless liners (6.5mm ID)	450°C	5180-4173	20298	20299

**Liner Seals for Varian 1078/1079**

Description	Max. temp.	Similar to Varian part #	qty.	cat.#
		392611919		
5mm Graphite Liner Seals for Varian 1078/1079 GCs	450°C	392534201	10-pk.	22683

**Viton® O-Rings for PerkinElmer AutoSys GCs**

Description	Max. temp.	Similar to PE part #	qty.	cat.#
Viton® O-Rings for PerkinElmer AutoSys GCs	250°C	N6101374	10-pk.	20262

**Graphite O-Rings for PerkinElmer AutoSys XL PSS**

Description	Max. temp.	Similar to PE part #	qty.	cat.#
Graphite O-Rings for PerkinElmer AutoSys XL PSS	450°C	N610-1751	10-pk.	21475
Graphite O-Rings for PerkinElmer AutoSys XL PSS	450°C	N610-1751	25-pk.	21476

**Viton® O-Rings for PerkinElmer PSS**

Description	Max. temp.	Similar to PE part #	qty.	cat.#
Viton® O-Rings for PerkinElmer PSS	250°C	N6101747	10-pk.	20366

**Graphite O-Rings for Shimadzu 17A and 2010 GCs**

Description	Max. temp.	Similar to Shimadzu part #	qty.	cat.#
Graphite O-Rings for Split Liners	450°C	221-48393-91	5-pk.	20243
Graphite O-Rings for Splitless Liners	450°C	221-47222-91	5-pk.	20244

**Viton® O-Rings for Shimadzu 17A and 2010 GCs**

Description	Max. temp.	Similar to Shimadzu part #	qty.	cat.#
Viton® O-Rings for Shimadzu 17A and 2010 GCs	250°C	036-11203-84	10-pk.	21477

**Septum Puller**

Remove septum, O-rings, stuck ferrule fragments; you'll find many more uses.

Description	qty.	cat.#
Septum Puller	ea.	20117

**Inlet Liner Removal Tool**

- Easily remove liner from injector—no more burned fingers.
- Made from high-temperature silicone.
- Won't chip or crack the liner.

Description	qty.	cat.#
Inlet Liner Removal Tool	3-pk.	20181





Dual Vespel® Ring Inlet Seals

Washerless, Leak-Tight Seals for Agilent GCs

- Vespel® ring embedded in bottom surface eliminates need for washer.
- Vespel® ring embedded in top surface reduces operator variability by requiring minimal torque to seal.
- Prevents oxygen from permeating the carrier gas, increasing column lifetime.

0.8mm ID Dual Vespel® Ring Inlet Seal	2-pk.	10-pk.
Siltek®	21242	21243
Gold-Plated	21240	21241
Stainless Steel	21238	21239
1.2mm ID Dual Vespel® Ring Inlet Seal	2-pk.	10-pk.
Siltek®	21248	21249
Gold-Plated	21246	21247
Stainless Steel	21244	21245



Replacement Inlet Seals with Washers

- Special grade of stainless steel that is softer and deforms more easily, creating a better seal.
- Increases column lifetime because oxygen cannot permeate into the carrier gas.
- Reduced noise benefits high-sensitivity detectors (e.g., ECDs, MSDs).
- Siltek® treatment provides the inertness similar to fused silica.
- Highly-polished stainless steel increases inertness to active compounds.
- All seals include washers.

Single-Column Installation, 0.8mm Opening*		0.25/0.32mm ID Dual-Column Installation, 1.2mm Opening		0.53mm ID Dual-Column Installation (1/16-inch opening)	
2-pk.	10-pk.	2-pk.	10-pk.	2-pk.	10-pk.
Stainless Steel Inlet Seal					
21315	21316	20390	20391	20392	20393
Gold-Plated Inlet Seal					
21317	21318	21305	21306	—	—
Siltek® Inlet Seal					
21319	21320	21307	21308	—	—

*0.8mm ID stainless steel inlet seal is similar to Agilent part #18740-20880,
0.8mm ID gold-plated inlet seal is similar to Agilent part #18740-20885.

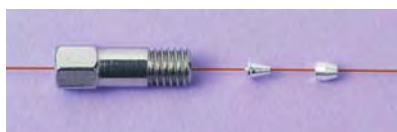


Alumaseal® Ferrules*

- Aluminum construction, will not crack or fragment.
- Eliminate out-gassing, make leak-tight seals, for less detector noise.
- No retightening required after temperature cycles—excellent for GC/MS.
- Unique two-piece design permanently locks on fused silica tubing without causing breakage.
- Will not stick in fittings, unlike Vespel® or graphite.
- Use with any 1/16" compression-type fitting.

Ferrule ID	Fits Column ID	qty.	cat.#
0.4mm	0.25mm	10-pk.	21472
0.5mm	0.32mm	10-pk.	21473
0.8mm	0.53mm	10-pk.	21474

*Patent pending.



Vespel® Ferrules

- 100% high-temperature polyimide.
- Stable to 350°C.
- Durable, leak-tight.

Graphite Ferrules

- High-purity, high-density graphite.
- Smoother surface and cleaner edges than conventional graphite ferrules.
- Contain no binders that can off-gas or adsorb analytes.
- Stable to 450°C.

Vespel®/Graphite Ferrules

- 60%/40% Vespel®/graphite blend, offering the best combination of sealing and ease of workability.
- Seal with minimal torque, reusable, and preferred for vacuum and high-pressure uses.
- Stable to 400°C.
- Recommended for mass spec transfer lines.

Capillary Ferrules—For 1/16-Inch Compression-Type Fittings

Ferrule ID	Fits Column ID	qty.	Vespel®	Graphite	Vespel®/Graphite
0.3mm	≤ 0.20mm	10-pk.	22213	20233	20275
0.4mm	0.25/0.28mm	10-pk.	22214	20200	20211
0.4mm	0.25/0.28mm	50-pk.	—	20227	20229
0.5mm	0.28/0.32mm	10-pk.	22215	20201	20212
0.5mm	0.28/0.32mm	50-pk.	—	20228	20231
0.6mm	0.28mm**	10-pk.	—	—	20232
0.8mm	0.45/0.53mm	10-pk.	22216	20202	20213
0.8mm	0.45/0.53mm	50-pk.	—	20224	20230
1.0mm	0.75mm*	10-pk.	22217	21058	—
1.2mm	0.75mm	10-pk.	22218	—	—
1.6mm	1.00mm*	10-pk.	—	21060	—



save **money!**

Buy ferrules in bulk 50-packs!

Compact Ferrules—For Agilent 5890/6890/6850 GCs

Ferrule ID	Fits Column ID	qty.	Graphite	Vespel®/Graphite
0.4mm	0.25/0.28mm	10-pk.	20250	20238
0.4mm	0.25/0.28mm	50-pk.	20251	20239
0.5mm	0.28/0.32mm	10-pk.	21007	20248
0.5mm	0.28/0.32mm	50-pk.	21008	20249
0.8mm	0.45/0.53mm	10-pk.	20252	20263
0.8mm	0.45/0.53mm	50-pk.	20253	20264
1.0mm	0.75mm*	10-pk.	21059	21056
1.6mm	1.00mm*	10-pk.	21061	21057

*For micropacked columns.

**For 0.28mm MXT® columns.

Encapsulated Ferrules—For 1/16-Inch Compression Fittings

- Reusable—will not deform and stick in fittings.
- Less torque needed to seal ferrule.
- Restek's unique blend of graphite minimizes fragmentation and outgassing.

Ferrule ID	Fits Column ID	qty.	cat.#
0.4mm	0.25mm	10-pk.	21036
0.5mm	0.32mm	10-pk.	21037
0.8mm	0.53mm	10-pk.	21038



Inlet and FID Maintenance Kits for Agilent GCs

- Include the most common consumable supplies.
- All parts meet or exceed performance by instrument manufacturer's parts.
- Parts list makes reordering easy.

FID Maintenance Kit



Inlet Maintenance Kit



FID kits include:

- 1/4-inch, 0.4, 0.5, and 0.8mm ID graphite ferrules.
- FID/NPD capillary adaptor.
- Capillary nuts.
- Jet reamers/ferrule removers.
- 1/4-inch nut.
- Scoring wafer.
- Capillary column caps.
- Ignitor for either Agilent 5890 or 6890/6850 GCs.
- FID flow measuring adaptor.
- 1/4- x 5/16-inch wrench.
- Installation gauge.
- Wire cleaning brush.
- High-performance Siltek®-treated FID jet for either Agilent 5890 (adaptable jet) or 6890/6850 (dedicated jet) GCs.
- 1/4-inch nut driver for jet removal.

Inlet kit includes:

- 0.4, 0.5, and 0.8mm ID graphite ferrules.
- Viton® O-rings.
- Capillary nuts.
- Inlet seals.
- Reducing nut.
- Scoring wafer.
- 11mm Thermolite® septa.
- 4.0mm single gooseneck liner.
- 4.0mm split liner with wool.
- Capillary column caps.
- 1/4- x 5/16-inch wrench.
- Septum puller.
- Installation gauge.
- Wire cleaning brush.
- Jet reamers/ferrule removers.
- Inlet liner removal tool.

Description	qty.	cat.#
Inlet Maintenance Kit for Agilent 5890/6890/6850 GCs	kit	21069
FID Maintenance Kit for Agilent 5890 GCs	kit	21070
FID Maintenance Kit for Agilent 6890/6850 GCs	kit	21071

Crimp-Top Vials, 2.0mL, 12 x 32mm, 11mm Crimp Finish

White graduated marking spots are a standard feature unless otherwise noted.

Crimp-Top Vial, Snap Seal™ Style (12 x 32mm, 11mm Crimp)

Description	100-pk.	1000-pk.
2.0mL Clear Glass Vial w/White Graduated Marking Spot*	24383	24384
2.0mL Amber Glass Vial w/White Graduated Marking Spot*	24385	24386
2.0mL Clear Glass Vial without Graduated Marking Spot	21152	21153

2.0mL,
11mm,
Crimp-Top
Vial11mm Aluminum Crimp
Seals with Septa**11mm Aluminum Crimp Seals w/Septa**

Description	100-pk.	500-pk.	1000-pk.
Silver Seal, PTFE/Natural Rubber Septa	21174	—	21175
Mixed Colors, PTFE/Natural Rubber Septa***	—	21724	—
Silver Seal, PTFE/Silicone Septa**	24359	—	24360
Mixed Colors, PTFE/Silicone Septa***	—	21725	—

Convenience Kits: Vials, Caps, & Septa

Description	100-pk.	1000-pk.
2.0mL Clear Vial, deactivated, PTFE/Natural Rubber Seal†	24671	24672
2.0mL Amber Vial, deactivated, PTFE/Natural Rubber Seal†	24673	24674
2.0mL Clear Vial, untreated, PTFE/Natural Rubber Seal	21196	21197
2.0mL Amber Vial, untreated, PTFE/Natural Rubber Seal	21198	21199
2.0mL Clear Vial, untreated, PTFE/Silicone Seal	24646	24647
2.0mL Amber Vial, untreated, PTFE/Silicone Seal	24648	24649

Glass, Limited
Volume Insert with
Bottom Spring**Limited Volume Inserts for 2mL Crimp-Top & Short-Cap, Screw-Thread Vials**

Description	100-pk.	1000-pk.
50µL Glass, Polypropylene, Bottom Spring	24513	21782
250µL Glass, Big Mouth Insert w/ Bottom Spring	21776	21777
250µL Glass, Big Mouth Insert w/ Glass Flange (Step™ Design)‡	24516	21779
350µL Glass, Flat Bottom Insert	21780	24517
350µL Glass, Flat Bottom Insert w/ ID Ring	24692	24693
250µL Polypropylene, Bottom Spring	24518	—
250µL Polypropylene, Top Flange	24519	—
250µL Polypropylene, No Spring	24520	—

*Colored marking spots available on request in blue, green, rust, or yellow (1000 packs only).

**PTFE/Silicone/PTFE available on request (1000 packs only).

***Individual colored seals available on request.

†Silcote™ CL7 deactivation.

‡Not to be used with 9mm screw-thread vials.

Standard Micro-Liter Syringes for Agilent 7673, 7683, and 6850 Autosamplers

- Hamilton and SGE syringes are designed and tested to meet critical autosampler performance.
- SGE manufactures autosampler syringes for every major GC instrument company.
- Needle point styles are designed to withstand multiple, fast injections through a septum.

Hamilton Syringes

Volume	Needle Term.	Needle Gauge	Needle Length	Point Style	Hamilton Model	cat.#	qty.	Restek cat.#
5µL	ASN	23s	1.71"	Agilent	75	87990	6-pk.	20170
5µL	ASN	26s	1.71"	Agilent	75	87989	6-pk.	21230
5µL	ASN	23s–26s	1.71"	Agilent	75	87994	6-pk.	24594
10µL	ASN	23s	1.71"	Agilent	701	80390*	6-pk.	20169
10µL	ASN	26s	1.71"	Agilent	701	80389	6-pk.	24599
10µL	ASN	23s–26s	1.71"	Agilent	701	80391	6-pk.	24600

**SGE Syringes**

Volume	Needle Term.	Needle Gauge	Needle Length	Point Style	SGE Model	cat.#	qty.	Restek cat.#
5µL	F	23	42mm	Cone	SK-5F-HP-0.63	001814	6-pk.	24783
5µL	F	26	42mm	Cone	SK-5F-HP-0.47	001804	6-pk.	24782
5µL	F	23–26s	42mm	Cone	SK-5F-HP-0.63/0.47	001822	6-pk.	21214
10µL	F	23	42mm	Cone	SK-10F-HP-0.63	002814	6-pk.	24787
10µL	F	26	42mm	Cone	SK-10F-HP-0.47	002804	6-pk.	24786
10µL	F	23–26s	42mm	Cone	SK-10F-HP-0.63/0.47	002822	6-pk.	21215



*Designated by Agilent as #80397.

23s—Single Gauge Needle

- The most popular gauge for Agilent 7673.
- Stocked for same-day shipment.
- Best for Merlin Microseal® septum and standard septum-equipped GCs.
- Packed column injection ports.
- Split/splitless injection ports.

26s—Single Gauge Needle

- On-column injection ports.
- Split/splitless injection ports.

23s–26s—Dual Gauge (tapered)

- Durability of a 23s gauge needle.
- Ability of a 26s gauge needle to perform split/splitless and on-column injections.

Needle Termination Codes

Hamilton: (ASN) Autosampler Cemented Needle

SGE: (F) Fixed Needle

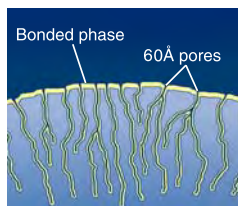
Allure™ Organic Acids**Physical Characteristics:**

particle size: 5µm, spherical
pore size: 60Å

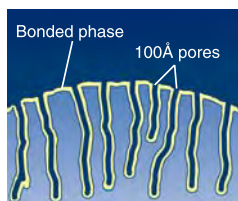
non-encapped
pH range: 2.5 to 7.5
temperature limit: 80°C

Chromatographic Properties:

Allure™ Organic Acids columns provide enhanced retention and selectivity for polar organic acids, allowing separations to be performed on a single 30cm column. An Allure™ Organic Acids column effectively resolves key organic acids such as tartaric and quinic acids, using the chromatographic conditions specified in AOAC method 986.13. Retention is stable and reproducible, even with the 100% aqueous mobile phase specified in the AOAC method.



Allure™ 60Å pore size provides maximum retention.



Ultra 100Å pore size provides moderate retention.

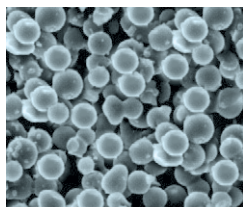
Length	3.2mm ID cat.#	4.6mm ID cat.#
5µm Column		
150mm	9165563	9165565
250mm		9165575
300mm		9165585

To order a 2.1mm, 3.2mm, or 4.6mm ID column with a Trident™ Integral Inlet Fitting, add "-700" to the catalog number for the column.

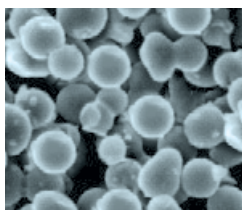
Example: 100mm x 4.6mm ID Ultra C18 column with Trident™ Integral Inlet Fitting: 9174315-700

Nominal additional charge

For guard cartridges for these columns, see page 44.

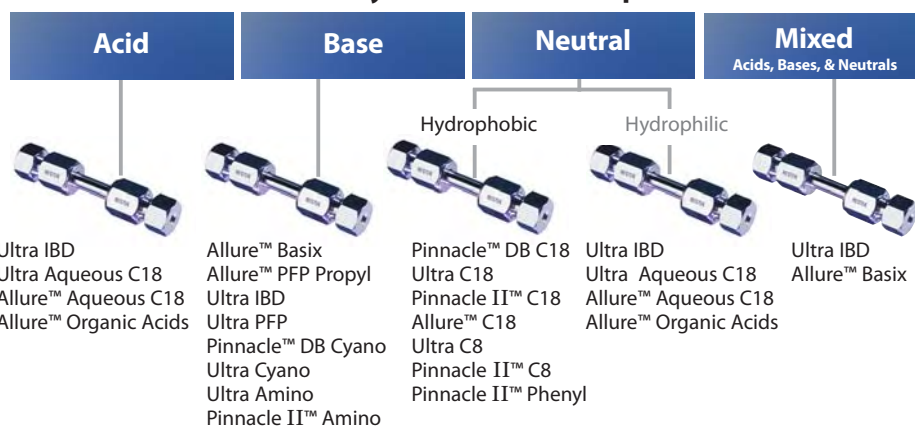


3µm particles provide fast separations.



5µm particles are ideal for general screening and initial method development.

Choose the best stationary phase for your application based on analyte functionality.

Analyte Functional Group

Ultra C8 (USP L7)**Excellent All-Purpose Reversed Phase Columns****Physical Characteristics:**

particle size: 3µm or 5µm, spherical fully end-capped
 pore size: 100Å pH range: 2.5 to 7.5
 carbon load: 12% temperature limit: 80°C

Chromatographic Properties:

A very retentive, high-purity, base-deactivated reversed phase packing that exhibits excellent peak shape for a wide range of compounds.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.0mm ID cat.#	4.6mm ID cat.#
3µm Columns					
30mm	9103331	9103332	9103333	—	9103335
50mm	9103351	9103352	9103353	—	9103355
100mm	9103311	9103312	9103313	—	9103315
5µm Columns					
30mm	9103531	9103532	9103533	—	9103535
50mm	9103551	9103552	9103553	—	9103555
100mm	9103511	9103512	9103513	9103514	9103515
150mm	9103561	9103562	9103563	9103564	9103565
200mm	9103521	9103522	9103523	—	9103525
250mm	9103571	9103572	9103573	—	9103575

Ultra C18 (USP L1)**Excellent All-Purpose Reversed Phase Columns****Physical Characteristics:**

particle size: 3µm or 5µm, spherical fully end-capped
 pore size: 100Å pH range: 2.5 to 7.5
 carbon load: 20% temperature limit: 80°C

Chromatographic Properties:

A very retentive, high-purity packing that exhibits excellent peak shape for a wide range of compounds. Excellent general-purpose reversed phase column.

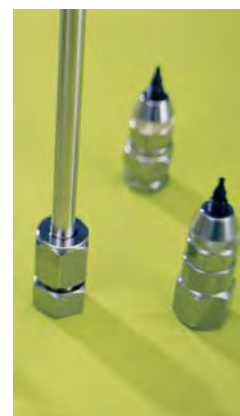
Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.0mm ID cat.#	4.6mm ID cat.#
3µm Columns					
30mm	9174331	9174332	9174333	—	9174335
50mm	9174351	9174352	9174353	—	9174355
100mm	9174311	9174312	9174313	—	9174315
5µm Columns					
30mm	9174531	9174532	9174533	—	9174535
50mm	9174551	9174552	9174553	—	9174555
100mm	9174511	9174512	9174513	9174514	9174515
150mm	9174561	9174562	9174563	9174564	9174565
200mm	9174521	9174522	9174523	—	9174525
250mm	9174571	9174572	9174573	—	9174575

To order a 2.1mm, 3.2mm, or 4.6mm ID column with a Trident™ Integral Inlet Fitting, add “-700” to the catalog number for the column.

Example: 100mm x 4.6mm ID Ultra C18 column with Trident™ Integral Inlet Fitting: 9174315-700

Nominal additional charge

For guard cartridges for these columns, see page 44.





Ultra Amino

Physical Characteristics:

particle size: 3 μ m or 5 μ m, spherical
pore size: 100Å
carbon load: 2%

pH range: 2.5 to 7.5
temperature limit: 80°C

Chromatographic Properties:

Recommended for normal phase analyses of mono- and disaccharides, or similar compounds. Also can serve as a weak anion exchanger, with aqueous buffers.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
3μm Columns				
30mm	9107331	9107332	9107333	9107335
50mm	9107351	9107352	9107353	9107355
100mm	9107311	9107312	9107313	9107315
5μm Columns				
30mm	9107531	9107532	9107533	9107535
50mm	9107551	9107552	9107553	9107555
100mm	9107511	9107512	9107513	9107515
150mm	9107561	9107562	9107563	9107565
200mm	9107521	9107522	9107523	9107525
250mm	9107571	9107572	9107573	9107575

Pinnacle II™ Amino

Physical Characteristics:

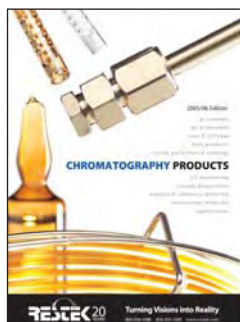
particle size: 3 μ m or 5 μ m, spherical
pore size: 110Å
carbon load: 2%

pH range: 2.5 to 7.5
temperature limit: 80°C

Chromatographic Properties:

HPLC analysis using an amino-based stationary phase is the most popular technique for routine analyses of simple sugars, using isocratic elution (e.g., acetonitrile:water, 75:25) and a refractive index detector (RID). The Pinnacle II™ Amino column is ideal for the purpose.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
3μm Columns				
30mm	9217331	9217332	9217333	9217335
50mm	9217351	9217352	9217353	9217355
100mm	9217311	9217312	9217313	9217315
5μm Columns				
30mm	9217531	9217532	9217533	9217535
50mm	9217551	9217552	9217553	9217555
100mm	9217511	9217512	9217513	9217515
150mm	9217561	9217562	9217563	9217565
200mm	9217521	9217522	9217523	9217525
250mm	9217571	9217572	9217573	9217575



for more info

Restek offers an extensive array of HPLC columns, accessories, and instrument parts. Call to request our HPLC catalog (cat. # 59241B) or visit us on the web at www.restek.com

Ultra Aqueous C18 (USP L1)**Physical Characteristics:**

particle size: 3µm or 5µm, spherical
pore size: 100Å

not end-capped
pH range: 2.5 to 7.5
temperature limit: 80°C

Chromatographic Properties:

Highly retentive and selective for reversed phase separations of polar analytes. Highly base deactivated. Compatible with highly aqueous (up to 100%) mobile phases.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
3µm Columns				
30mm	9178331	9178332	9178333	9178335
50mm	9178351	9178352	9178353	9178355
100mm	9178311	9178312	9178313	9178315
5µm Columns				
30mm	9178531	9178532	9178533	9178535
50mm	9178551	9178552	9178553	9178555
100mm	9178511	9178512	9178513	9178515
150mm	9178561	9178562	9178563	9178565
200mm	9178521	9178522	9178523	9178525
250mm	9178571	9178572	9178573	9178575

Ultra Phenyl (USP L11)**Physical Characteristics:**

particle size: 3µm or 5µm, spherical
pore size: 100Å
carbon load: 10%

fully end-capped
pH range: 2.5 to 7.5
temperature limit: 80°C

Chromatographic Properties:

High-purity, highly retentive, base-deactivated phase with alternative selectivity to straight chain hydrocarbon phases, especially for aromatic analytes.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
3µm Columns				
30mm	9105331	9105332	9105333	9105335
50mm	9105351	9105352	9105353	9105355
100mm	9105311	9105312	9105313	9105315
5µm Columns				
30mm	9105531	9105532	9105533	9105535
50mm	9105551	9105552	9105553	9105555
100mm	9105511	9105512	9105513	9105515
150mm	9105561	9105562	9105563	9105565
200mm	9105521	9105522	9105523	9105525
250mm	9105571	9105572	9105573	9105575

To order a 2.1mm, 3.2mm, or 4.6mm ID column with a Trident™ Integral Inlet Fitting, add “-700” to the catalog number for the column.

Example: 100mm x 4.6mm ID Ultra C18 column with Trident™ Integral Inlet Fitting: 9174315-700

Nominal additional charge

For guard cartridges for these columns, see page 44.

did you know?

Ultra Aqueous C18 is the ideal column for high water solubility or low organic solubility compounds that require >90% water in the mobile phase. Excellent for water soluble vitamins and organic acids.

HPLC Columns, Syringe Filters, Guard Cartridges



Pinnacle II™ C18 (USP L1)

Physical Characteristics:

particle size: 3µm or 5µm, spherical	fully end-capped
pore size: 110Å	pH range: 2.5 to 7.5
carbon load: 13%	temperature limit: 80°C

Chromatographic Properties:

Excellent choice as a general purpose C18 column. Intermediate carbon loading and surface area, suitable for a wide range of neutral hydrophobic compounds. Replaces Hypersil® ODS and Pinnacle™ C18.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.0mm ID cat.#	4.6mm ID cat.#
3µm Columns					
30mm	9214331	9214332	9214333	—	9214335
50mm	9214351	9214352	9214353	—	9214355
100mm	9214311	9214312	9214313	—	9214315
5µm Columns					
30mm	9214531	9214532	9214533	—	9214535
50mm	9214551	9214552	9214553	—	9214555
100mm	9214511	9214512	9214513	9214514	9214515
150mm	9214561	9214562	9214563	9214564	9214565
200mm	9214521	9214522	9214523	—	9214525
250mm	9214571	9214572	9214573	—	9214575

To order a 2.1mm, 3.2mm, or 4.6mm ID column with a Trident™ Integral Inlet Fitting, add "-700" to the catalog number for the column.

Example: 100mm x 4.6mm ID Ultra C18 column with Trident™ Integral Inlet Fitting: 9174315-700

Nominal additional charge

For guard cartridges for these columns, see below.

Resprep™ Syringe Filters

- Solvent-resistant polypropylene housing.
- Glass fiber prefilter ensures better flow characteristics.
- Most popular filter sizes and membrane porosities.
- Non-leaching nylon or PTFE.



Filter Diameter	Porosity	qty.	Nylon	PTFE
13mm	0.20µm	100-pk.	26066	26068
13mm	0.45µm	100-pk.	26067	26069
25mm	0.20µm	50-pk.	26070	26072
25mm	0.45µm	50-pk.	26071	26073
25mm	1.00µm	50-pk.	—	26074

please note

For additional sample preparation products, request our chromatography products catalog.

Trident™ HPLC Guard Column Cartridges

Guard Column Cartridges	3-pk. (10 x 2.1mm)	3-pk. (10 x 4.0mm)	2-pk. (20 x 2.1mm)	2-pk. (20 x 4.0mm)
Allure™ Organic Acids	916550212	916550210	916550222	916550220
Pinnacle II™ Amino	921750212	921750210	921750222	921750220
Pinnacle II™ C18	921450212	921450210	921450222	921450220
Ultra Amino	910750212	910750210	910750222	910750220
Ultra Aqueous C18	917850212	917850210	917850222	917850220
Ultra C8	910350212	910350210	910350222	910350220
Ultra C18	917450212	917450210	917450222	917450220
Ultra Phenyl	910550212	910550210	910550222	910550220



10 & 20mm Guard Cartridges

Trident™ Direct Guard Column System

Easy to Use, Low Dead Volume—The Ultimate Combination of Convenience and Column Protection

Unlike “one size fits all” guard systems, the Trident™ Direct system gives you the power to select the right level of protection for your analysis. The system offers three levels of protection and guard cartridges in four dimensions, with a variety of bonded phases to match your analytical column. The economical, leak-free cartridge design provides an unprecedented combination of convenience, economy, and reliability. The foundation of the Trident™ Direct system is a reusable direct connect holder that easily attaches to any HPLC column using CPI- or Waters®-style end fittings.* The system is available in configurations to match different protection level needs: in-line filter, in-line filter with holder for 10mm guard cartridge, and in-line filter with holder for 20mm guard cartridge. The guard cartridges are available in 2.1 and 4.0mm ID and are interchangeable within the appropriate length holder.

Description	qty.	cat.#
High-pressure filter	ea.	25082
10mm guard cartridge holder without filter	ea.	25083
10mm guard cartridge holder with filter	ea.	25084
20mm guard cartridge holder without filter	ea.	25085
20mm guard cartridge holder with filter	ea.	25086
Connection tip for Waters®-style end fittings	ea.	25088
PEEK® tip standard fittings	ea.	25087
Replacement cap frits: 4mm, 2.0µm	5-pk.	25022
Replacement cap frits: 4mm, 0.5µm	5-pk.	25023
Replacement cap frits: 2mm, 2.0µm	5-pk.	25057

*The standard PEEK® tip in Trident™ Direct systems is compatible with Parker®, Upchurch®, Valco®, and other CPI-style fittings. To use Trident™ Direct systems with Waters®-style end fittings, replace the tip with cat.# 25088.

Restek's Trident™ Integral System

- Convenient and economical leak-free guard column system, extremely easy to install.
- Versatile configuration protects against all levels of contamination.
- Integral design eliminates troublesome tubing connections.

The system's foundation consists of the analytical column configured with our exclusive Trident™ end fitting and XF fitting. This configuration contains the standard internal frit as well as a replaceable cap frit, which can easily be changed without disturbing the packed bed. Changing the external frit can reverse the effects of accumulated particles, such as high backpressure or peak distortion. To obtain this basic configuration, simply order any Restek HPLC column, and add the suffix -700 to the catalog number for the column. Nominal additional charge.

For maximum protection against contaminants and particulate matter, the system can be configured with an integral guard cartridge holder (XG-XF), a guard cartridge, and a replaceable external frit. To obtain this configuration, simply order any Restek HPLC column, add the suffix -700 to the catalog number for the column, and order the appropriate XG-XF male fitting (cat.# 25026 or 25062) and Trident™ guard cartridges.

Description	qty.	cat.#
XG-XF Fitting for 10mm Guard Cartridge	ea.	25026
XG-XF Fitting for 20mm Guard Cartridge	ea.	25062
Replacement XF Filter Fitting	ea.	25024
Replacement cap frits: 4mm, 2.0µm	5-pk.	25022
Replacement cap frits: 4mm, 0.5µm	5-pk.	25023
Replacement cap frits: 2mm, 2.0µm	5-pk.	25057



HPLC column (with -700 extension) with guard cartridge, XG-XF fitting, cap frit, and XF end fitting.

Trident™ Direct provides three levels of protection



Trident™ Direct high-pressure filter
Protection against particulate matter.



Trident™ Direct 10mm guard cartridge holder with filter
Protection against particulate matter and moderate protection against irreversibly adsorbed compounds.

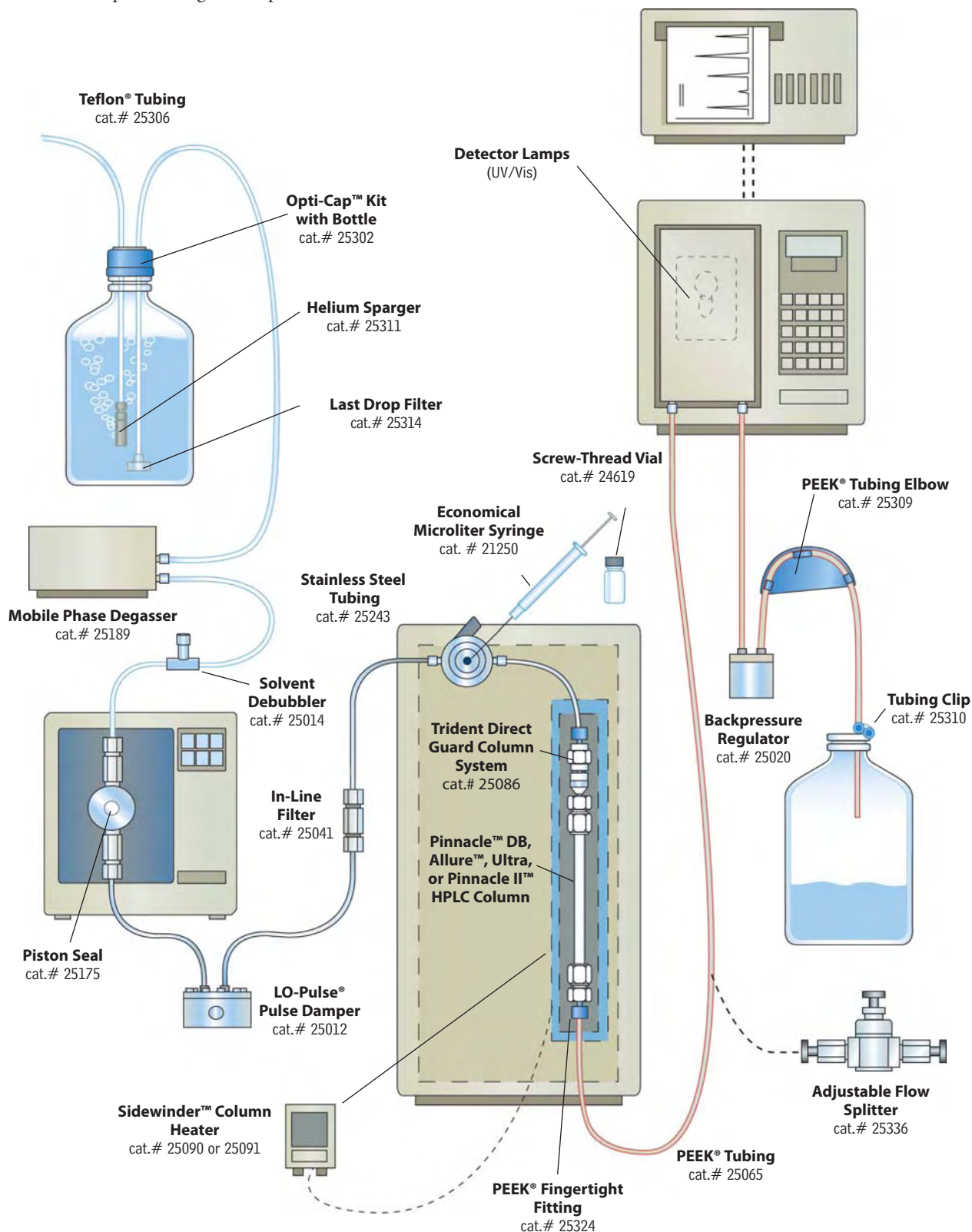


Trident™ Direct 20mm guard cartridge holder with filter
Protection against particulate matter and maximum protection against irreversibly adsorbed compounds.

HPLC Accessories

Restek has the HPLC Columns and Accessories You Need.

We offer a wide selection of HPLC replacement parts and accessories, such as mobile phase degassers, tubing, flow splitters, syringes, vials, and column protection products. Use the page numbers shown here to locate specific categories of products.



High-Pressure Frit-Type In-Line Filters

Restek's high-pressure in-line filter is a stand-alone version of the Trident™ column protection system. The filter is specifically designed for ease of use, low dead-volume, and flexibility. The filter has a replaceable, PEEK® encapsulated 316 stainless steel frit with a surface area of 12mm². The standard frit shipped with the filter has a 2.0µm porosity; however, it may be replaced with an optional 0.5µm porosity frit. Use of this filter can greatly extend column life, thereby reducing costs and saving maintenance time. Tubing OD 1/16"; Connectors—CPI

Description	Porosity	qty.	cat.#
Frit-Type In-Line Filter	2.0µm	ea.	25041
Replacement cap frits: 4mm	0.5µm	5-pk.	25023
Replacement cap frits: 4mm	2.0µm	5-pk.	25022

High-Pressure Cup-Type In-Line Filters

High-pressure cup-type filters can be used in fluid streams operating to 15,000psi. The cup-shaped filter elements have a large (2.5 cm²) surface area to give long operating lifetime. Mounted in screw-type adapters, they are easily removed for cleaning. Normally, backflushing and cleaning in an ultrasonic bath with an appropriate solvent will restore them. If they become permanently clogged, replacement elements are available.

Housings and all wetted parts are type 316 stainless steel. Filters are packaged with appropriate gland nuts and ferrules. A bulkhead type is available for thru-panel mounting. Tubing OD 1/16"; Connectors—CPI

Description	Porosity	qty.	cat.#
Cup-Type In-Line Filter	0.5µm	ea.	25000
Cup-Type In-Line Filter	2.0µm	ea.	25001
Replacement Filter Elements & Seals	0.5µm	2-pk.	25002
Replacement Filter Elements & Seals	2.0µm	2-pk.	25003

Low-Pressure Slip-On Inlet Filter for Mobile Phase Reservoir

A type 316 stainless steel tip with a Tefzel® collar seals to a corrosion-resistant type 316 stainless steel filter element. The slip-on filter easily attaches to the pump inlet line, without the use of wrenches. The universal tip accommodates standard Teflon® tubing inner diameters. The cylindrical filter is standard 10µm porosity. 1/8" OD (fits Altex, ISCO, LDC, Varian, Waters, PerkinElmer, and other pumps)

Description	qty.	cat.#
Slip-on Inlet Filter	ea.	25008

Low-Pressure CPI Inlet Filter for Mobile Phase Reservoir

A type 316 stainless steel knurled cap and Tefzel® CPI ferrule seals to 1/8" OD Teflon® tubing when finger-tightened onto the precision-machined filter holder. The filter element is replaceable. Standard 10µm porosity protects delicate pump components from particles but introduces very little pressure drop. 1/8" OD. May be used as a helium sparging diffuser.

Description	qty.	cat.#
CPI Inlet Filter	ea.	25009
Replacement Elements: 10µm filter	2-pk.	25010

Mobile Phase Spargers and Filters

These helium spargers offer an inexpensive way to prepare and maintain mobile phases free of dissolved gas. They are made from 316 stainless steel and PEEK® and are compatible with most solvents.

Description	qty.	cat.#
Sparge Filter: 2µm	ea.	25311
Inlet Filter: 10µm	ea.	25312
Inlet Filter: 20µm	ea.	25313





Last Drop Filter

The flat filter element sits parallel to the bottom of the mobile phase reservoir, allowing the filter to draw 98% of the mobile phase without drawing air into the system. Conventional cylindrical mobile phase filters begin to draw air into the system when approximately 10% of the solvent remains in the reservoir. The Last Drop Filter allows more analyses per batch of mobile phase and helps reduce hazardous waste. 22.1mm OD.

Description	qty.	cat.#
Last Drop Filter: 2µm	ea.	25314
Last Drop Filter: 10µm	ea.	25315

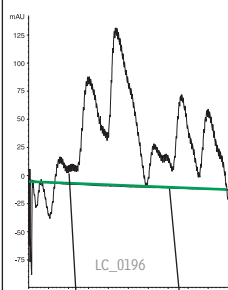


Kontes All-Glass Microfiltration Apparatus

47mm filtration apparatus with fritted glass support is recommended for routine filtration of corrosive liquids and removal of particles from HPLC solvents. The ground joint connection eliminates phthalate contamination that can occur when using silicone or neoprene stoppers. The support base has a coarse porosity glass frit and an integral vacuum connection, located above the drip tip to prevent contamination of the vacuum line with filtrate droplets. Each apparatus includes a funnel, an anodized aluminum clamp, a 47mm fritted glass support base, and a filtration flask.

Description	qty.	cat.#
300mL Funnel, 1000mL Flask	ea.	KT953825-0000
500mL Funnel, 2000mL Flask	ea.	KT953835-0000
1000mL Funnel, 4000mL Flask	ea.	KT953845-0000

Degasys Ultimate Degasser provides highly stable baselines



Mobile Phase: water:methanol
50:50
Flow: 1.0 mL/min.
Det.: UV @ 210nm

Mobile Phase Degasser

Dissolved oxygen can cause flow rate instability and increased baseline noise. Also, it has a quenching effect on fluorescence detection and increases the background of UV detectors. Dissolved gases can out-gas in the HPLC system, forming bubbles in check valves, at connections, or in detector flow cells.

In-line vacuum degassing is more effective at removing dissolved gas from mobile phases than sonication or helium sparging. In-line degassers work by withdrawing gas across a gas-permeable membrane encased in a sealed chamber. Traditionally, the membrane has been made of PTFE tubing, but the Degasys Ultimate Degasser uses tubing composed of an amorphous fluoropolymer that is 200 to 300 times more gas permeable than PTFE. This translates into the ability to use shorter tubing for removing dissolved gas. This new material also has better tubular burst strength than PTFE. To prevent cross contamination, each channel on this Degasys unit is individually encased within its own vacuum chamber.



Specifications:

Residual Oxygen ¹	Pressure Loss ¹	Internal Volume	Wetted Parts	Max Flow Rate
0.9ppm	0.24psi	500µL	Teflon® AF	7mL/min./channel
			PTFE	
			ETFE	
			PPS	

¹ At a flow rate of 1mL/min.

Description	qty.	cat.#
110V Mobile Phase Degasser (4 Channel, 7mL/min./channel)	ea.	25189
220V Mobile Phase Degasser (4 Channel, 7mL/min./channel)	ea.	25194

Solvent Debubbler

Bubbles in an HPLC system can cause check valve malfunctions and pump cavitation, seriously affecting pump performance. The debubbler removes bubbles from the fluid stream before it enters the pump.

Special geometry at the base of the housing allows bubbles entrained in the inlet fluid stream to rise and be trapped in the reservoir. The gas/liquid interface is easily visible through the translucent wall of the device. Loosening the airtight cap releases the trapped gas. The debubbler is fitted with a bracket and universal connecting tips.



Description	qty.	cat.#
Solvent Debubbler with Bracket	ea.	25014

Sonic Debubbler

- Fast.
- Easy to use.
- Less solvent waste; less clean-up.

Just touch the Sonic Debubbler to the inlet line or check valve — sonic vibrations will quickly dislodge or redissolve trapped air bubbles. Reduces downtime or conversion time from one mobile phase to another.

Description	qty.	cat.#
Sonic Debubbler (110V)	ea.	20444
Sonic Debubbler (220V)	ea.	25098



CE

Sidewinder™ Column Heater

- Easy to set up!
- Controls temperature from 5°C above ambient to 85°C.
- Lightweight, compact design fits in small spaces.
- Column holder can be placed in any orientation.
- Automatically shuts down when samples are finished.
- Power requirements of 100-240VAC.



CE

This unique design completely encloses any HPLC analytical column up to 25cm in length. Two lengths of heater jackets are available: the smaller jacket accommodates columns up to 10cm in length, while the longer one holds columns up to 25cm in length. The control module provides optimum heating performance, accuracy to within 1°C, and stability to within 0.1°C.

Description	qty.	cat.#
Temperature Control Module and Long Column Holder	ea.	25090
Temperature Control Module and Short Column Holder	ea.	25091

Mobile Phase Pre-heater

- Minimizes temperature changes, to help keep analyte peaks sharp.
- Heats mobile phase before entering heated column.

Description	qty.	cat.#
Mobile Phase Pre-heater	ea.	25099



Survival Kits for HPLC

For start-up and standard use in all HPLC systems.

The Restek Survival Kit is an invaluable spare parts kit that contains the tools and supplies essential for setting up and maintaining your HPLC system.

Restek Survival Kit includes:

- PEEK® Column Connector (beige, round body), 10-pk.
- PEEK® Tubing, 1/16" OD x 0.005" ID x 3m (red stripe), ea.
- PEEK® Tubing, 1/16" OD x 0.007" ID x 3m (yellow stripe), ea.
- PEEK® Tubing, 1/16" OD x 0.010" ID x 3m (blue stripe), ea.
- PEEK® Tubing Elbow 90°, 5-pk.
- PEEK® Tubing Elbow 180°, 5-pk.
- Teflon® Tubing, 1/8" OD x 0.063" ID x 3m (1.6mm ID), ea.
- Teflon® Tubing, 1/8" OD x 0.094" ID x 3m (2.4mm ID), ea.
- Tubing Clip, 5-pk.
- ValvTool Wrench, ea.
- Open-End Wrenches (1/4" x 5/16"), 2-pk.
- Clean-Cut™ Tubing Cutter, ea.
- Replacement Blade for Clean-Cut™ Cutter, ea.
- PEEK® Union Connector 1/16", 2-pk.
- Sparge Filter: 2µm, ea.
- Inlet Filter: 10µm, ea.

Stainless Steel Survival Kit includes:

- HPLC Capillary Tubing, SS, 1/16" x 0.005" x 5cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.005" x 10cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.005" x 20cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.005" x 30cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.007" x 5cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.007" x 10cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.007" x 20cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.007" x 30cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.010" x 5cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.010" x 10cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.010" x 20cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.010" x 30cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.020" x 5cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.020" x 10cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.020" x 20cm, 3-pk.
- HPLC Capillary Tubing, SS, 1/16" x 0.020" x 30cm, 3-pk.
- 1/16" Rheodyne® Style Nut, 10-pk.
- 1/16" Rheodyne® Style Ferrule, 10-pk.
- ValvTool Wrench, ea.
- 1/16" Stainless Steel Ferrules, 10-pk.
- 1/16" Stainless Steel Nuts, 10-pk.
- Zero-Dead-Volume Internal Union, ea.



Restek Survival Kit

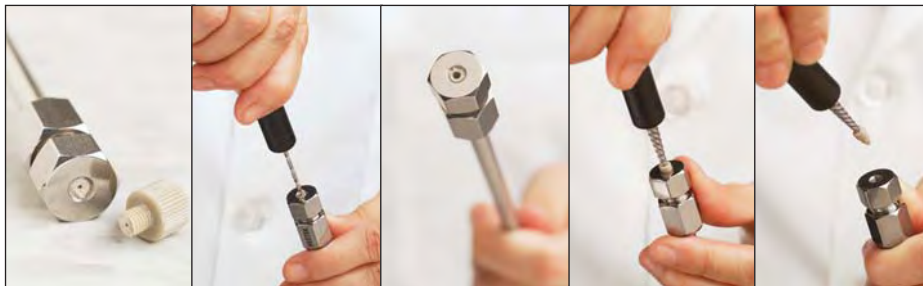


Stainless Steel Survival Kit

Description	qty.	cat.#
Restek Survival Kit for HPLC	kit	25322
Stainless Steel Survival Kit for HPLC	kit	25097

PEEK® Fitting Extractor

Drill into the broken fitting, then screw the extractor into the fitting and remove it easily.

**Description**

PEEK® Fitting Extractor

qty.

ea.

cat.#

25325

PEEK® Union Connector

Allows you to quickly and reliably connect two pieces of 1/16-inch tubing. End fittings included.

Description

PEEK® Union Connector 1/16"

qty.

2-pk.

cat.#

25323

Zero-Dead-Volume Internal Union

Ends of tubing seat squarely at bottoms of fitting details. 300 series stainless steel. For 1/16-inch OD tubing. Stainless steel ferrules included.

Description**Union Bore****Valco® #****qty.****cat.#**

Internal Union

0.15mm

ZU1XC

ea.

20147

Internal Union

0.25mm

ZU1C

ea.

20148

Internal Union

0.75mm

ZU1

ea.

20149

Internal Union

1/16"

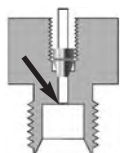
ZU1T

ea.

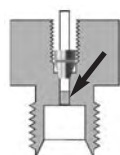
20150



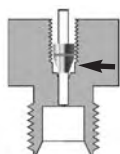
Figure 1 Problems arise from incompatible fittings.



Tubing and ferrule seated correctly



Tubing not seated, causing dead volume



Ferrule can't seal

Improving Column Connections

A good connection is necessary for trouble-free chromatography. Connecting incompatible fittings from different manufacturers can result in leaks, poor peak shape, and increased void volume (Figure 1). Each type of end-fitting has a unique seat depth or style. Generally, Restek, Valco®, Parker, and Upchurch Scientific fittings are interchangeable; whereas Waters™, SSI, Rheodyne®, and Gasukura fittings are not (Figure 2).

Our wrenchless universal 10-32 PEEK® column connector (cat.# 25015) can be used with any style of end fitting, and all 1/16-inch tubing. It is reusable and will adjust to any seat, depth, or style.

Figure 2 Fitting styles differ among various manufacturers.



Parker



Valco®



Waters™



Rheodyne®

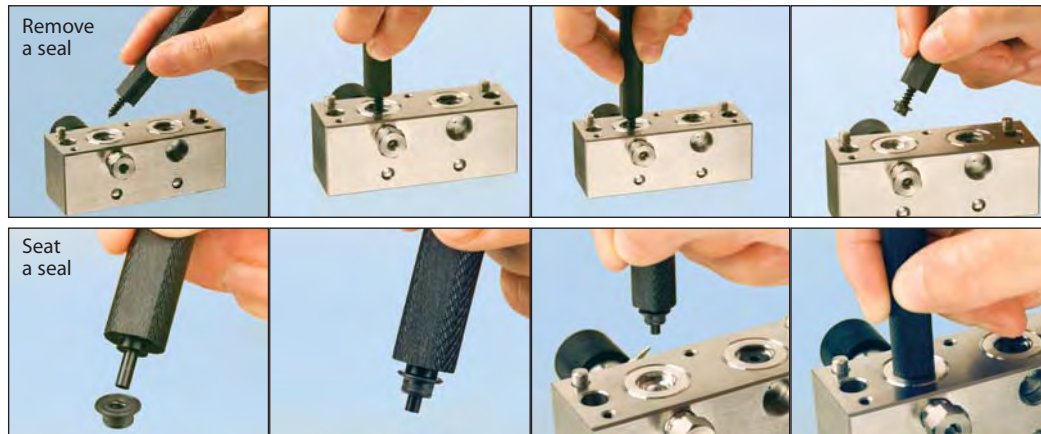


Swagelok® Parker A-Lok

HPLC Piston Seal Insertion Tool

- Simplify pump maintenance.
- One end removes old piston seal, other end easily and securely installs new seal.

Do you have to search for a paper clip or screw to remove worn seals from your HPLC pump? Then, once you get the old seal out, do you struggle to correctly seat the new seal? Now Restek has a tool that can help. Use one end to remove your old seal, then simply slip your new seal on the other end and push it flush into position. The tool cannot mar the surrounding metal surface of the pump housing.



Use the flat side of the Piston Seal Insertion Tool to seat a Waters™ face seal.

Description	qty.	cat. #
HPLC Piston Seal Insertion Tool	ea.	21356

Secure-Fit Fittings

A good connection between HPLC components is necessary for trouble-free chromatography. Secure-Fit connectors from Restek and Selerity Technologies ensure a consistent, leak-free seal—and they eliminate excess dead volume! An internal spring mechanism holds the capillary tubing at the proper depth in the female fitting. This seal is maintained while you finger-tighten the nut. These fittings are available in stainless steel or PEEK®, and in a variety of tubing lengths and internal diameters.

Length	0.005" ID	0.007" ID	0.010" ID
Stainless Steel Secure-Fit Fittings—Single Ended			
6cm	25181	25185	25190
10cm	25182	25186	25191
20cm	25183	25187	25192
30cm	25184	25188	25193
Stainless Steel Secure-Fit Fittings—Double Ended			
10cm	25208	25211	25214
20cm	25209	25212	25215
30cm	25210	25213	25216
PEEK® Secure-Fit Fittings—Single Ended			
6cm	25230	25234	25217
10cm	25231	25235	25218
20cm	25232	25236	25219
30cm	25233	25237	25220
PEEK® Secure-Fit Fittings—Double Ended			
10cm	25221	25224	25227
20cm	25222	25225	25228
30cm	25223	25226	25229



HPLC Accessories



PEEK® Tubing Elbows

Tubing Elbows (90° and 180°) are ideal for routing 1/16-inch PEEK® tubing through your system. Simply snap the tubing into the elbow. Prevent pinching of PEEK® tubing which can cause high pressure.

Description	qty.	cat.#
PEEK® Tubing Elbow 90°	5-pk.	25308
PEEK® Tubing Elbow 180°	5-pk.	25309



Tubing Dressing Tool

Opens stainless steel tubing bore and removes burrs. For 1/16-inch OD tubing or 1/8-inch OD tubing.

Description	qty.	cat.#
1/16" Tubing Dressing Tool	ea.	20188
Replacement Insert for 1/16" Tubing Dressing Tool	ea.	20189
1/8" Tubing Dressing Tool	ea.	20190
Replacement Insert for 1/8" Tubing Dressing Tool	ea.	20191



Clean-Cut™ Tubing Cutter

- Burr-free, perpendicular cuts that will not distort the tubing OD or close the ID.
- Use on PEEK®, Teflon®, Tefzel®, other polymeric tubing.

Description	qty.	cat.#
Clean-Cut™ Tubing Cutter	ea.	25069
Replacement Blade for Clean-Cut™ Cutter	ea.	25070



Universal 10-32 PEEK® Column Connectors and Plugs

Universal PEEK® Connectors allow easy installation of all 1/16-inch tubing, including stainless steel. See "Improving Column Connections" data on page 254.

Description	qty.	cat.#
PEEK® Column Connector (beige, round body)	10-pk.	25015
PEEK® Column Plug (black)	10-pk.	25016
PEEK® Fingertight Fittings (blue, flat-sided)	10-pk.	25324



Rheodyne® Style Nut and Ferrule

Replacement long nut for connecting stainless steel tubing to a Rheodyne® 6-port valve or other Rheodyne® part.

Description	qty.	cat.#
1/16" Rheodyne® Style Nut	10-pk.	25095
1/16" Rheodyne® Style Ferrule	10-pk.	25096



ValvTool Wrench

The ValvTool is a time-saving device that provides easy access to many hard-to-reach Rheodyne® or Valco® valves. For 1/4-inch nuts.

Description	qty.	cat.#
ValvTool Wrench	ea.	25321



Inert PEEK® Tubing

- Replaces stainless steel, titanium, Teflon® and Tefzel® tubing.
- Less oxygen permeable and more temperature resistant (to 350°C) than Teflon® or Tefzel® tubing.
- Use with PEEK® fingertight or flangeless fittings.
- Use to 7,000psi.

Description	Color Code	qty.	cat.#
PEEK® Tubing, 1/16" OD x 0.0025" ID x 1m (natural)	natural	3-pk.	25320
PEEK® Tubing, 1/16" OD x 0.005" ID x 3m (red stripe)	red stripe	ea.	25065
PEEK® Tubing, 1/16" OD x 0.007" ID x 3m (yellow stripe)	yellow stripe	ea.	25066
PEEK® Tubing, 1/16" OD x 0.010" ID x 3m (blue stripe)	blue stripe	ea.	25067
PEEK® Tubing, 1/16" OD x 0.020" ID x 3m (orange stripe)	orange stripe	ea.	25068

HPLC Stainless Steel Capillary Tubing

- 316 grade stainless steel.
- Precise pre-cut lengths.
- Smooth surface finish.
- Ultra clean.



Whether you need to replace system tubing as part of your troubleshooting or are looking to reduce the dwell volume of your system as you move to narrower columns, Restek has the quality tubing in the lengths and IDs you need. Each ID is color-coded so it is easy to identify and replace correctly.

Length	ID	OD	qty.	cat.#
5cm	0.005"	1/16"	3-pk.	25240
10cm	0.005"	1/16"	3-pk.	25241
20cm	0.005"	1/16"	3-pk.	25242
30cm	0.005"	1/16"	3-pk.	25243

Teflon® Tubing

- Ideal for mobile phase inlet lines.
- Chemically inert.
- Use to 500psi and 80°C.

Description	qty.	cat.#
Teflon® Tubing, 1/8" OD x 0.063" ID x 3m (1.6mm ID)	ea.	25306
Teflon® Tubing, 1/8" OD x 0.094" ID x 3m (2.4mm ID)	ea.	25307



Tubing Clip

Securely holds 1/16-inch or 1/8-inch tubing in beaker, flask, or bottle up to 4mm thick.

Description	qty.	cat.#
Tubing Clip	5-pk.	25310



LCLocker™ HPLC Organizer



Deluxe BenchBooster™ Organizer



Mini pHPerch™ Storage Unit



TopLoader™ BalanceBank™ Storage Unit



HPLC 30-Column Storage Cabinet



Book Holders



Open Supply Bins, 13-bin unit



Glove Box Dispensers

Description	dimensions	qty.	cat.#
LCLocker™ HPLC Organizer	24 x 12 x 6"	ea.	25149
Deluxe BenchBooster™ Organizer	24 x 7 x 12"	ea.	25150
Mini pHPerch™ Storage Unit	13 x 12 x 6"	ea.	25147
TopLoader™ BalanceBank™ Storage Unit	12 x 12 x 7"	ea.	25148
HPLC 30-Column Storage Cabinet	17 7/8 x 15 x 2 7/8"	ea.	25159
Book Holder, Small	0.75" ID	ea.	25151
Book Holder, Large	1.5" ID	ea.	25152
Open Supply Bin, 4-bin Unit	24 x 12 x 10"	ea.	25153
Open Supply Bin, 5-bin Unit	12 x 16 x 10.5"	ea.	25154
Open Supply Bin, 13-bin Unit	12 x 7.5 x 12"	ea.	25155
Glove Box Dispenser, Single	5 7/8 x 11 1/2 x 4 1/2"	ea.	25156
Glove Box Dispenser, Double	10 7/8 x 11 1/2 x 4 1/2"	ea.	25157
Glove Box Dispenser, Triple	15 7/8 x 11 1/2 x 4 1/2"	ea.	25158

Analytical Reference Materials

free data packs

Restek now offers free downloadable data packs for analytical reference material products.

Just visit our website at www.restek.com/datapacks.

Enter the catalog number and serial number for the product you ordered and obtain a printable PDF file.

Marine Oil FAME Mix (20 components)

Chain	Description	% by Weight
C14:0	methyl myristate	6.0
C14:1	methyl myristoleate	1.0
C16:0	methyl palmitate	16.0
C16:1	methyl palmitoleate	5.0
C18:0	methyl stearate	8.0
C18:1	methyl oleate	13.0
C18:1	methyl vaccenate	4.0
C18:2	methyl linoleate	2.0
C18:3	methyl linolenate	2.0
C20:0	methyl arachidate	1.0
C20:1	methyl 11-eicosenoate	9.0
C20:2	methyl 11-14-eicosadienoate	1.0
C20:4	methyl arachidonate	3.0
C20:3	methyl 11-14-17-eicosatrienoate	1.0
C20:5	methyl eicosapentaenoate	10.0
C22:0	methyl behenate	1.0
C22:1	methyl erucate	3.0
C22:6	methyl docosahexaenoate	12.0
C24:0	methyl lingnocerate	1.0
C24:1	methyl nervonate	1.0

cat. # 35066 (100mg)

NLEA FAME Mix (28 components)

Chain	% by Weight	Chain	% by Weight
C4:0	1.5	C18:1(<i>trans</i> -9)	2.5
C6:0	1.5	C18:1(<i>cis</i> -9)	15.0
C8:0	2.0	C18:2(all- <i>trans</i> -9,12)	2.5
C10:0	2.5	C18:2(all- <i>cis</i> -9,12)	10.0
C11:0	2.5	C18:3(all- <i>cis</i> -9,12,15)	5.0
C12:0	5.0	C20:0	2.5
C13:	2.5	C20:1(<i>cis</i> -11)	1.5
C14:0	2.5	C20:5(all- <i>cis</i> -5,8,11,14,17)	2.5
C14:1(<i>cis</i> -9)	1.5	C22:0	2.5
C15:0	1.5	C22:1(<i>cis</i> -13)	1.5
C16:0	10.0	C22:6	2.5
C16:1(<i>cis</i> -9)	5.0	(all- <i>cis</i> -4,7,10,13,16,19)	2.5
C17:0	2.5	C23:0	1.5
C18:0	5.0	C24:0	2.5
		C24:1(<i>cis</i> -15)	2.5

30mg/mL total in methylene chloride, 1mL/ampul
cat. # 35078 (ea.)

cis/trans FAME Mix (8 components)

Description	% by Weight
methyl elaidate (C18:1 <i>trans</i> -9)	10.0
methyl linoleate (C18:2 <i>cis</i> -9,12)	20.0
methyl oleate (C18:1 <i>cis</i> -9)	10.0
methyl petroselinic acid (C18:1 <i>cis</i> -6)	8.0
methyl petroselinic acid (C18:1 <i>trans</i> -6)	8.0
methyl stearate (C18:0)	20.0
methyl transvaccenate (C18:1 <i>trans</i> -11)	12.0
methyl vaccenate (C18:1 <i>cis</i> -11)	12.0

10mg/mL total in methylene chloride, 1mL/ampul
cat. # 35079 (ea.)

Food Industry FAME Mix (37 components)

Chain	% by Weight	Chain	% by Weight
C4:0	4.0	C18:2(all- <i>cis</i> -9,12)	2.0
C6:0	4.0	C18:3(all- <i>cis</i> -6,9,12)	2.0
C8:0	4.0	C18:3(all- <i>cis</i> -9,12,15)	2.0
C10:0	4.0	C20:0	4.0
C11:0	2.0	C20:1(<i>cis</i> -11)	2.0
C12:0	4.0	C20:2(all- <i>cis</i> -11,14)	2.0
C13:	2.0	C20:3(all- <i>cis</i> -8,11,14)	2.0
C14:0	4.0	C20:3(all- <i>cis</i> -11,14,17)	2.0
C14:1(<i>cis</i> -9)	2.0	C20:4(all- <i>cis</i> -5,8,11,14)	2.0
C15:0	2.0	C20:5(all- <i>cis</i> -5,8,11,14,17)	2.0
C15:1(<i>cis</i> -10)	2.0	C21:0	2.0
C16:0	6.0	C22:0	4.0
C16:1(<i>cis</i> -9)	2.0	C22:1(<i>cis</i> -13)	2.0
C17:0	2.0	C22:2(all- <i>cis</i> -13,16)	2.0
C17:1(<i>cis</i> -10)	2.0		
C18:0	4.0	(all- <i>cis</i> -4,7,10,13,16,19)	2.0
C18:1(<i>trans</i> -9)	2.0	C23:0	2.0
C18:1(<i>cis</i> -9)	4.0	C24:0	4.0
C18:2(all- <i>trans</i> -9,12)	2.0	C24:1(<i>cis</i> -15)	2.0

30mg/mL total in methylene chloride, 1mL/ampul
cat. # 35077 (ea.)

Fruit Juice Organic Acid Standard

citric acid	2000µg/ml	quinic acid	2000
fumaric acid	10*	tartaric acid	2000
malic acid	2000		

In water, 1mL/ampul

cat. # 35080 (ea.)

In water, 5mL/ampul

cat. # 35081 (ea.)

*Fumaric acid is a trace impurity in malic acid, as well as an added component of the mix. The amount of fumaric acid in malic acid will not affect the stated concentration of malic acid, but can represent a significant and variable deviation from the low concentration of fumaric acid stated to be in the mix. All other components of the mix are at the specified concentration.

Fragrance Materials Test Mix

The Fragrance Materials Association (FMA) has proposed a method for analyzing essential oils on polar and non-polar capillary GC columns. A performance evaluation mixture should be used to aid in detecting inlet problems, stationary phase degradation, loss of resolution, changes in sensitivity, and the presence of reactive sites in the sample pathway. Our test mix is consistent with the mixture proposed by the FMA. The required 5% test solution is made by diluting the 0.5mL of neat mixture to 10mL with acetone. The working solution will be stable for up to one week if transferred to a dark container and stored refrigerated.

benzyl salicylate	362 parts	geraniol	6 parts
cinnamic aldehyde	5 parts	hydroxycitronellal	50 parts
cinnamic alcohol	3 parts	d-limonene	200 parts
cinnamyl acetate	3 parts	thymol crystal	3 parts
ethyl butyrate	362 parts	vanillin	1 part
eucalyptol	5 parts	benzoic acid	1% of mix

Neat, 0.5mL in an amber ampul

cat. # 31807 (ea.)

No data pack available.

HPLC Reversed Phase Test Mix #1

Routine analysis using this product can assist in determining the need to perform column and/or system maintenance.

benzene	3.00mg/mL
uracil	0.02
naphthalene	0.50
biphenyl	0.06

In methanol:water (75:25), 1mL/ampul

cat. # 35005 (ea.)

HPLC Normal Phase Test Mix #1

Routine analysis using this product can assist in determining the need to perform column and/or system maintenance.

benzene	1.00mg/mL
benzaldehyde	0.04
benzyl alcohol	3.00
4-methoxybenzyl alcohol	2.00

In hexane, 1mL/ampul

cat. # 35004 (ea.)

Carbohydrate HPLC Performance Check Mix

Performance qualification (PQ) determines the precision of the HPLC system. Our performance check mix for HPLC / RI consists of five simple sugars in varied concentrations. We prepare the reference material in water, lyophilize it, and pack it dry for enhanced stability.

glucose	2.0µg/mL*
fructose	2.1
lactose	4.4
maltose	4.5
sucrose	4.0

Dry components in 4mL screw-cap vial.

Reconstitute in 1mL acetonitrile:water (75:25) to 2.0, 2.1, 4.4, 4.5, 4.0 mg/mL, respectively.

cat. # 31809 (ea.)

*Final concentration when 1mL solvent added.

Grob Test Mix (Capillary GC)

nC10-FAME	0.42mg/mL
nC11-FAME	0.42
nC12-FAME	0.41
2,3-butanediol	0.53
dicyclohexylamine	0.31
2,6-dimethylaniline	0.32
2,6-dimethylphenol	0.32
2-ethylhexanoic acid	0.38
nonanal	0.40
1-octanol	0.36
undecane	0.29
decane	0.28

In methylene chloride, 1mL/ampul

cat. # 35000 (ea.)

searching for the **perfect** solution?

Restek, "the company chromatographers trust", should be your first choice for custom-made reference materials. Maximum convenience, maximum value, minimum time spent blending calibration mixtures in your laboratory.

- Quotations supplied quickly.
- Mixtures made to your EXACT specifications.
- Most reference materials shipped within 5-10 working days after receipt of your order*

We have over 2,000 pure, characterized, neat compounds in our inventory!

For our Custom Reference Materials Request Form, see page 48.

* Availability of raw materials and final product testing requested may affect delivery of some mixtures.



Custom Reference Materials Request Form

Take these **eight** steps to create the right solution:

1. Mixture Description: _____
2. Solvent: _____
3. Number of Components: _____
4. Volume per ampul (select): 1mL, 2mL, 5mL, 10mL or other _____ mL
5. Quantity of ampuls: _____
6. Testing and documentation that best meets your requirements:
 - ☐ Gravimetric Documentation: Lot Sheet with balance printout attached.
 - ☐ Qualitative Documentation: Certificate of Composition, Chromatogram, and Gravimetric Documentation.
 - ☐ Quantitative Documentation: Certificate of Analysis and Data Pack.

7. Compound(s): (list or attach sheet; include CAS number)

Compound 01: _____	Concentration: _____
Compound 02: _____	Concentration: _____
Compound 03: _____	Concentration: _____
Compound 04: _____	Concentration: _____
Compound 05: _____	Concentration: _____
Compound 06: _____	Concentration: _____
Compound 07: _____	Concentration: _____
Compound 08: _____	Concentration: _____
Compound 09: _____	Concentration: _____
Compound 10: _____	Concentration: _____
Compound 11: _____	Concentration: _____
Compound 12: _____	Concentration: _____
Compound 13: _____	Concentration: _____
Compound 14: _____	Concentration: _____
Compound 15: _____	Concentration: _____
Compound 16: _____	Concentration: _____
Compound 17: _____	Concentration: _____
Compound 18: _____	Concentration: _____
Compound 19: _____	Concentration: _____
Compound 20: _____	Concentration: _____

8. Concentration Units

- ☐ mg/mL
 ☐ µg/mL
 ☐ ng/mL
 ☐ vol./wt. %
 ☐ wt./wt. %
 ☐ other _____

Contact Information:

Name: _____ Date: _____

Company/Location: _____

Phone #: _____ FAX #: _____

E-mail: _____

U.S. Customers

FAX#: (814) 355-2895

email: standards@restekcorp.com

online form: www.restek.com

International Customers

Contact Your

Restek Representative.

ALL mixtures are produced in accordance with our ISO 9001:2000 registration.

Analytical balances are calibrated daily at seven mass levels using NIST traceable weights.

ALL raw materials used are a minimum of 97% pure unless otherwise specified.



Website NEW : www.chromalytic.com.au E-mail : info@chromtech.net.au Tel: 03 9762 2034 . . . in AUSTRALIA

Three simple words...

Plus 1™

Exceeding your expectations in everything we do.

Innovation

Turning visions into reality™.

Execution

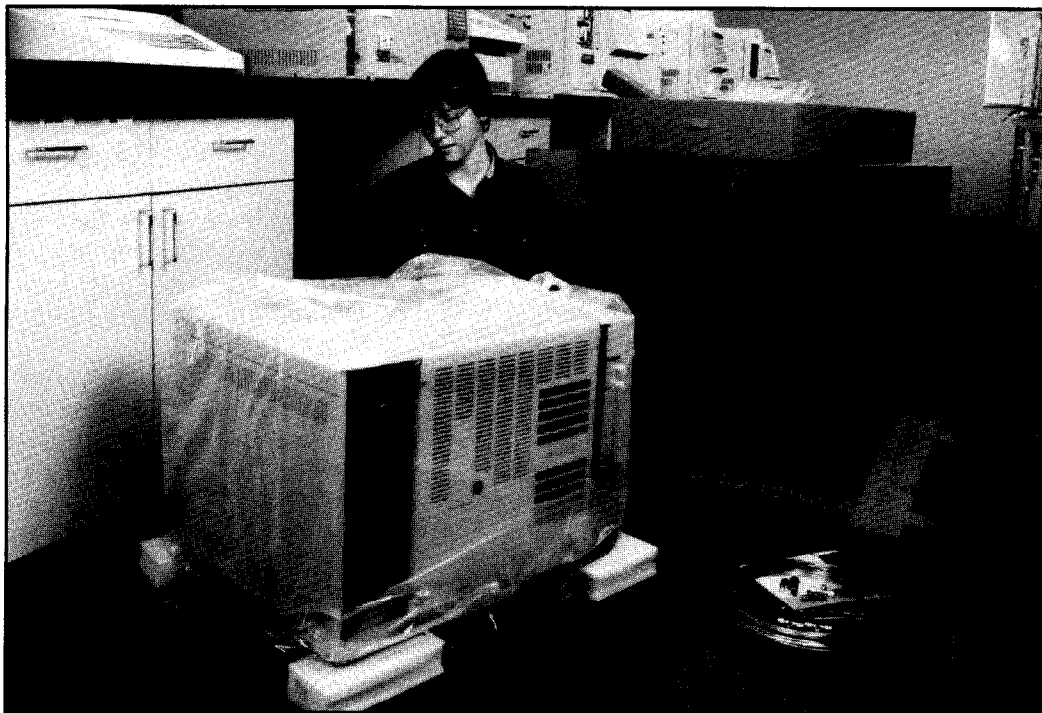
On-time delivery of products and services.

Restek's vision is to be the company that chromatographers trust by providing the highest quality, most innovative products and services throughout the world.

We will soon reach our goal of 100% employee ownership. As owners, our success depends on your success.



A Guide to GC Set-up



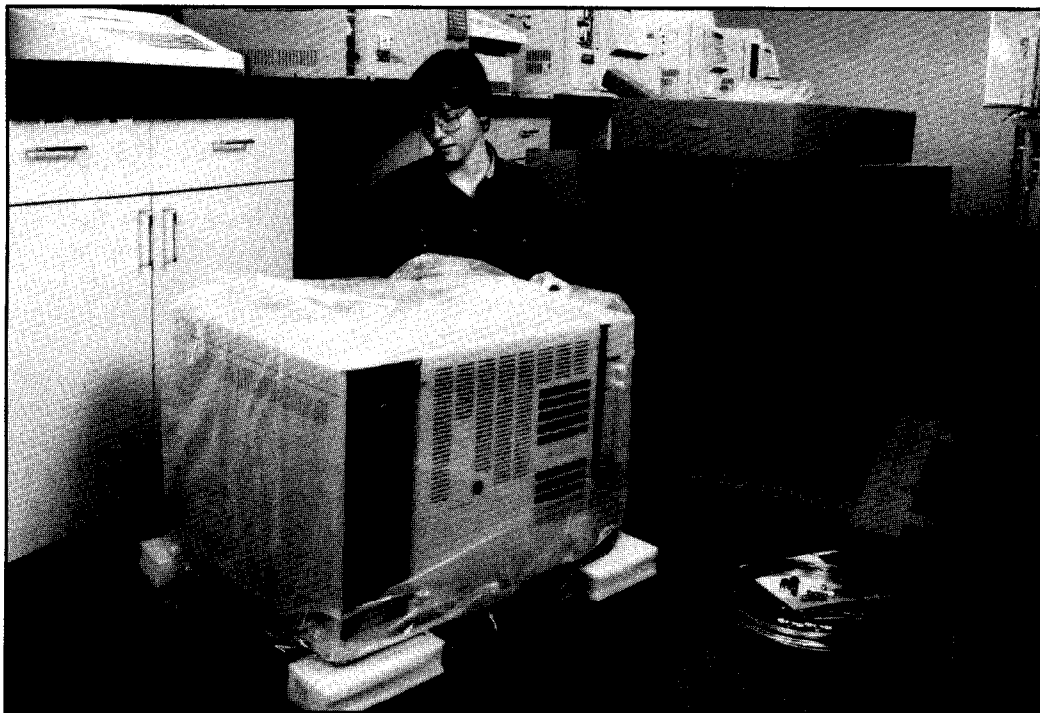
This guide covers:

- Pre-installation instructions
- Tools required to plumb a GC
- Choosing the Proper GC gases
- Plumbing Gases to the GC
- GC gas purification
- Leak checking
- Instrument logbook
- Accessories

RESTEK
CORPORATION

RESTEK Australian Distributor - Chromalytic Technology Pty Ltd . . . Fax : +61 3 9761 1169

A Guide to GC Set-up



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RESTEK
CORPORATION

RESTEK Australian Distributor - Chromalytic Technology Pty Ltd . . . Fax : +61 3 9761 1169

2
Gas chromatographs are shipped with an instrument manual that includes information on the pneumatics, electronics, computer hardware, and software installed in the new instrument. Unfortunately, practical instruction on how to set up the gas chromatograph is one area that many manuals do not adequately cover. This guide presents the basic steps in setting up a new GC and useful information on efficient and proper installation.

Index

Pre-Installation Instructions	2
Tools Required to Plumb the GC	2
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Plumbing Gases to the GC	4
GC Gas Purification	8
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Accessories	13
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Pre-Installation Instructions

The first, and perhaps most important, rule of setting up a new GC is to never throw anything away. All items are grouped and labeled by the manufacturer. Keeping all parts and manuals together will significantly reduce the chance of misplacing critical documentation or spare parts. It is a good idea to save the original box and packing materials in case the GC must be returned to the manufacturer. The GC oven fan is often bolted in place during transportation. The shipping bolt must be removed from the back of the GC before turning on.

Next, determine where to install the GC. Allocate sufficient bench space to permit installation of recorders, chromatography data systems, autosamplers, and other GC equipment. Two inches of space at the sides of the GC permits free air circulation. Allow two feet of access space at the rear of the GC for ease of service or future plumbing changes. Make the installation site accessible to easily change traps and connect gas lines.

Do not place the GC near a heating or air conditioning vent. Variations in room temperature can affect the heated zones of the GC. Chromatographically this problem is seen as retention time or baseline instability as the heating or AC units cycle on and off. A constant room temperature and a site free of hot or cold spots ensures optimum GC performance.

If the lab is not equipped with existing gas lines, set-up the GC in a location near the gas source. This will minimize the amount of tubing required to plumb new instruments. Continuous lengths of tubing between the cylinder and GC manifold should be used to eliminate the possibility of fitting leaks. If several GCs are being plumbed on the same carrier gas line, connecting tees should be easily accessible for leak checking and troubleshooting. (Do not hide tees or connectors in a ceiling or wall. This makes it difficult to periodically leak check!)

Determine the power requirements of the GC. If the power requirement is less than 15 amps, the instrument may be plugged into a 15 or 20 amp branch circuit. If the unit draws 15 amps or more, the GC power cord will have a 20 amp plug and must be plugged into a 20 amp circuit (a 20 amp plug looks similar to a standard three-prong plug, except that one prong is turned at a right angle towards the other one). If the plug on the GC doesn't fit the outlets in your lab, consult a qualified electrician before proceeding!

Generally, only one GC should be plugged into a single 15 or 20 amp branch circuit. Plugging multiple GCs into the same electrical circuit may cause the circuit breaker to trip on occasions when two instruments are heating at the same time.

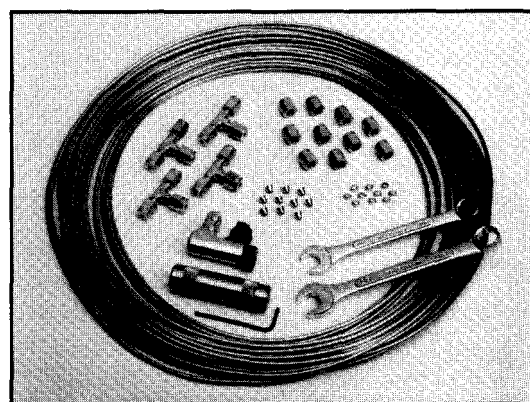
If possible, integrators or data systems should be plugged into the same outlet or circuit as the GC from which it is acquiring data. This will help to prevent ground loop currents from developing between the two instruments, which can contribute to baseline noise. To further reduce electrical noise, use high quality, shielded signal cables and keep the cables as short as possible.

Tools Required to Plumb the GC

Once the instrument site is prepared, you are ready to consider what gases are needed for the GC. Items necessary to install a new GC include the following:

(See *pages 13-25 for catalog numbers.*)

Wrenches (1/8, 1/4, 7/16, 9/16, and 1/2-inch) • Phillips & flat head screwdrivers • Solvent rinsed & heat treated stainless steel tubing • Hoke plug valves • SS diaphragm regulators • MINICYL regulators • Ferrules • Tubing cutter • Tubing bender • Reamer • Files • Replacement fittings • Adjustable wrench • Teflon tape • Electronic leak detector • Brass tees • Swagelok nuts & ferrules • Pigtail fittings • Traps • Reducers • Septa • Deactivated sleeves • O-rings • Capillary column

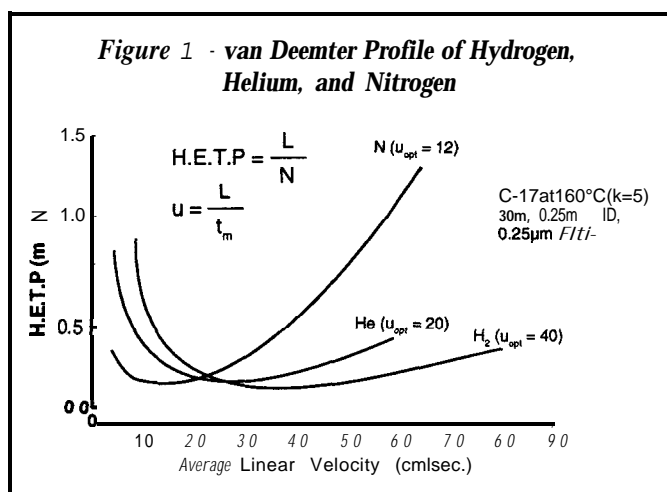


3 Choosing the Proper GC Gases

Using the correct carrier and detector gases are an important factor in installing a new GC. The five gases commonly used as carrier gas and detector fuels in capillary gas chromatography are helium, hydrogen, nitrogen, argon-methane, and air. The types of gases necessary are partly determined by the detection system used. Factors to consider for each individual gas are discussed below.

Carrier gas choice

Carrier gases that exhibit a broad minimum on a van Deemter profile are essential in obtaining optimum performance. Volumetric flow through a capillary column is affected by temperature. When temperature programming from ambient to 300°C, the flow rate can decrease by 40 percent. A carrier gas that retains high efficiency over a wide range of flow rates and temperatures is essential in obtaining good resolution throughout a temperature programmed run. Figure 1 shows the van Deemter profile for hydrogen, helium, and nitrogen carrier gases.



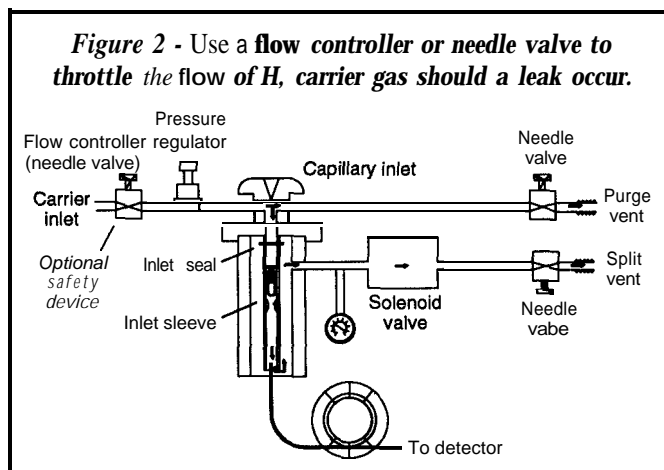
Hydrogen is the fastest carrier gas (u_{opt}), with an optimum linear velocity of 40cm/sec, and exhibits the flattest van Deemter profile. Helium is the next best choice; with an optimum linear velocity of $u_{opt} = 20$ cm/sec. Nitrogen's performance is inferior with capillary columns because of its slow linear velocity, $u_{opt} = 12$ cm/sec. Argon-methane has a slower optimum linear velocity than nitrogen and is not recommended for use as a carrier gas with capillary columns. Air is not recommended as a carrier gas because it can cause stationary phase oxidation.

With hydrogen and helium as carrier gases, the minimum H.E.T.P. values can be maintained over a broader range of linear velocities than with nitrogen, and high linear velocities can be used without sacrificing efficiency. Nitrogen is beneficial only when analyzing highly volatile gases under narrow temperature ranges where increasing stationary phase interaction is desirable. Otherwise, the use of N_2 results in longer analysis times and a loss of resolution for compounds analyzed on a wide temperature range.

Exert Caution when Using Hydrogen as a Carrier Gas

Hydrogen is explosive when concentrations exceed 4% in air. Proper safety precautions should be utilized to prevent an explosion within the column oven. Most gas chromatographs are designed with spring loaded doors, perforated or corrugated metal column ovens, and back pressure/flow controlled pneumatics to minimize the hazards when using hydrogen carrier gas. Additional precautions include:

- Frequently checking for leaks using an electronic leak detector (Restek's Leak Detective™, cat.# 21607, 110 volts/cat.# 21609, 220 volts).
- Using electronic sensors that shut down the carrier gas flow in the event of pressure loss.
- Minimizing the amount of carrier gas that could be expelled in the column oven if a leak were to occur by installing a flow controller (needle valve) prior to the carrier inlet bulkhead fitting to throttle the flow of gas (for head pressure controlled systems only) as shown in Figure 2.



Fully open the flow controller (needle valve) and obtain the proper column head pressure, split vent flow, and septum purge flow rates. Decrease the needle valve flow rate until the head pressure gauge begins to drop (throttle point). Next, increase the flow controller (needle valve) setting so that the right amount of flow is available to the system. Should a leak occur, the flow controller will throttle the flow, preventing a large amount of hydrogen from entering the oven.

Make-up and Detector Fuel Gases

Choosing the correct make-up and detector gases will depend on both the detector and application. Most GC detectors operate best with a total gas flow of approximately 30ml/min. to ensure high sensitivity and excellent peak symmetry. Refer to your GC manual for optimum flow rates on different instruments. Carrier gas flows for capillary columns range from 0.5 to 10ml/min. which are well below the range where most detectors exhibit optimal performance. To minimize detector dead volume, make-up gas is often added at the exit end of the column to increase the total flow entering the detector. Make-up gas helps to efficiently sweep detector dead volume thereby enhancing detector sensitivity. Make-up

gas can be added directly to the hydrogen flame gas for flame ionization detectors (FID), nitrogen phosphorous detectors (NPD), and flame photometric detectors (FPD) or added to the column effluent by an adaptor fitting. However, GCs such as Perkin-Elmer and Fisons do not require make-up gas.

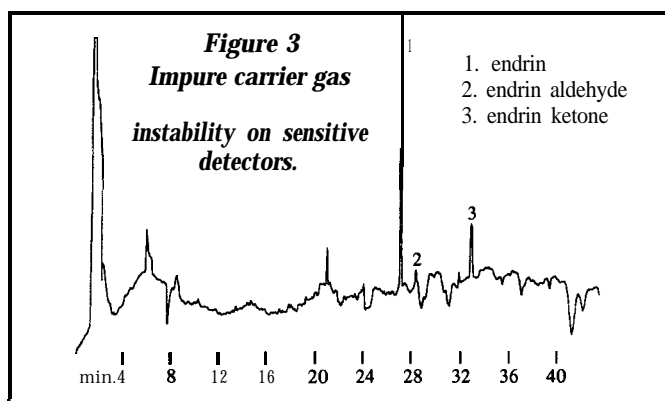
Combustion type detectors (FID, NPD, FPD) use three gases: make-up, hydrogen (fuel gas), and air (combustion/oxidizing gas). For non-combustion detectors, such as the thermal conductivity detector (TCD), electron capture (ECD), and photo ionization detector (PID), only carrier and make-up gases are required. In the case of the electrolytic conductivity detector (ELCD), the make-up gas is hydrogen, as a reaction gas in the halogen and nitrogen mode or air in the sulfur mode. Table I shows recommended gases for various detectors.

Table I - Carrier and detector fuel gases used with various GC detectors:

Detector	TCD	ECD	FID	NPD	PPD	ELCD	PID
Carrier Gases	He	He	He	He	He	He	He
	H ₂		H ₂	H ₂	H ₂	H ₂	H ₂
	N ₂	N ₂	N ₂	N ₂	N ₂		N ₂
Combustion/Reaction Gases			H ₂ Air	H ₂ Air	H ₂ Air	H ₂	
Make-up Gases	N ₂	N ₂	N ₂	N ₂	N ₂		N ₂
	He	ArCH ₄	He	He	He		He

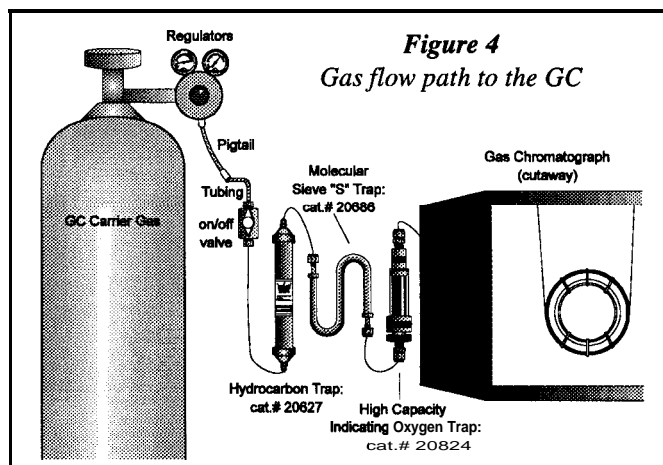
Recommended Gas Purities

Gas purity is very important. The expense of using high purity gases in combination with carrier gas purifiers will be offset by longer column lifetime and less GC maintenance. Carrier gas should contain less than 1ppm of oxygen, moisture, or other trace contaminants to prevent column degradation, increase column lifetime, and decrease stationary phase bleed. Carrier gas impurities can also contribute to detector noise. Figure 3 illustrates contamination on a sensitive ECD and shows how an impure carrier gas can affect detector performance. Contaminants such as trace hydrocarbons can be detected by an FID during a temperature programmed run, causing ghost peaks to appear. Make-up and fuel gases should be contaminant-free to reduce baseline fluctuations and excessive detector noise.



Plumbing Gases to the GC

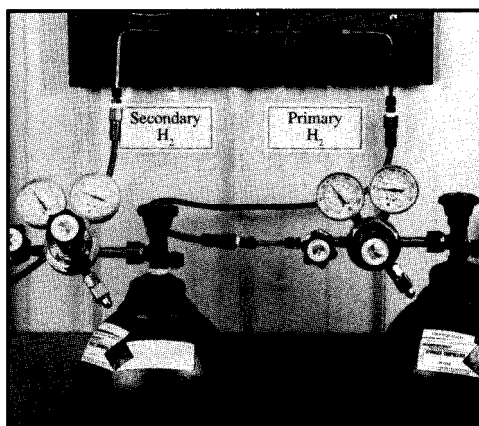
Once the proper carrier and detector gases are selected, they must be connected to the instrument. The gas flow path travels through regulators, pigtails, tubing, valves, and traps (Figure 4). Each component in the flow path will be discussed in sequence with helpful hints on their proper use.



Gas Sources



Gases are most often supplied to the instrument by gas cylinders. Begin installation at the cylinder and work towards the GC. Cylinders are under very high pressure and dropping one could result in an extremely dangerous situation. All cylinders (full or empty) should be securely chained to a wall or bench for safety. Any spare cylinders should also be chained to a wall in their storage location with the valve cap intact. It is advisable to have back-up cylinders on all gas lines to avoid any interruption of flow. This is particularly important for carrier gas. Should you lose carrier flow while the column is being heated, irreversible column damage may occur. Two-stage pressure regulators are used with gas cylinders to reduce the pressure of a gas supplied from a high pressure source to a desired working pressure. For safety reasons, when removing a regulator from a cylinder, always position yourself so that the regulator is pointing away from you.



It is common for a newly installed gas cylinder to shut down unpredictably (within the first 24 hours) if the main valve is not completely opened when it is installed. As the cylinder pressure decreases, the force against the valve seat decreases, allowing it to close. Always make sure cylinder valves are completely open when installing new tanks and completely closed before removing the regulator.

As a general rule, change a cylinder when the pressure regulator indicates that there is 200-300psi remaining in the cylinder. As the cylinder pressure drops, the concentration of impurities such as moisture and hydrocarbons increase. Therefore, column damage or premature purifier consumption will occur if you attempt to "save money" by using all the gas left in a cylinder. In addition, if the cylinder pressure drops below the supply pressure required by the GC, retention times and detector sensitivities can slowly change and affect the validity of your data.

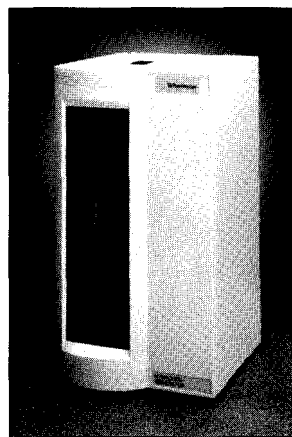
Hints for Proper Handling of Gas Cylinders

Observe safe laboratory practice in the transportation, storage, and usage of gas cylinders under high pressure:

- * Never move a cylinder with a regulator installed. Make sure safety caps are in place over the gas valve when transporting the cylinder.
- † Always chain or strap cylinders to stationary objects in the laboratory and while in storage
- * Always use cylinder condition labels to show whether tank is FULL, IN USE, or EMPTY.
- Always leave at least 200psi residual gas in a depleted cylinder. Store the empty tank in the storage area with the tank valve closed. Mark and date the empty cylinder.
- . Do not expose cylinders to temperatures above 125°F.

Gas Generators

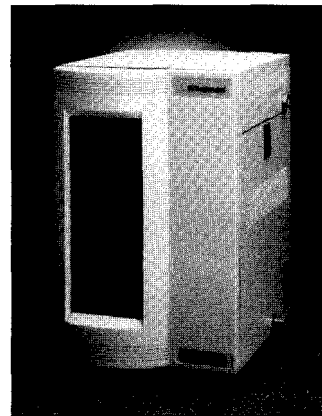
As an alternative to gas cylinders, many labs use gas generators. Generators reduce the costs and safety hazards involved with high pressure gas cylinders. Hydrogen generators supply hydrogen from the electrolysis of water. These units are convenient, safe to use, and produce very pure hydrogen. Air compressors can be used for air supply. However, most compressed air contains hydrocarbons from oil based lubricants. Compressed air that contains hydrocarbons or sulfur gases is not recommended for operating an FID, FPD, TSD, or ELCD. It is advisable to use filters and purifiers to remove hydrocarbon contamination from the compressed air source.



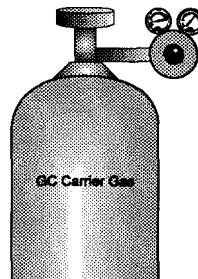
Hydrogen Generator:
(see page 23 for cat.%)



Nitrogen Generator:
(see page 24 for cat.#)



Zero Air Generator: (see page 24 for cat.#s)



Regulators

The purpose of a pressure regulator is to maintain constant gas pressure to the GC. Regulators may be classified as two types: cylinder regulators and line regulators. Cylinder regulators attach directly to the cylinder valve. The cylinder regulator reduces the gas pressure from the cylinder pressure (usually 2500psi for a new cylinder) down to a more usable pressure (around

100psi for gas chromatography). Cylinder regulators have two pressure gauges: an inlet, or high pressure gauge which reads the cylinder pressure; and a delivery, or outlet pressure gauge. This final delivery pressure is user adjustable by turning the large knob on the front of the regulator.

Cylinder regulators may be either single-stage or two-stage regulators. Two-stage regulators actually employ two regulators back-to-back in one housing. The first stage reduces the cylinder pressure to 200-600psi, while the second stage performs the final pressure reduction. Two-stage regulators are less prone to "creep" (a slow increase in delivery pressure as the tank empties) but have a lower flow capacity than single-stage regulators. Although more expensive, two-stage regulators should be used when a very constant delivery pressure is required, such as when controlling gas flows for a gas chromatograph.

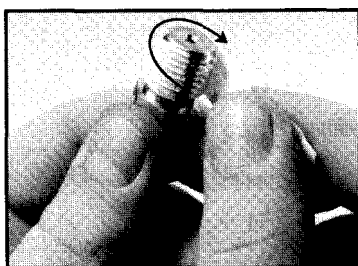
Line regulators have a lower allowable inlet pressure (typically 300psi) and must never be attached directly to a gas cylinder. Line regulators are used to further reduce the pressure of a gas from the supply line to that required at the point of use. Line regulators are always single-stage regulators, and may be equipped with a single pressure gauge to indicate outlet pressure. (See page 25 for regulators.)

Oxygen, moisture, and elastomeric contaminants can migrate through rubber or elastomeric diaphragms and enter the carrier gas. Therefore, all regulators should incorporate metal (Teflon® coated stainless steel) diaphragms to assure that contaminants will not enter the gas line. Consult Table II to determine the proper type and size of cylinder valves as described by the Compressed Gas Association (CGA) numbers for each regulator.

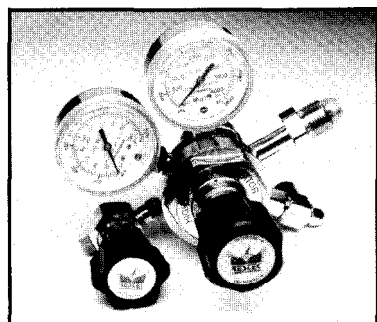
Table II - High Purity Two-Stage Regulators
Proper type and size of cylinder valves:

CGA 580	N ₂ , He & Ar	cat.# 20606
CGA 350	H ₂	cat.# 20607
CGA 590	Air	cat.# 20608

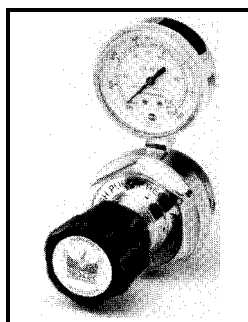
When installing any regulator, be sure to use high quality Teflon® tape on all machine thread connections, DO NOT use Teflon tape on Swagelok-type compression fittings.



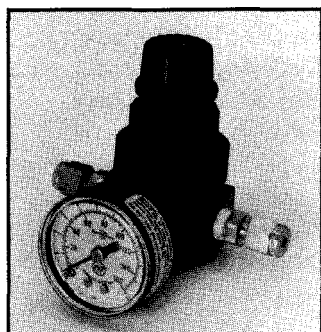
Wind Teflon tape clockwise to ensure a good seal.



High Purity Two-Stage Regulator (CGA 580):
cat.# 20606



High Purity Single-Stage Regulator:
cat.# 20609

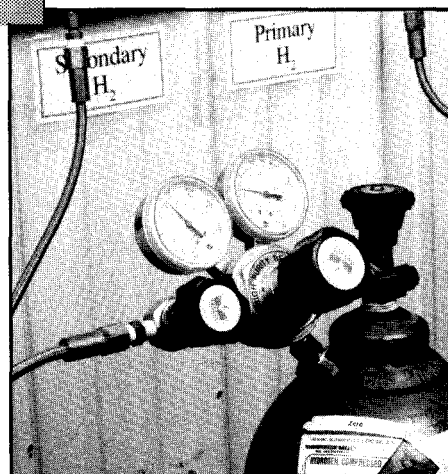


MINICYL General Purpose 1/8" Regulator: cat.# 20610

Flexible Pigtails



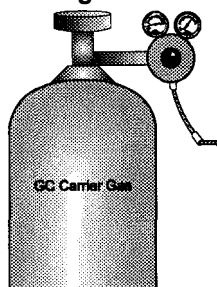
Pigtails (cat.# 20615) are commonly installed between the two-stage regulator and the gas lines. They allow the necessary flexibility in removing regulators from the cylinder. Pigtails are constructed of braided stainless steel with an inert Teflon core.*



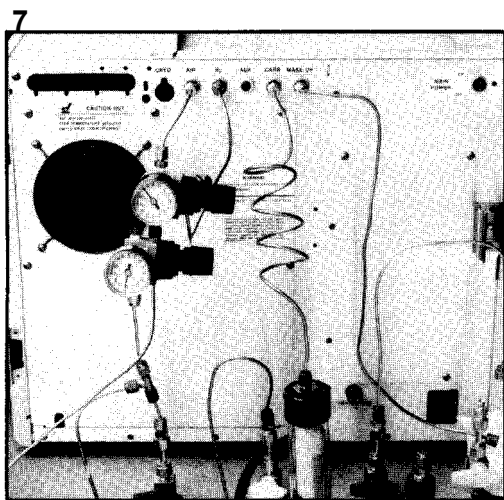
Flexible pigtails make it easier to change gas cylinders.

** Some O₂ and moisture will diffuse through the Teflon core stainless steel braided pigtails. Always use oxygen and moisture traps downstream of flexible pigtails.*

Tubing



The next step in plumbing GC gases to the instrument is choosing the appropriate tubing size. Most GCs are plumbed with 1/8" bulkhead fittings, therefore 1/8" tubing is commonly used. The location of the gas cylinders also determines if a larger diameter tubing is required between the tanks and the GC. If only one GC is being plumbed from tanks located in the same room, 1/8" tubing is sufficient. However, if the tanks are located outside the room or if several GCs are being plumbed from the same source, 1/4" tubing is recommended to reduce pressure drop in the line and supply adequate gas for several instruments. When using 1/4" tubing, plumb the GC with a 1/4" line from the tanks to back of the GC. Then use a 1/4 to 1/8" female reducer (cat.# 21825) to allow attachment to the first purifier. Connect both purifiers to the carrier gas line with 1/8" tubing loosely coiled. When measuring the 1/8" tubing, provide extra length to coil the tubing into shock loops. Shock loops will prevent instrument vibrations from being transferred to the supply lines and loosening fittings or breaking gas purification traps. Additionally, shock loops allow an instrument to be moved on the lab bench. Complete the installation by plumbing 1/8" tubing from the outlet of the last purifier to the GC bulkhead fitting.



Shock loops to prevent purifier breakage can be made by bending 1/8" tubing into a coil shape.

It is essential to use clean chromatographic grade tubing prior to installing a GC. Tubing can contain residual hydrocarbon contamination from the drawing process. These contaminants can migrate into the gas stream causing elevated background noise and increase instrument down time. Tubing can be solvent rinsed with methanol or other various solvents that do not provide a response on the detector being used. (Caution: do not use methylene chloride when using ECDs.) Restek offers a full line of pre-cleaned, heat treated tubing to plumb GCs without the need for solvent rinsing (see page 16). GC manufacturers recommend copper or stainless steel tubing for plumbing gas lines between the gas source and the instrument. Plastic tubing material such as Teflon, polyvinyl chloride, or Tygon should not be used when plumbing GCs since these materials will allow air and water to diffuse into the gas lines. In addition, plastic tubing can give off organic impurities which can cause ghost peaks and baseline instability.

Tubing Cutting and Bending

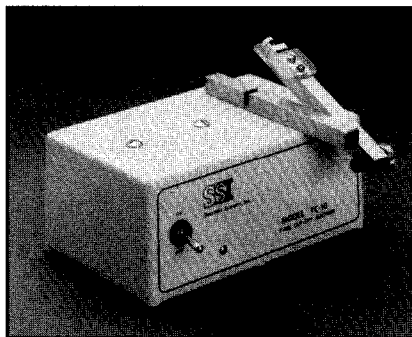
The first step toward leak free plumbing is correctly cutting and bending the tubing. Either a hand-held or a motorized tubing cutter can be used to cut tubing. With a hand-held device, the tubing is scored by guiding a cutting wheel along the outside surface of the tubing. By increasing the pressure, the cutting wheel is forced into the tubing, thereby making a cut. With a motorized cutter, the cutting wheel is driven by a high speed motor and the tubing is hand-fed onto the spinning wheel. The mechanical cutter*, though more expensive, will easily pay for itself when plumbing several instruments since it is faster and makes a clean, open cut.

* Catalog number 20186 is recommended for 1/16" and 1/8" tubing only.



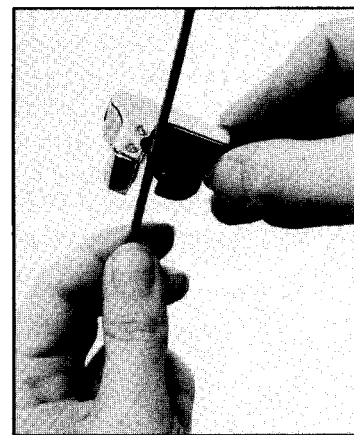
*To place an order, call
customer service or
your local distributor*

When using a tubing cutter, a burr or ridge will form on the tubing end. This burr must be removed to allow unobstructed gas flow and to obtain a leak-free connection with the compression fittings. Use a file or exterior deburring tool to remove the burr on the outside of the tubing and an interior deburring tool for the inside. Restek also offers a special tool that deburrs the inside and outside of tubing simultaneously (cat.# 20134 for 1/4" and 1/8" tubing, or cat.# 20188 for 1/16" tubing). Always hold the tubing open end down when deburring to prevent fragments from falling into the tubing.



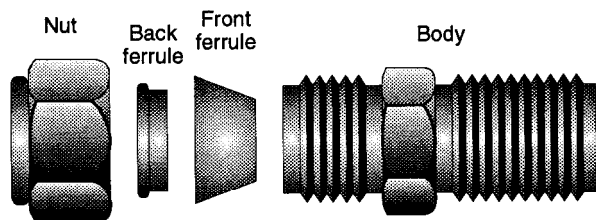
SSI Tubing Cutting Machine:
cat.# 20186

*Small tubing cutters
allow cuts to be made in tight
places (cat.# 20184)*



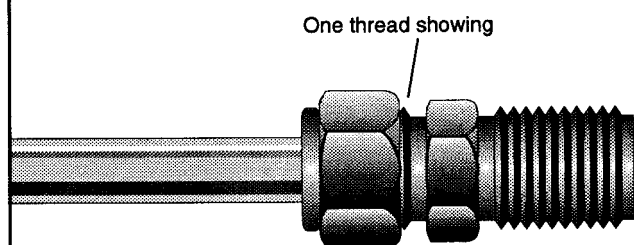
Tubing Benders

Tubing often requires bending during installation. Copper and thin walled 1/8" stainless steel tubing can easily be bent by hand. However, heavy walled 1/8 and 1/4" stainless steel tubing will require a tubing bender. A tubing bender incorporates the use of lever arms that reduce the force required to bend the tubing. Bends should be made with a uniform radius and should not kink or deform the tubing in a manner that obstructs flow. Try the bending procedure on some spare tubing first to help avoid costly mistakes on expensive tubing.

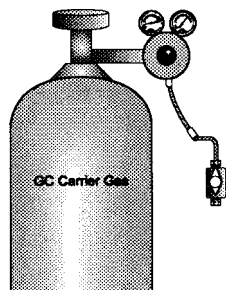
Figure 6Compression fitting **with two-piece ferrule design**

Fittings

Compression fittings provide gas-tight, leak-free connections without the use of Teflon tape or adhesives. A Swagelok-type compression fitting consists of a nut, a back ferrule, a front ferrule, and the fitting (union, elbow, tee, etc.) as shown in Figure 6. Slide the nut and ferrules onto clean, deburred tubing and insert the tubing into the fitting as far as possible. Hand tighten the nut, then use a wrench to tighten further. For $\frac{1}{8}$ " tubing, tighten the nut $\frac{3}{4}$ -turn past finger-tight. For $\frac{1}{4}$ " tubing, tighten the nut $1\frac{1}{2}$ turns past finger-tight. When tightened, the back ferrule forces itself into the front ferrule causing it to compress and grip the tubing forming a leak-free seal. Be careful not to over-tighten the nut, or the tubing and ferrules can become deformed and not seal. A properly tightened compression fitting usually shows one thread from the back of the nut (Figure 7). Overtightened fittings show no thread and are prone to leakage.

Figure 7 - A properly tightened compression fitting

Valves



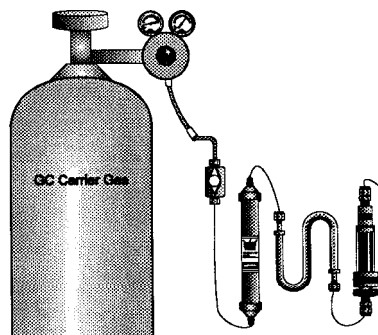
To expedite troubleshooting, the entire GC network should incorporate valving. $\frac{1}{8}$ " brass plug valves (cat# 21889) are recommended to isolate the system to check for leaks or to allow the GC to be taken off-line for repairs. Plug valves use a rotating cylinder to control gas flow in one direction only. Ball valves use a ball encased in Teflon packing and allow

flow in either direction. One drawback of ball valves is the potential for Teflon to flow form and cause the valve to leak when used under fluctuating temperature conditions.

Valves should be placed before gas purification traps to allow simple trap replacement without shutting down other GCs on the same line. For easy identification and troubleshooting, label or color code each valve throughout the system to help identify each gas type. After the system is pressurized, leak check valves in all possible positions using a thermal conductivity leak detector (cat.# 21607).

Caution: Two different gas types should never be connected together by a tee or a valve to allow easy change-over of carrier gases. Mixing will inevitably occur making troubleshooting very difficult.

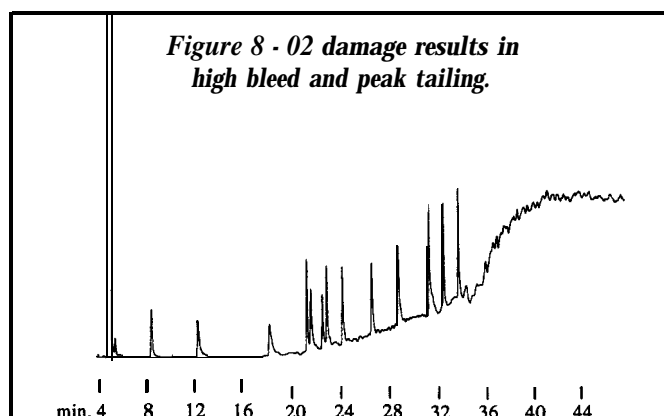
GC Gas Purification



Clean carrier gas is the key to longer column lifetime and less detector noise. Oxygen and moisture can enter the carrier gas cylinder through fitting leaks or connectors that utilize rubber o-rings. Also, contamination of the tubing with solvents or

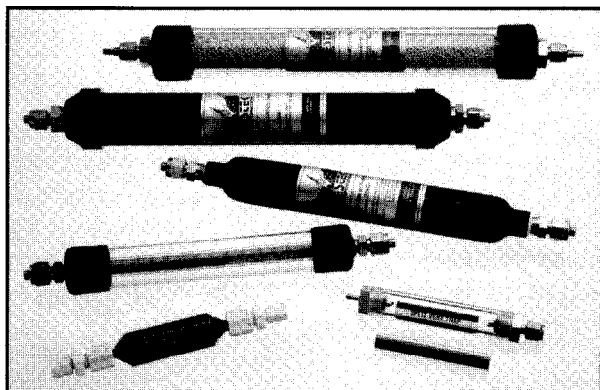
lubricating oils can increase background noise and cause ghost peaks with GC systems. Therefore, traps should always be used (even with ultra high purity gases) to prevent impurities from entering the GC system. Individual traps are designed to remove moisture, oxygen, hydrocarbons and other contaminants from the gas supply. Traps are available with either $\frac{1}{4}$ or $\frac{1}{8}$ " compression fittings and are typically constructed with metal or glass bodies. Plastic bodied traps should never be used since oxygen and moisture permeation will occur. Several common carrier, make-up, and detector gas purifiers are discussed in the following section.

The most common contaminants in carrier gas are oxygen, water, and hydrocarbons. Both oxygen and moisture degrade the stationary phase and shorten column lifetime. Hydrocarbons cause ghost peaks or increase detector noise. Oxygen contamination in carrier gas can produce excessive column bleed at high temperatures (Figure 8). Although some

Figure 8 - O₂ damage results in high bleed and peak tailing.

stationary⁹ phases are more resistant to oxidation (methyl and phenyl/methyl polysiloxanes), all stationary phases will eventually degrade when exposed to oxygen in the carrier gas at high temperatures.

Oxygen can be removed using getters, or materials that adsorb or chemically react with oxygen. Some getters must be heated to effectively remove oxygen, while others can be used at room temperature. Getters also differ in their capacity and mechanism to remove oxygen. Heated getters may release hydrogen or other impurities into the carrier gas stream, whereas most room temperature getters simply bind or react with oxygen. Some room temperature getters are extremely reactive when broken, therefore care must be taken not to break the trap or expose the trap material. Getters can also remove trace moisture but this diminishes their capacity to remove oxygen. Removing moisture with molecular sieve traps is more effective and will extend the lifetime for most getters. Molecular sieve traps exhibit excellent capacity for removing trace levels of moisture from carrier gas. Indicating molecular sieve traps are available, however, the indicating media is only sensitive to high levels of water and are not usually recommended. The 1/8" "S" type molecular sieve traps (cat# 20686) are usually the best choice for chromatographers. They are packed, activated at oven temperatures of 300°C sealed, and are ready to-use. Because of their small size, they can be reconditioned in a GC oven when contaminated.



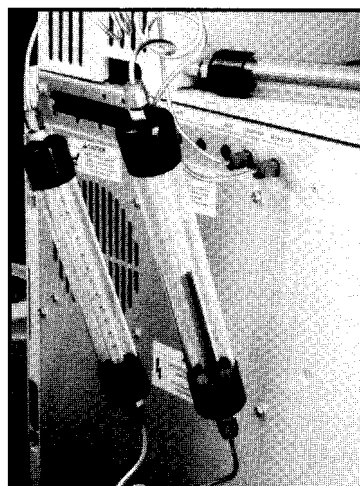
A wide assortment of traps are available for carrier, makeup, and detector fuel gases.

Hydrocarbon impurities in the carrier gas lines will result in detector instability, ghost peaks, and in extreme cases will result in column contamination. High levels of hydrocarbon impurities are not usually present in commercially available carrier gas sources, therefore most chromatographers do not find hydrocarbon traps necessary.

Hydrocarbon and solvent contamination is frequently removed using activated coconut charcoal. Since indicating hydrocarbon traps are not available for carrier gas lines, the analyst must note the date of installation and change the trap after approximately six months of use. Indicating traps, which are available from compressed air lines (1/8":cat.# 20637), should be used when oil lubricated air compressors are used as the FID air source.

What are the differences between indicating and non-indicating traps?

Some traps can indicate oxygen, moisture, or hydrocarbon removal by changing color. Indicating traps are made with glass housings to allow visual inspection of the color change. Although glass housings are fragile, they prevent oxygen from diffusing into the carrier gas and allow visual indication of the purifier activity level. Plastic materials are permeable to oxygen and are not recommended for any trap installed on a carrier gas line. Non-indicating traps are generally contained in a metal housing for strength and ruggedness. It is important that indicating oxygen traps are made with either glass or metal housings.



Indicating traps change colors as they are depleted, telling you when it is time to change them.

Indicating traps have an advantage over non-indicating traps since you can visually determine when to install a new trap. With non-indicating traps, it is impossible to accurately determine when the trap needs to be replaced. Non-indicating, high capacity traps should be installed prior to an indicating trap. When the indicating trap shows a color change, the non-indicating trap has been depleted and should be changed.

In what order should the traps be installed?

The order in which the traps are placed in the carrier gas flow path and their proximity to the GC is very important. Figure 9 on page 10 shows the recommended order for installing carrier gas traps. The hydrocarbon trap should be placed first in line from the carrier gas tank. This is to prevent trace hydrocarbons from contaminating the molecular sieve trap. The molecular sieve trap should be placed after the hydrocarbon trap to remove water. The oxygen trap should be placed closest to the GC bulkhead fitting. In general, traps should be installed on each GC as close to the bulkhead fitting as possible. Traps installed near the gas cylinder will not remove oxygen that may enter the carrier gas from leaky fittings downstream.

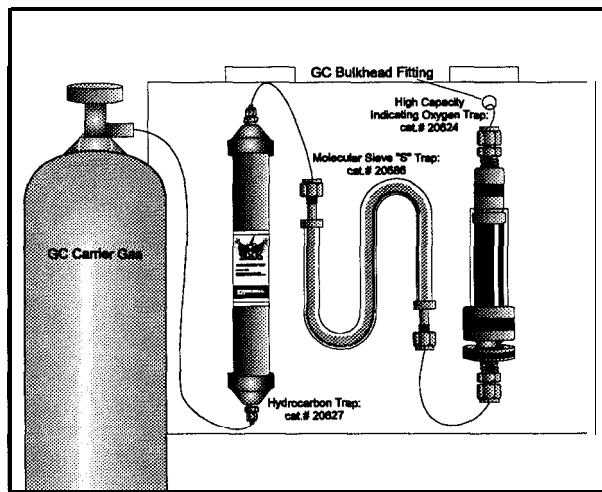
Traps should be installed vertically to avoid channeling. Channeling results from the packing material settling which, when a trap is positioned horizontally, may allow carrier gas to pass through without sufficient interaction with the packing.

Figure 9 - Trap recommendations, order, and installation tips.**Hints for Using Traps:**

- Install close to GC bulkhead fitting.
- Install vertically.
- If using a high capacity, non-indicating O₂ trap, use a low capacity indicating trap after it.

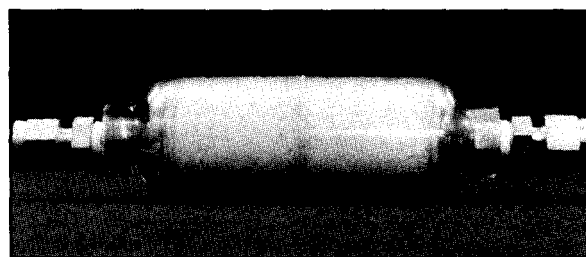
Trap Recommendations:**Carrier gas:** oxygen, moisture, and hydrocarbon (optional)**Make-up gas:** none required***Air (FID, etc.):** hydrocarbon (when trace oils are suspected)**H₂ (FID, etc.):** none required**ELCD reaction gas:** hydrocarbon

* Use oxygen and moisture traps on ECD make-up gas.

**Should purifiers be used for other gases?**

In addition to carrier gas, traps can also be used for other gases such as make-up and detector gases. Make-up gas for Flame Ionization Detectors (PID) does not require purification unless the PID is operated at high sensitivities. However, oxygen and moisture traps are highly recommended for make-up gas when operating sensitive detectors such as Electron Capture Detectors (ECD). The hydrogen reaction gas used for sensitive Electrolytic Conductivity Detectors (ELCD) also requires a hydrocarbon trap to remove trace impurities. These impurities can cause baseline instability and decrease the lifetime of the nickel reaction tube.

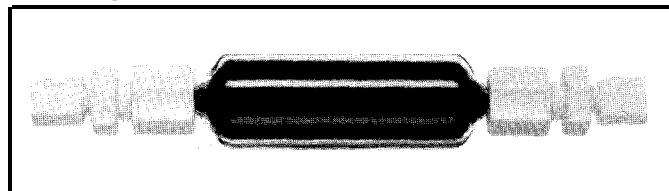
Many analysts use "house" compressed air from oil based compressors which can emit hydrocarbon vapors. Trace oil vapors can increase the background noise and contaminate PID detectors. A new compressed air trap (cat.# 20637) is available which reduces oil vapors to levels less than Sppm. This trap changes color as oil vapors are detected to indicate when the trap is depleted. Because of its plastic body, the

*Compressed air traps change colors to indicate oil contamination.*

indicating compressed air trap is not recommended for carrier gas lines.

After running the gas supply lines to the lab bench, it is time to plumb the gas supply lines to the GC. Special care should be taken to connect the correct gas type to the appropriate fitting on the GC. The gas inlets for the GC will be located in different places depending on the make and model of your instrument. Refer to your installation manual for the location of the gas inlets.

Hints for ECDs: Always use high purity moisture traps like our 1/8" "S" trap (cat.# 20686) and 1/8" high purity oxygen trap (cat.# 20624) on ECD carrier and make-up gas lines.

Traps are Necessary for Split Vents to Prevent Breathing Air Contamination

Potentially hazardous or carcinogenic chemicals can enter the lab atmosphere through the split vent in a capillary GC. As much as 99% of the sample injected vents to the air where chemists working nearby breathe these pollutants. This problem is further magnified when multiple GCs are used in the same lab. Split vent traps, packed with charcoal, reduce the uncontrolled release of hazardous materials into the lab.

The best trapping material is activated coconut charcoal due

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to its tenacious trapping ability. Narrow 1/4" trap bodies cause increased back pressure on the inlet system and severely increase retention times. In addition, the excessive back pressure on the split vent outlet can cause the back pressure regulator to perform erratically when the solvent expansion pulse occurs. Therefore, a large trap body design maximizes the quantity of charcoal that comes in contact with the sample vapor stream without causing unreasonable back pressure. Trap bodies made from solvent resistant plastics

either crack or leak with continuous solvent exposure. A glass trap body provides the best resistance and longevity from repeated solvent injections.

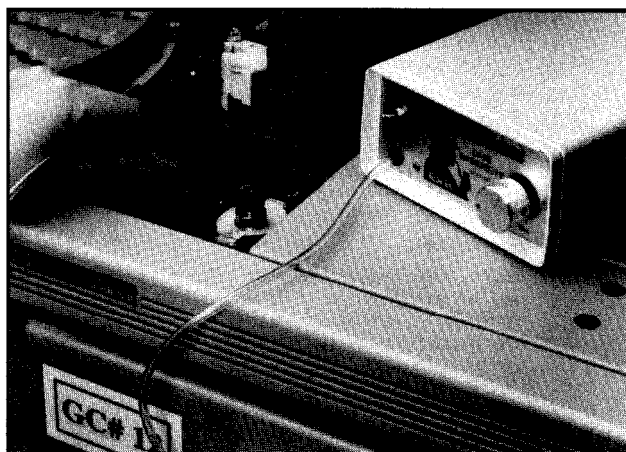
The trap capacity determines the number of injections that can be performed before solvent breakthrough occurs. High capacity traps provide protection for thirteen hundred injections or fifty days if one analysis is performed per hour.

Leak Checking

Many GC problems can be avoided by leak checking the system during the plumbing process. Loss of GC gases, reduced trap lifetime, damage to capillary columns, and increased detector maintenance will result if a leak is present. Leak checking the instrument before column installation and conditioning prevents column degradation indicated by high bleed and short lifetime. Irreversible damage can occur if a column is exposed to oxygen at high temperatures. Additionally, some detectors (for example, an ECD) are very sensitive to oxygen and can easily be damaged by oxygen exposure. Leak checking should be performed from the tanks to the GC, including all the fittings inside the GC. The GC fan should be turned off during leak checking. Next, check the external fittings along the carrier gas line for leaks. Leak detectors such as the Restek Leak Detective (110 volts: cat.# 21607, 220 volts: cat.# 21609), Gow-Mac Leak Detector (cat.# 20130), or Compact Leak Detector (cat.# 21605), detect minute traces of helium or hydrogen without contaminating the system. Never use liquid leak detectors that contain soap or surfactants. Liquids can be drawn inside the fitting at the site of a leak by the Venturi effect and contaminate the system.

If a thermal conductivity leak detector is not available, a pressure decay test can also be used to find major gas leaks.

To perform a pressure decay test, first cap off all possible gas outlets including the injection port and the detector fittings. Next, shut off the gas supply at the cylinder. In a leak free system, the line pressure observed at the two-stage regulator will hold constant for 15 minutes or longer. A rapid loss of pressure indicates that leaks are present. If this is the case, isolate smaller sections of the plumbing by capping off the line closer to the cylinder and recheck the pressure drop after closing off the gas supply. Repeat this process until the leak is found, then retest the entire system to ensure pressure is maintained.



Proper leak checks increase column lifetime/reduce detector noise.

Instrument Logbook

Another step in setting up a GC is creating an instrument logbook. Detailed documentation is crucial to the operation of any testing lab, therefore, record all of the steps involved in installing the new GC in your laboratory. Additionally, develop a GC maintenance schedule and document all maintenance performed (Figure 10 on page 12). A routine GC maintenance schedule will minimize system troubleshooting, increase sample throughput, and improve analytical accuracy.

A good GC maintenance program focuses on the inlet, capillary column, detector(s), oven calibration, traps and purifiers, and leak checking. GC documentation should also include analytical information (number of analytical sequences run, type and number of samples analyzed, appearance of sample) and any troubleshooting or repair work performed on the instrument. Documentation and routine maintenance will make future troubleshooting efforts less time consuming.

Figure 10 <i>Routine GC Maintenance Documentation</i>		GC1	GC2	GC3	GC4	GC5	GC6	GC7	GC8	GC9	GC10
GC and Column Documentation	Column Information Column installed Column catalog # Column serial # Date of installation Analysts initials GC# Ferrules used with column Carrier gas used Linear velocity Dead time Date column removed from GC Analysts initials Columns sealed with										
	Analytical Information # of standards run Type of standards run # of samples run Type of samples run Solvent sample standards were diluted with Sample concentration Injection size Septa used Inlet sleeve Packing materials? Injector temperature Injection mode Split vent flow Septum purge Splitless hold time										
Routine GC Maintenance	Injector Replace septa Replace sleeve Replace column ferrule Replace injector fitting Replace inlet seal Replace o-ring Clean needle guide Clean needle disk Clean inlet seal Clean injection port Clean septum nut Leak check										
	Detector Replace detector sleeve Replace detector fitting Replace detector ferrules Clean detector Clean detector port Clean collector Leak check										
	Instrument Replace GC traps Replace chemical filters Leak check ah fittings Check gas flow rates Check make-up gas flows										
	Misc. Sweep oven Special problems										

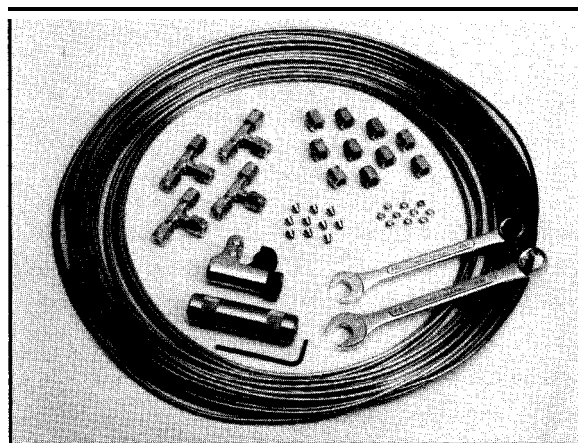
Products Used for GC Setup

GC Installation Kit

This kit contains the necessary tubing and fittings to add an additional GC to your lab bench. Also included in the kit are four 1/8" tees, so GC gases like hydrogen, helium, nitrogen, and air can be routed to the new inlet or detector from the existing gas lines. Additional parts, such as purifiers or regulators, may be ordered separately to customize the GC installation to your lab's specifications.

Kit includes: one tubing cutter, one 1/8" x 1/4" reamer, one 7/16" wrench, one 1/2" wrench, four 1/8" brass tees, ten 1/8" brass nuts, ten brass front and back ferrules, and 50' of pre-cleaned 1/8" copper tubing.

GC Installation Kit: cat.# 21325



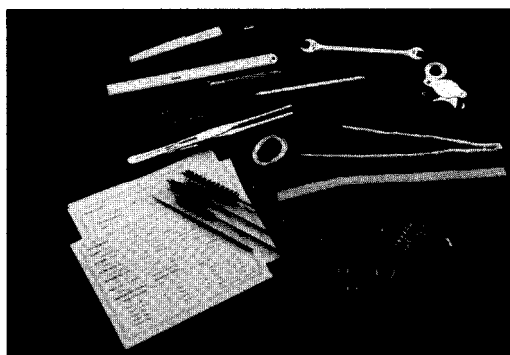
MLE Tool Kit

This kit contains all the tools necessary for installing and maintaining your capillary columns.

The MLE Tool Kit Includes:

- Rubber-tipped Slide-Lok tweezers
- 15cm compact steel ruler
- Sapphire scribe
- Pocket magnifier
- Pin vise with three drills (0.41, 0.5, 0.8mm)
- Four-inch tapered needle file
- Six stainless steel jet reamers (0.25-0.65mm)
- Five self-adhesive capillary column labels
- Septum puller
- Three nylon brushes (1/8", 3/16", 1/4")
- Pipe cleaner (one-foot)
- 1/4-3/16" wrench
- One-meter of high temperature string (400C)

- Three stainless steel brushes (3/16", 1/4", 3/8")
- Stainless steel toothbrush
- Glass wool puller/insertor
- Capillary column installation guide
- Chromatography Essentials Wall Chart

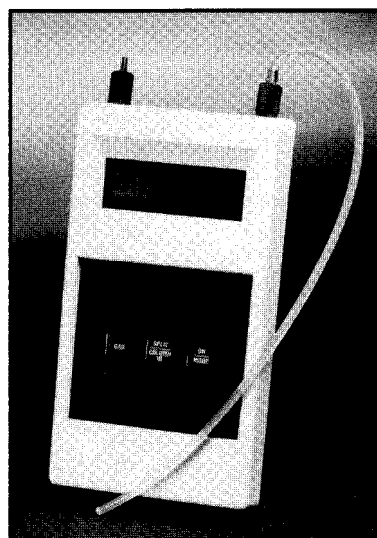


MLE Tool Kit:
cat.# 20118

Electronic Flow Calibrator

- NIST traceable calibration
- Displays flow, linear velocity, or split ratio
- Measures 0.1 to 500ml/min.
- Works with hydrogen, helium, nitrogen, air, or argon/methane
- Battery operated for easy portability

The Electronic Flow Calibrator can be used to measure any of the common gases used with GCs. Each flow meter is calibrated using NIST traceable standards. Flows from 0.1 to 500cc/min. can be accurately measured and easily switched between gases. The Electronic Flow Calibrator measures mass flow that is independent of changes in ambient temperature and pressure which can affect soap film or acoustic sensor meters.



Electronic Flow Calibrator: cat # 21606

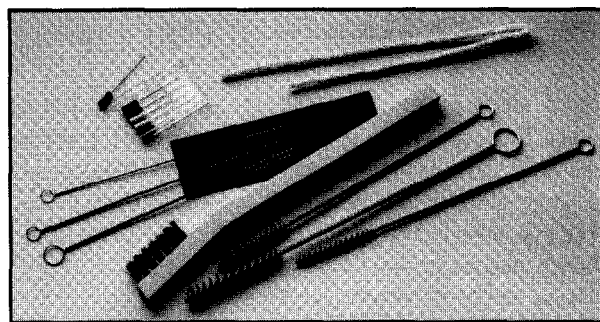
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FID/Injector Cleaning Kit

Kit includes everything needed to keep your FID and injection ports clean.

- Nylon tube brushes (1/8", 3/16", 1/4") pipe cleaners
- Stainless steel tube brushes (1/8", 3/16", 1/4")
- Stainless steel surface brush
- Stainless steel jet reamers
- Emery cloth

FID Injector Cleaning Kit: cat.# 20120

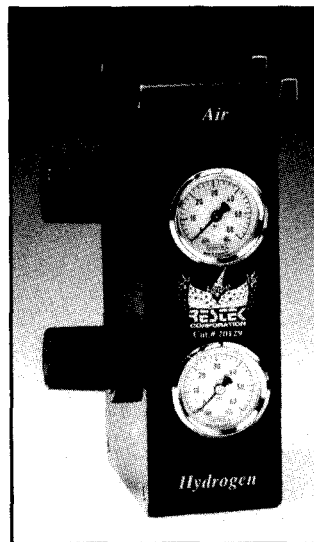


FID Gauge Pack

- Conveniently mounts **on the** side of the HP 5890 GC.
- Standard 1/4" inlet and outlet fittings.
- Up to 250psi inlet pressures, 0-60psi outlet pressures.
- Front mounted gauges for easy visibility.
- Side mounted regulator knobs for easy control.

HP 5890 GCs do not include the pressure regulators necessary to adjust the air and hydrogen flows to the Flame Ionization Detector (FID). Since most labs operate their gas supply lines at a higher pressure than necessary for the proper operation of the FID, chromatographers must supply their own regulators to adjust flows for optimum detector performance.

Restek's FID Gauge Pack simplifies GC installation by incorporating pressure regulators and gauges for both air and hydrogen in a single enclosure. The unit conveniently mounts on the side of the GC, and 1/4" bulkhead fittings allow convenient hook-up to instrument and supply lines.



FID Gauge Pack:
cat.# 20129

*Control HP 5890 FID Gases
with Restek's Gauge Pack.*

Make-up Gas Kits

Universal 1/4" & 1/8" Make-up Gas Kits*

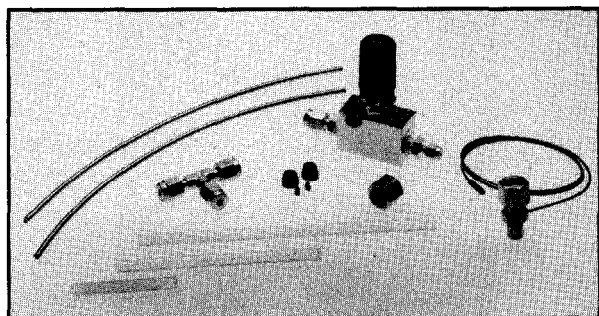
Each Kit Includes:

- Custom stainless steel make-up gas fittings.
- A high performance variable flow restrictor.
- Standard 1/8" tee and 2 x 9" sections of 1/8" stainless steel tubing for connection to the GC bulkhead fitting.
- Three different sleeve lengths; 2" (51mm), 4" (102mm), and 6" (153mm). The 1/4" kit contains 1/4" OD by 1mm ID

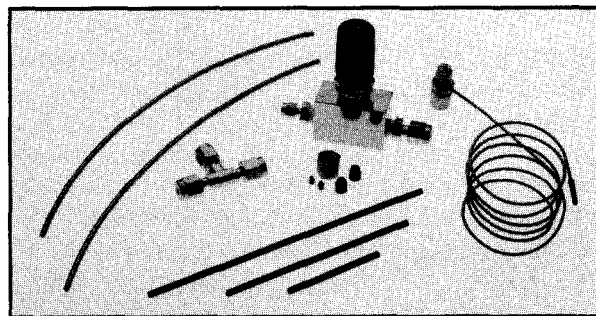
** The ECD/FID Dual Purpose Make-up Gas Fitting is recommended for use with HP GCs.*

deactivated glass sleeves with one end slotted and one end chamfered. The 1/8" kit contains 1/8" 2mm ID fused silica lined Silcosteel sleeves.

- All nuts and ferrules necessary for connecting the sleeve to the detector and the column to the make-up gas fitting.
- Complete step-by-step instructions.



1/4" Make-up Gas Kit: cat.# 20325

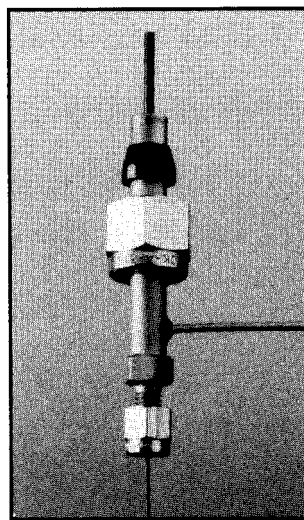


1/8" Make-up Gas Kit: cat.# 20326

ECD/FID Dual Purpose Make-up Gas Kit for HP 5890 GCs

This ECD make-up gas fitting uses a straight section of inert SilcosteelP tubing as the capillary column guide to replace the fused silica column guide found in the original equipment design. To use the fitting with an FID, remove the Silcosteel guide. A replacement fitting with a special end connector is also available for GCs that are factory equipped with make-up gas.

The replacement fitting includes a 1/4" nut and graphite ferrule and a 1/16" nut with a 0.4mm ID graphite capillary ferrule. The complete kit (for GCs not factory equipped with make-up gas) includes a make-up gas fitting, 1/4" nut and ferrule, 1/16" nut and a 0.4mm ID graphite capillary ferrule, and a high quality fine metering valve.



Make-up gas kit:

cat.# 21300

Replacement fitting:

cat.# 21301

Silcosteel guide:

cat.# 21302

Ferrules

Restek offers graphite and Vespel/graphite (60%/40%) ferrules to use with capillary columns. The graphite ferrules are made from highly-compressed ribbon that will not crack or split under torque. The Vespel/graphite ferrules are designed to seal easily with minimal torque, are reusable, and are preferred for vacuum and high pressure uses. Both ferrule types are stable to 400°C.

Capillary Ferrules for 1/16" Swagelok-type fittings			
Ferrule ID	Fits column ID	Graphite 10-pack cat.#	Vespel/graphite 10-pack cat.#
0.3mm	0.20mm	20233	—
0.4mm	0.25mm	20200	20211
0.5mm	0.32mm	20201	20212
0.6mm	0.28mm	—	20232
0.8mm	0.53mm	20202	20213
Ferrule ID	Fits column ID	Graphite 50-pack cat.#	Vespel/graphite 50-pack cat.#
0.4mm	0.25mm	20221	20229
0.5mm	0.32mm	20228	20231
0.8mm	0.53mm	20224	20230
Compact Ferrules for HP GCs (for capillary injection ports)			
Ferrule ID	Fits column ID	Graphite 10-pack cat.#	Graphite 50-pack cat.#
.4/.5mm	0.25-0.32mm	20250	20251
0.8mm	0.53mm	20252	20253
Standard Ferrules (for 1/16", 1/8", and 1/4" fittings)			
Fitting Size	Ferrule ID		Vespel/graphite S-pack cat.#
1/4"	3/16"		20258
Fitting Size	Ferrule ID	Graphite 10-pack cat.#	Vespel/graphite 10-pack cat.#
1/16"	1/16"	20207	20218
1/8"	1/8"	20208	20219
1/8"	rd. to 1/16"	20209	20220
1/4"	1/4"	20210	20221
1/4"	rd. to W	20225	20222
W	rd. to 1/16"	20226	20223

Two-hole Ferrules (for 1/16" Swagelok-type fittings)			
Ferrule ID	Fits column ID	Graphite 5-pack cat.#	Vespel/graphite 5-pack cat.#
0.4mm	0.25mm	20235	20241
0.5mm	0.32mm	20235	20242
Two-hole Ferrules (for 1/8" Swagelok-type fittings)			
Ferrule ID	Fits column ID	Graphite 5-pack cat.#	Vespel/graphite 5-pack cat.#
0.8mm	0.53mm	20245	20246
Reducing Ferrules			
Fitting Size	Fits column ID	Graphite 5-pack cat.#	Vespel/graphite 5-pack cat.#
1/8"	0.25mm	20205	20254
1/8"	0.32mm	20205	20255
1/8"	0.53mm	20206	20215
1/4"	0.25mm	20203	20256
1/4"	0.32mm	20203	20257
1/4"	0.53mm	20204	20217
Blank Ferrules (for 1/16" capillary fittings)			
Fitting Size	Ferrule ID	Vespel/graphite 10-pack cat.#	
1/16"	no hole	20240	

Buy ferrules in bulk 50-packs and SAVE!

Instrument Grade Stainless Steel Tubing

Heat-Treated Stainless Steel Tubing

- Solvent cleaned and heat-treated to remove volatile impurities.
- Ideal for sensitive GC detectors such as ECDs, MSDs, NPDs, and ELCDs that require pure carrier gas.
- Perfect for concentrating inlet systems such as purge and trap or thermal desorption units.
- Available in continuous lengths to minimize potential leaky connectors.
- Economically priced..

Heat-treated to eliminate contaminants

Most stainless steel tubing contains hydrocarbon impurities that migrate into the carrier gas stream and cause background noise or ghost peaks. Solvent cleaned tubing can still contain contaminants that were either insoluble in the cleaning solvent, or left over as a residue from the cleaning process. Restek's chemists have found that gradient solvent rinsing, used in combination with a high temperature treatment under a flow of clean carrier gas, is the best way to guarantee ultra high purity carrier gas lines. Background contamination is eliminated and new instruments can be plumbed with confidence.

Available in longer lengths

Tight manufacturing tolerances ensure close inside and outside dimensions. The 304 stainless steel tubing is annealed for added flexibility. Continuous lengths up to 500 feet are available to eliminate the need for connectors.*



Plumb your instrument with the best. Try Restek's heat-treated, solvent cleaned, instrument grade tubing.

Top quality heat-treated stainless steel tubing priced conservatively

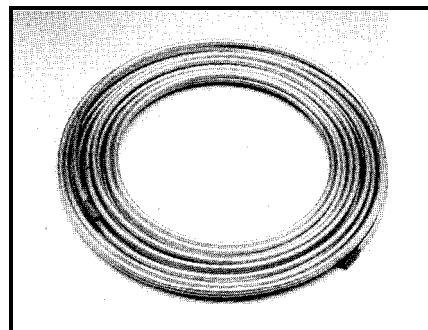
Stainless Steel Tubing Dimensions	25 ft. cat.#	26-100 ft. cat.#	>100 ft. cat.#
0.01" ID, 1/16"OD	21500	21501	21502
0.02" ID, 1/16"OD	21503	21504	21505
0.03" ID, 1/16"OD	21506	21507	21508
0.04" ID, 1/16"OD	21509	21510	21511
0.085" ID 1/8" OD	21512	21513	21514
0.21" ID, 1/4"OD	21515	21516	21517

* Please inquire before ordering to determine the availability of continuous lengths up to 500 feet. The availability of long lengths is subject to inventory constraints.

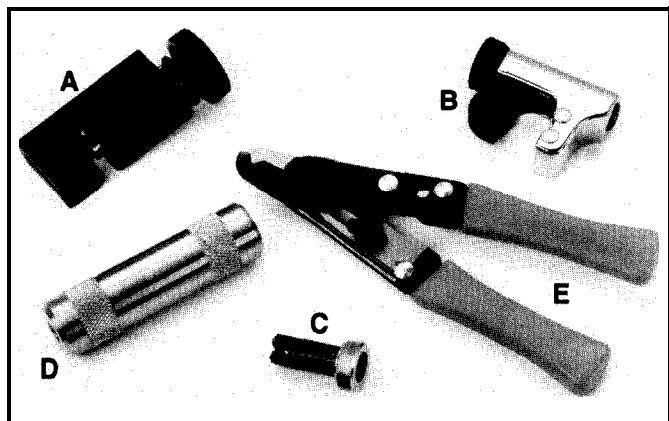
Pre-Cleaned Copper Tubing

cat&	OD	wall	ID	length
21590	1/8"	0.030"	0.065"	50 ft.
21592	1/4"	0.030"	0.190"	50 ft.

- Adheres to ASTM B-280.
- Specially cleaned to be chromatographically free of background contamination.
- Use for plumbing GC systems.



Tubing Cutters



A) Restek 1/16" Tubing Cutter

- Produces square, even cuts for 1/16" tubing.
- Eliminates distortion of the tubing.
- Replaceable cutting wheel.

Restek 1/16" Tubing Cutter: cat.# 20192

Replacement Wheels: cat.# 20185, 3-pack

B) Imp™ Tubing Cutter

- Excellent for cutting 1/8" or 1/4" metal tubing
- Compact size is ideal for tight spaces.
- Replaceable cutting wheel.

Imp™ Tubing Cutter: cat.# 20184

Replacement Wheels: cat.# 20185, 3-pack

C) Tubing Cutter Dressing Tool

Designed to work separately or with the SSI tube cutting machine, these dressing tools do a magnificent job at opening the column bore and removing burrs from the column exterior. Several twists and the bore is fully open.

1/16" **Dressing Tool: cat.# 20188**

1/16" **Replacement Insert: cat.# 20189**

1/8" **Dressing Tool: cat.# 20190**

1/8" **Replacement Insert: cat.# 20191**

D) Restek Tubing Reamer

This combination 1/4" and 1/8" tubing reamer incorporates a non-slip safety design and is excellent for deburring stainless steel tubing.

Tubing Reamer: cat.# 20134

E) Cutting Pliers

- Ideal for cutting 1/16" tubing.
- Cuts quickly, reducing distortion of the bore.
- Clean cuts eliminate need for deburring.

Cutting Pliers: cat.# 20193

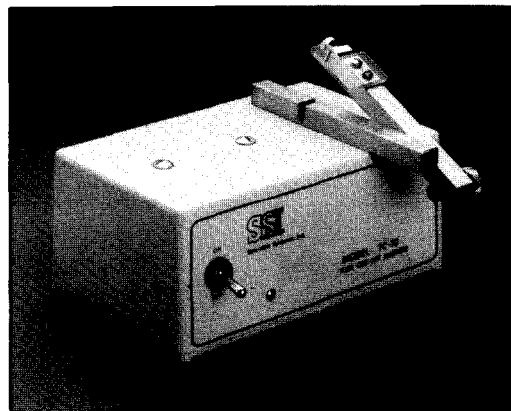
SSI Tube Cutting Machine

- Works for 1/8" and 1/16" tubing.
- Electrically operated bench-top model.

This high speed tube cutting machine squarely cuts either 1/8" or 1/16" tubing (with inner diameters as low as 0.010"). The tubing is held in a specially designed clamp at the end of a safety swing arm. As the arm is lowered, the tubing is guided through a narrow slot where it is cut by a dry abrasive cutting wheel. A dressing tool on the swing arm is provided to deburr and ream the inside and outside of the tubing.






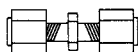
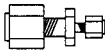
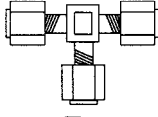
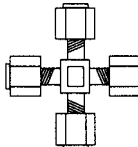
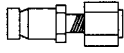
SSI Tube Cutting Machine: cat.# 20186

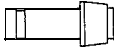
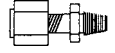
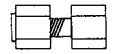
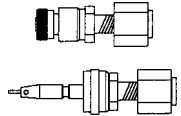
Replacement Wheels: cat.# 20187, 3-pack



Tube & Pipe Fittings

Restek offers an extensive line of brass and stainless steel tube and pipe fittings to help you install and maintain your gas chromatography equipment. Parker's (A-Lok) two-piece ferrule design, along with NPT fittings, are ideal for installing new equipment, modifying existing instrumentation, or replacing worn connections. Quick couplings are also available to connect and disconnect gas lines quickly and easily. If you frequently relocate instrumentation, switch carrier gases, or change make-up gases for different detection systems, these quick couplings are a worthwhile addition to your system.




Fitting Type	Size (inches)	Brass cat.#	Brass quant.	Stainless Steel cat.#	Stainless Steel quant.	Replaces Parker #	Replaces Swagelok #
 Nut	1/16	21800	20	21900	5	1NU1	102-1
	1/8	21801	40	21901	10	2NU2	202-1
	1/4	21802	40	21902	10	4NU4	402-1
 Front Ferrule	1/16	21803	20	21903	10	1FF1	103-1
	1/8	21804	40	21904	20	2FF2	203-1
	1/4	21805	40	21905	20	4FF4	403-1
 Back Ferrule	1/16	21806	20	21906	10	1BF1	101-1
	1/8	21807	40	21907	20	2BF2	204-1
	1/4	21808	40	21908	20	4BF4	404-1
 Nut & Ferrule Set	1/16	21809	10	21909	2	---	---
	1/8	21810	20	21910	5	---	---
	1/4	21811	20	21911	5	---	---
 Plug	1/16	21815	5	21915	2	1BLP1	100-P
	1/8	21816	10	21916	4	2BLP2	200-P
	1/4	21817	10	21917	4	4BLP4	400-P
 Union	1/16-1/16	21818	3	21918	1	1SC1	100-6
	1/8-1/8	21819	5	21919	2	2SC2	200-6
	1/4-1/4	21820	5	21920	2	4SC4	400-6
 Reducing Union	1/8-1/16	21823	5	21923	1	2RU1	200-6-1
	1/4-1/16	21824	5	21924	2	4RU1	400-6-1
	1/4-1/8	21825	5	21925	2	4RU2	400-6-2
 Tee	1/16	21826	2	21926	1	1ET1	100-3
	1/8	21827	2	21927	1	2ET2	200-3
	1/4	21828	2	21928	1	4ET4	400-3
 Cross	1/8	21829	2	21929	1	2ECR2	200-4
	1/4	21830	2	21930	1	4ECR4	400-4
 Tube to Compression Reducer	1/8-1/16	21831	5	21931	2	2TUR1	100-R-2
	1/4-1/16	21832	5	21932	2	4TUR1	100-R-4
	1/8-1/4	21833	5	21933	2	2TUR4	400-R-2
	1/4-1/8	21834	5	21934	2	4TUR2	200-R-4

Fitting Type	Size	Brass cat.#	Brass quant.	Stainless cat.#	Steel quant.	Replaces Parker #	Replaces Swagelok #
 Port Connector	1/8	21835	5	21935	2	2PC2	201-PC
	1/4	21836	10	21936	2	4PC4	401-PC
	1/8-1/4	21837	5	21937	2	2PC4	401-PC-2
 Male NPT Connector	1/8-1/8 NPT	21841	10	21941	2	2 MSC 2N	200-1-2
	1/4-1/4 NPT	21842	10	21942	2	4 MSC 4N	400-1-4
	1/16-1/8 NPT	21843	5	21943	2	1 MSC 2N	100-1-2
	1/8-1/4 NPT	21844	10	21944	2	2 MSC 4N	200-1-4
	1/4-1/8 NPT	21845	10	21945	2	4 MSC 2N	400-1-2
 Female NPT Connector	1/8-1/8 NPT	21846	5	21946	2	2 FSC 2N	200-7-2
	1/4-1/4 NPT	21847	5	21947	2	4 FSC 4N	400-7-4
	1/4-1/8 NPT	21848	5	21948	2	4 FSC 2N	400-7-2
 Quick Couplings	1/8 male*	21857	1	21957	1	2A-Q4VN	QC4D-200
	1/8 male	21858	1	21958	1	2A-Q4P	QC4S-200
	1/8 female*	21859	1	21959	1	2A-Q4CN	QC4B-200
	1/4 male*	21860	1	21960	1	4A-Q4VN	QC4D-400
	1/4 male	21861	1	21961	1	4A-Q4P	QC4S-400
	1/4 female*	21862	1	21962	1	4A-Q4CN	QC4B-400

* Includes self-sealing shut off valve.

Shut-off Valves

Hoke Toggle, Ball, and Plug Valves

Valve type	Brass		Stainless Steel	
	1/8"	1/4"	1/8	1/4"
 Toggle	21885	21886	21985	21986
 Ball	21887	21888	21987	21988
 Plug	21889	21890	21989	21990

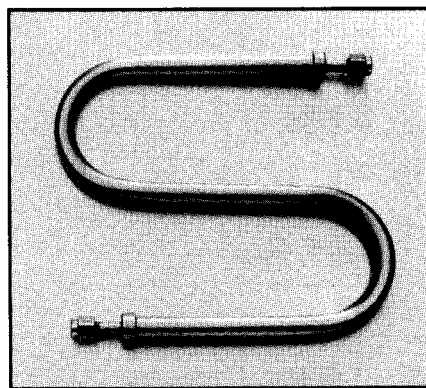
Restek offers Hoke toggle, ball, and plug valves. These high quality, precision machined valves are available in 1/8" and 1/4" sizes in both brass and stainless steel. The toggle valves are ideal for applications when instant on/off control is necessary. Hoke toggle valves are rated to 200psig at 21°C and have a maximum operating temperature of 149°C. The forged body ball valves are ideal for high pressure applications. Hoke ball valves have a floating ball to assist in sealing and reduce operating torque and dual seats provide leak-tight bidirectional sealing. They are rated to 6000p at 21C and have a maximum operating temperature of 249°C. The plug valves are ideal for applications where throttling or on/off operations are required. Hoke plug valves have dual retaining rings to prevent accidental plug removal. They are rated to 3000psig at 21°C and have a maximum operating temperature of 205°C.

Purifiers/Traps

Rechargeable Molecular Sieve "S" Trap

- Increases column and O₂ trap lifetime.
- Reduces baseline noise for sensitive detectors such as ECDs and mass spectrometers.

While glass indicating moisture traps offer convenience, they are fragile and add to waste disposal problems since they are discarded when spent. Restek's rechargeable molecular sieve moisture trap offers the best alternative. The "S" design allows the trap to be regenerated in the GC oven. The metal body and single end fitting design eliminate the possibility of leakage or breakage and the built-in 40 micron frit prevents particulate contamination from damaging regulators or needle valves. Each trap is individually activated to insure maximum reactivity for removing moisture.

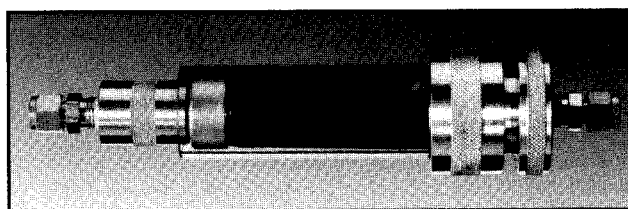


1/4" female Swagelok-type fitting: cat.# 20685

1/8" female Swagelok-type fitting: cat.# 20686

High Capacity Indicating Oxygen Trap

- Indicator changes color as O₂ & H₂O are trapped.
- Heavy duty body design virtually eliminates breakage.
- High capacity (>300s.c.f.) design lasts longer than three smaller traps.
- Economic replacement cartridges change easily.
- Usable with multiple GC systems.
- Removes impurities at flow rates up to 230s.c.f.
- Removes O₂, H₂O, and trace contaminants.
- Usable with all carrier gases.
- Ambient operating temperature, 100psig operating pressure.
- Built-in microparticulate frit.
- Discharge Gas Purity:
O₂ < 0.1ppm where inlet does not exceed 15ppm.
H₂O < 0.5ppm where inlet does not exceed 10 ppm.



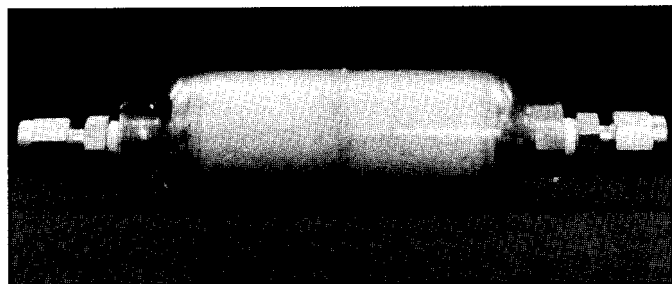
Includes cartridge housing and one cartridge.

1/4" tube compression fitting: cat.# 20623

1/8" tube compression fitting: cat.# 20624

Refill cartridge (fits 1/4 or 1/8" cat.# 20625

Indicating Hydrocarbon Trap for Compressed Air



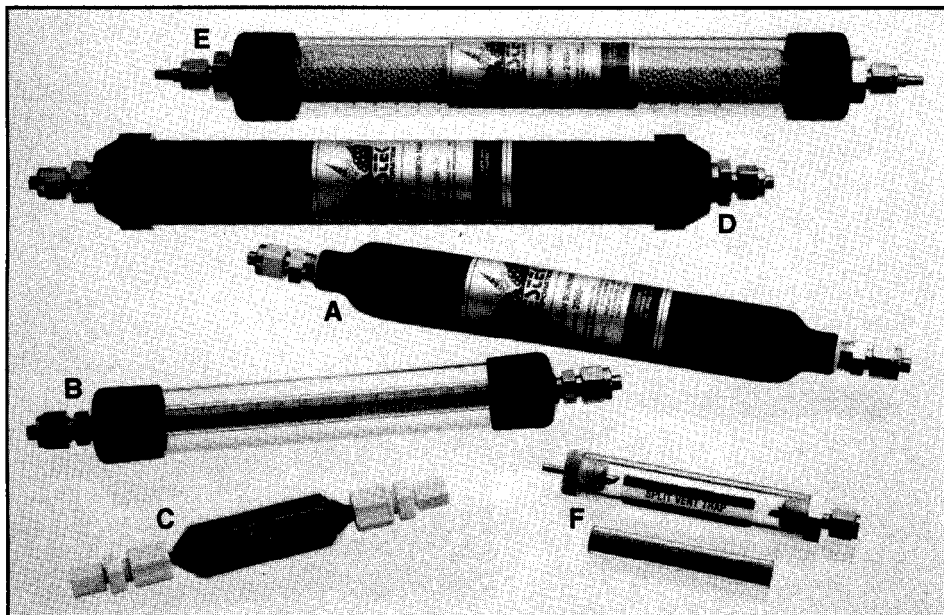
Many laboratories now run Flame Ionization Detectors (FID) from oil lubricated air compressors or from house air lines. Although most of these systems have extensive filtration devices to remove oil vapors or mist from the air stream, there is no way to determine if these filters are doing their job or when to change them. The only indication that the filters are not working is when oil contamination reaches the detector and creates massive baseline disturbances, ghost

peaks, and clogged jets. By then, it is too late and only laborious solvent rinsing of the gas lines and detector fittings will restore the stability of the FID.

The Restek Wizards have come up with a novel solution to this problem. Our Indicating Hydrocarbon Trap changes color as it absorbs oil vapors. This trap gives advanced warning should the filters on the compressed air lines ever fail. In addition to changing color, the trap reduces oil vapor concentration down to five parts per million to prevent gross contamination of the instrument lines. Available with 1/8" or 1/4" tube compression fittings, these traps are a must for any lab using oil lubricated air compressors as a gas source for their FIDs.

1/4" Indicating Hydrocarbon Trap: cat.# 20636

1/8" Indicating Hydrocarbon Trap: cat.# 20637



A) High Capacity Oxygen Trap

- Most effective oxygen trap available.
 - Long life, typically lasts for over five 200cf cylinders.
 - Traps carrier gas contaminating moisture.
- 1/4" female Swagelok Type fitting: cat.# 20690
1/8" female Swagelok Type fitting: cat.# 20601

B) Indicating Oxygen Trap

- Reduces both O₂ & H₂O to less than 15ppb.
 - Indicator changes color as O₂ & H₂O are trapped.
 - Quickly identifies contaminated gas cylinders or carrier gas leaks before column damage occurs.
 - Heavy-walled glass body prevents O₂ & H₂O diffusion.
 - Integral protective plastic shield for maximum safety.
 - 40µm frit prevents microparticulate damage to needle valves and flow controllers.
 - Pre-purged with helium for fast stabilization time.
 - Vespel sealing rings used.
 - 100psig maximum operating pressure.
- 1/4" female Swagelok-type fitting: cat.# 20603
1/8" female Swagelok-type fitting: cat.# 20602

Return unbroken traps to us and give them a new life!

Regenerated Trap 1/4" fitting: cat.# 20617

Regenerated Trap 1/8" fitting: cat.# 20616

C) High Capacity Split Vent Trap

The use of split vent traps packed with activated coconut charcoal reduces the release of hazardous materials in our breathing space. This large bodied trap allows 1,500 injections to be performed before solvent breakthrough occurs. Replace the trap once a month or after 1,500 injections. Includes connecting lines and mounting kit.
cat# 20698
cal# 20699, 5pack

D) Hydrocarbon Trap

- Removes trace carrier gas impurities for sensitive detectors such as ECDs, PIDs, & MS.
 - 20µm frit prevents particulate contamination.
 - Contains fine mesh coconut shell activated charcoal.
 - Stops carrier gas interferences with purge & trap systems.
 - Refillable and rechargeable.
- 1/4" tube compression fitting: cat.# 20628
1/8" tube compression fitting: cat.# 20627
Carbon refill (two recharges): cat.# 20626

E) Indicating Moisture Trap

- Reduces both O₂ & H₂O to less than 15ppb.
 - Reduces noise for high sensitivity detectors.
 - Indicator changes color as H₂O is trapped.
 - Heavy-walled glass body prevents O₂ & H₂O infusion.
 - Integral protective plastic shield for maximum safety.
 - 40µm frit prevents microparticulate damage to needle valves and flow controllers.
 - Pre-purged with helium for fast stabilization time.
- 1/4" female Swagelok-type fitting: cat.# 20604
1/8" female Swagelok-type fitting: cat.# 20605

F) Split Vent Trap Kit

Stop polluting the environment. Install a trap to the split/splitless vent port outlet. The easy to install trap incorporates a male and female 1/8" fitting to accommodate most GCs. Special impregnated high capacity carbon filter traps most chemicals. Each kit includes three replacement traps. Replace after 300 injections.

Split Vent Trap Kit: cat.# 20640

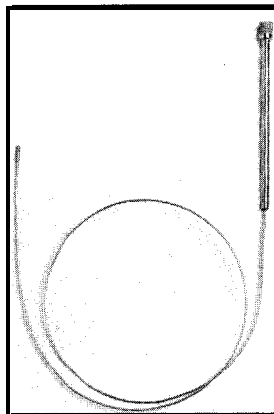
Replacement cartridges: cat.# 20641, 3-pack

Replacement Chemical Trap for HP 5890 GCs

GC carrier gas line contamination is a difficult problem to diagnose and correct. Carrier gas line contamination can occur from carrier gas impurities or by sample backflash. Contamination can appear as ghost peaks or a rising baseline not attributed to the inlet, column, or detector. **HP 5890 GCs** incorporate a small chemical filter in the carrier gas line to reduce the possibility of contamination. However, the trap capacity is low and must be periodically changed or contamination will occur. It is recommended the traps be replaced annually under normal operating conditions or more frequently under heavy usage.

Restek's chemical trap is easy to install and attaches to the same fittings as the original equipment. It incorporates built-in frits and adsorbents to remove both moisture and hydrocarbons. Additionally, Restek's trap can be regenerated to

remove trapped contaminants and restored to its original performance.



Replacement Chemical Trap for HP 5890 GCs: cat.# 21610

Leak Detectors

Restek's Leak Detective

- Detects minute leaks not possible with liquid leak detectors*.
- Compact, lightweight, hand-held design.
- Lowest cost thermal conductivity leak detector available.
- Contamination-free leak detection.
- Battery or line operated.
- Detects helium or hydrogen leaks at >20ul/min. or >200ppm.

Restek's new Leak Detective is the affordable solution for GC leak detection. Leaks can increase detector noise, cause baseline instability, waste carrier gas, and shorten column lifetimes. The Leak Detective detects minute gas leaks which may go undetected by liquid leak detectors.

The compact design of the Leak Detective ensures comfortable hand-held operation. Trace leaks of both helium and hydrogen** can be detected. Sensitivity is similar to other models on the market with detectability of helium or hydrogen and leak rates of 20ul/min. or an absolute concentration less than 200ppm. Leaks are indicated by an audible alarm, as

well as an LED readout. Two 9-volt batteries (included) provide 10-12 hours of continuous operation, or the unit can be used with an AC adaptor (included).



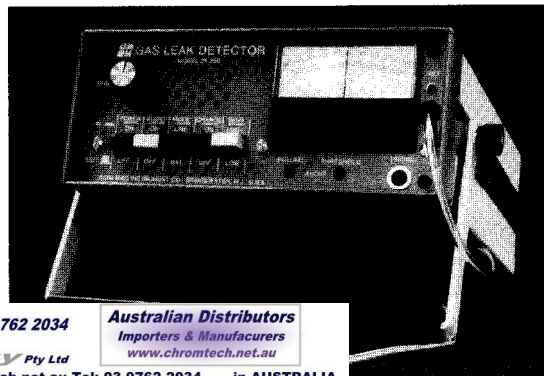
Restek's Leak Detective (110 volts): cat.# 21607
(220 volts): cat.# 21609

**** Not designed for use in explosive atmospheres.**

Gow-Mac Leak Detector

- Identifies minute leaks that are undetectable by liquid leak detectors*.
- Contamination and residue-free leak detection.
- Prolongs column life.

The Gow-Mac leak detector is a must for all capillary chromatographers. It is a portable unit that operates on line voltage or on an internal, rechargeable lead/acid gel battery.



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Gow-Mac Leak Detector: c

* Never use liquid leak detectors on actually drawn into the column.

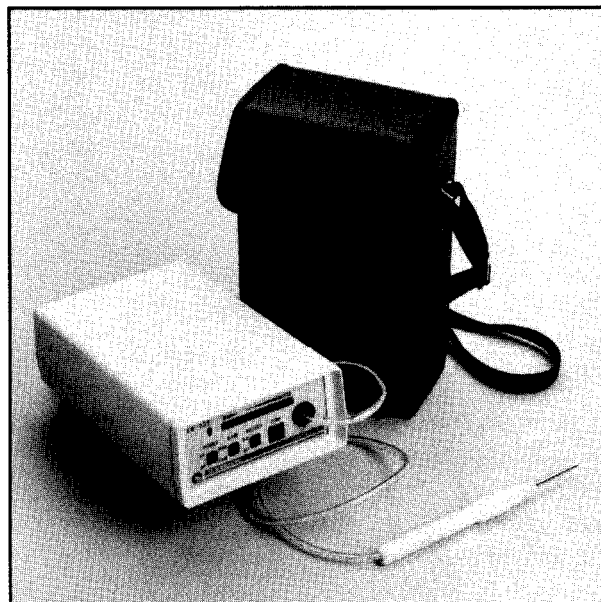
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Website NEW : www.chromalytic.com.au E-mail : info@chromtech.net.au Tel: 03 9762 2034 . . . in AUSTRALIA

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- . Portable
- . Quick response
- . High sensitivity
- . Simple operation
- . Contamination-free leak detection

Leaks in a gas chromatographic system can cause problems ranging from increased detector noise, baseline instability, and short column lifetime, to wasting expensive carrier gases. Electronic leak detectors allow analysts to detect minute gas leaks undetected by liquid leak detectors. This leak detector's compact size is designed for easy transport and hand-held usage. Simple push button operation allows one-touch zero adjustment, while the low dead volume sampling line provides quick sample response. Trace leaks of both helium and hydrogen* can be detected using the high sensitivity range. Four AA alkaline batteries (not included) provide up to 12 hours of continuous operation.

* Not designed for use in explosive atmospheres.



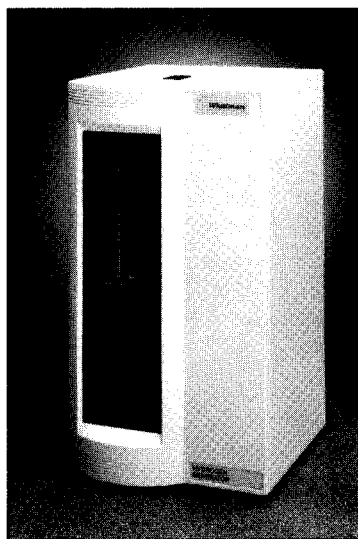
Compact Leak Detector: cat.# 21605

Generators

Whatman Hydrogen Generators

Whatman hydrogen generators produce ultra-pure hydrogen for use as a GC carrier gas or a fuel gas for FIDs, NPDs, FPDs, or a reaction gas for ELCDs. Hydrogen is produced by electrolytic dissociation of water and purified using a palladium membrane.

- . Produces ultra-pure hydrogen (99.9999+%).
- . Available in two sizes, 150cc/min. or 300cc/min.
- . Pressure output from 0 to 60psig.
- . Requires deionized water and electricity only.
- . Low maintenance, no dessicant cartridges required.
- . Meets OSHA requirements.
- . Less than 50ml of hydrogen stored at any time.
- . Safety features include automatic shutdown for over-pressure, electrolyte leak, and low water supply.
- . Compact size takes up little bench space.
- . Leasing available for low monthly payments.



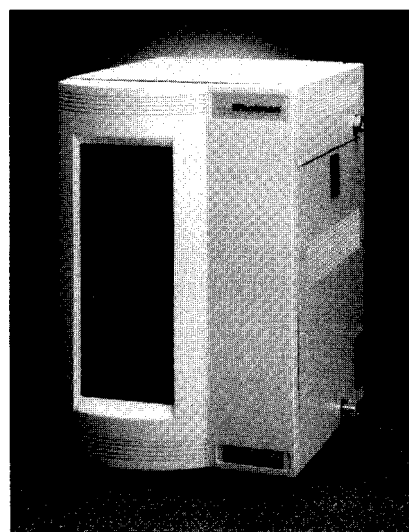
Hydrogen Generator
(150cc/min.):
cat.# 20687

Hydrogen Generator
(300cc/min.):
cat.# 20688

Whatman air generators turn your in-house compressed air supply into ultra-pure air for use with FIDs. Four sizes are available to supply from two to fifty FIDs.

- Produces ultra-pure air (less than 0.1ppm total hydrocarbons).
- Available in four sizes (650cc/min. to 18000cc/min.).
- Pressure output up to 125psig.
- Typical payback is less than one year.
- Three year supply of filter elements supplied with each unit.
- Compact size takes up little bench space.
- Installs easily and operates quietly.
- Leasing available for low monthly payments.

1000cc/min. cat.# 20684	3500cc/min. cat.# 20680	7000cc/min. cat.# 20681	18000cc/min. cat.# 20682
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Zero Air Generator

Whatman Nitrogen Generators

Whatman nitrogen generators produce ultra-pure nitrogen for use as a GC carrier gas or make-up gas for FIDs, NPDs, ECDs, or FPDs. The generator can also be used for evaporating solvents, switching valves, purging valves, and purging chambers. The nitrogen is produced by a three stage process which first eliminates hydrocarbons from the compressed air supply, then removes O₂ to less than 25ppm and removes water vapor and CO₂. The final step removes trace levels of oxygen, water vapor, carbon monoxide, carbon dioxide, and hydrocarbons to less than 1ppm.

- Produces ultra-pure nitrogen (99.9995%).
- 550cc/min. capacity.
- Only requires a compressed air source and a 100, 60 Hz electrical supply.
- Contains a downstream scrubber for maximum purity.
- Provides a supply of gas to maintain consistent analytical results.
- Safe, reliable, low maintenance.
- Compact size takes up little bench space.
- Frees valuable floor space by eliminating costly, inconvenient gas cylinders.
- Leasing available for low monthly payments.

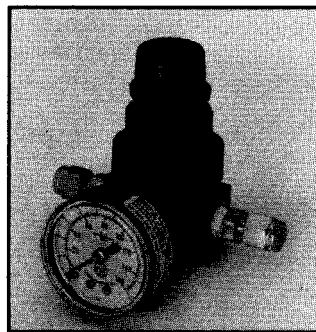


Nitrogen Generator (550ml/min.): cat.# 20696

Regulators

MINICYL General Purpose Regulator

This compact general purpose regulator has hundreds of laboratory applications including air-drying glassware, sparging or evaporating solutions, and controlling pneumatic valves. It is constructed of light-weight aluminum with an elastomer diaphragm. Each regulator comes with a 0-60psig gauge and either 1/8" or 1/4" female tube fittings.



1/8" female fitting: cat.# 20610

1/4" female fitting: cat.# 20611

High Purity Two-Stage Regulator

These two-stage regulators are ideal for use with high purity gases. They are constructed of chrome-plated forged brass with stainless steel diaphragms on both stages. A diffusion-resistant diaphragm valve is installed at the outlet of the regulator for easy on/off control. These regulators are designed to deliver constant pressure regardless of fluctuations in tank pressure. The maximum inlet pressure is 3000 psig and the outlet pressure range is 5-125psig. The outlet port accepts 1/4" male NPT fittings. Available for flammable gases, inert gases, and air.



CGA 580 (N₂, He, & Ar): cat.# 20606

CGA 350 (H₂ and P₂): cat.# 20607

CGA 590 (Air): cat.# 20608

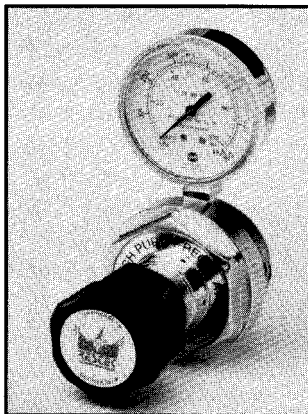
1/4" NPT Chrome Plated Nuts & Nipples with Internal Frit

CGA 580 (N₂, He, & Ar): cat.# 20878

CGA 350 (H₂ and P₂): cat.# 20879

CGA 590 (Air): cat.# 20880

High Purity Single-Stage Regulator

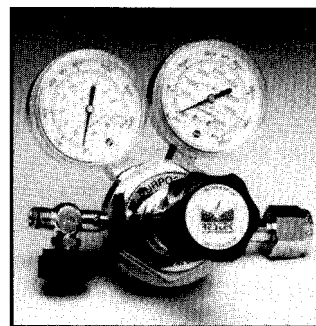


This single-stage regulator is ideal for low-pressure lines supplying gases to gas chromatographs. It is constructed of chrome-plated forged brass with a stainless steel diaphragm. The maximum inlet pressure is 350psig and the outlet pressure range is 4-80psig. The inlet and outlet ports accept 1/4" male NPT fittings.

cat# 20609

General Purpose Two-Stage Air Regulator

General purpose regulators are perfect for supplying air to FIDs, NPDs, mechanical actuators, and other applications where diffusion through a polymeric diaphragm is not a concern. These two-stage regulators are constructed of chrome-plated brass with nylon reinforced diaphragms. Maximum inlet pressure is 3000psig and outlet pressure range is 2-125psig. Outlet connection is a 1/4" male NPT fitting. Available for industrial air (CGA 590) and breathing air (CGA 346).

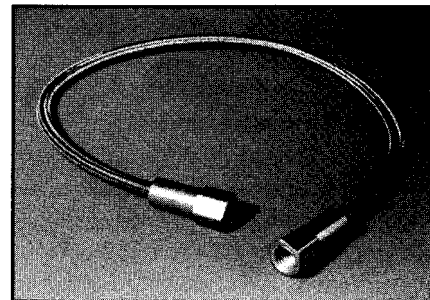


CGA 346: cat.# 21675

CGA 590: cat.# 21674

Flexible Pigtails

These flexible pigtails are 24 inches long and are constructed of braided stainless steel with a Teflon core. The Teflon core provides an inert gas pathway. They are rated for pressures up to 300psig. Each end is equipped with 1/4" NPT male fittings.



Flexible Pigtail: cat.# 20615

Educational Materials

Restek's Capillary Chromatography Seminar

Our seminar is geared towards the practical aspects of capillary chromatography. Information used on a day-to-day basis is presented. The following is a description of Restek's general capillary chromatography seminar:

New Multimedia Presentation Format!

Capillary Background and Terminology

This section discusses the essential basics of capillary chromatography. What are the differences between a packed and capillary column? What types of capillary columns are available to an analyst? How do they work? Concepts of chromatographic retention, separation efficiency, and selectivity will be presented on a practical, rather than theoretical level. Chromatographic equations and symbols will be defined and applied in a user-friendly format.

Inlet Systems and Injection Techniques

This section focuses on how carrier gas flow is controlled in a GC and discusses all of the common capillary injection techniques including split, splitless, flash direct and on-column, cool on-column, temperature programmable, and purge & trap. Advantages and disadvantages of each injection technique will be fully discussed with helpful hints on ways to optimize injector reproducibility.

Detection Systems

This section discusses background terminology pertaining to detectors such as sensitivity, selectivity, and dynamic range. The most commonly used capillary detectors including PID, TCD, MSD, ECD, PID, ELCD, NPD, and FPD are fully discussed with detailed detector specifications and operating hints. Animation is utilized to demonstrate the sample flow path and mechanism of detection.

Column Selection

This section utilizes many application chromatograms to show chromatographers the practical effects of changing column parameters. The effect of how column ID, film thickness, and length affect both resolution and total speed of analysis are demonstrated chromatographically to show chromatographers how to utilize and optimize each of these parameters. Stationary phase selectivity is demonstrated from a structural and functional composition. The use of dual column confirmational analysis is also discussed highlighting different ways to perform the analysis depending upon the instrument used.

Installation, Stand-by Operation, and Maintenance

This section discusses capillary carrier gas choice and how it affects sample analysis times and resolution. It covers pre-analysis instrument preparation, including regulator, trap, and purifier use, and sleeve deactivation procedures, plus additional inlet and detector considerations. A trouble-free installation sequence and proper column conditioning is presented to prevent inadvertent column damage. Stand-by operational hints are also discussed to maximize the column lifetime. The column and system maintenance section discusses how to minimize system problems and instrument down time by performing routine system maintenance. Prevention of column contamination is shown through the use of packed inlets or guard columns. Ways to rejuvenate a contaminated column will also be discussed. System integrity can be monitored through test mixtures, selective indicators, and GC optimization. Sources and solutions for common problems will be shown and discussed.



Application Specific Seminars from the Restek Wizards:

- GC Method Development
- Environmental
- Clinical/Forensics
- Petroleum

Have questions about the course content? Would you like an estimate for your location? Call (800) 356-1688 and our marketing department will be happy to answer your questions.

Restek's Ideal Training Tool for New Chromatographers!

The technical wizards at Restek produced an instructional video that takes the mystery and frustration out of capillary column installation.

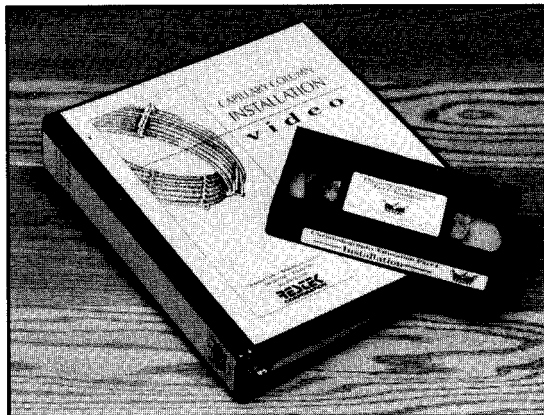
This essential resource covers critical points in the proper installation of a capillary column such as:

- Choice of carrier gas
- Instrument preparation
- Trouble-free installation
- Leak checking
- Setting carrier and detector gas flows
- Column conditioning

The installation sequence allows you to install your capillary column correctly, minimize downtime, and get your GC up and running quickly. The video follows ASTM Method E1510-93 for installing fused silica open tubular capillary

columns in gas chromatographs to insure that your lab conforms with industry standards.

We guarantee this video will be the most useful instructional tool you'll ever use or we'll refund your money!



VHS: cat.# 20490; PAL: cat.# 2049 1



Receive Technical Literature From Restek

Catalogs

- *Chromatography Products Catalog*
- *Chemical Standards Catalog*
- *Environmental Chromatography Products Catalog*
- *MXP Capillary Column Catalog*
- *Clinical, Forensic and Pharmaceutical Chromatography Products*
- *Capillary Column Installation Guide*
- *Performance Products for HP GCs*

Technical Guides

- *Helpful Hints for Analyzing Volatile Organics*
- *Operating Hints for Split/Plitless Injection*
- *Guide to Minimizing Septa Problems*
- *A Guide When Injecting Dirty Samples*
- *Guide to Direct/On-Column Flash Vaporization Injection*
- *Guide to the Analysis of Petroleum Hydrocarbons in Soil and Water*

Bi-Monthly Newsletter

- *The Restek Advantage*

Wall Charts

- *Capillary Chromatography Essentials Wall Chart*
- *EPA Method Reference Wall Chart*

Contact your local distributor for free literature

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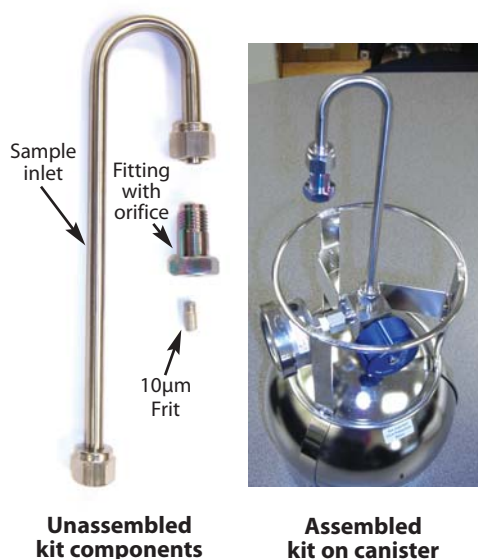
A Guide to Whole Air Canister Sampling

Equipment Needed and Practical Techniques for Collecting Air Samples

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Preparing the Sampling Train	6
Preparing the Canister	7
Field Sampling	7
Analysis of Collected Samples	9
Cleaning the Sampling Train	10
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Figure 1 Canister grab sampling kit.



I. Introduction

Ambient air sampling involves collecting a representative sample of ambient air for analysis. There are two general approaches: 1) “whole air” sampling with canisters or Tedlar® bags and 2) “in-field concentration” sampling using sorbent tubes or cold traps. In this guide, we focus on collecting whole air samples in canisters, a flexible technique with many applications (Table I).

Table I Canister applications.

Methods	US EPA TO-14A, TO-15; ASTM D5466 OSHA PV2120; NIOSH Protocol Draft
Sampling Environment	Ambient air, indoor air, vapor intrusion, emergency response
VOC Range	<C3 to ~C10
Sampling Type	Grab & integrated sampling
Sensitivity	ppt to ppm

Passive vs. Active Sampling

In canister sampling, two sampling techniques are commonly used: passive sampling and active sampling. Active sampling requires the use of a pumping device whereas passive sampling does not.

In passive sampling, an air sample is pulled through a flow controller into an evacuated canister over a chosen period of time, ranging from 5 minutes to 24 hours. The sampling period and the flow rate determine the canister volume required. In active sampling, a pump is used to push the sample through a mass flow controller and into the canister. Additional sample can be collected, relative to the amount that can be collected by passive sampling, by pressurizing the canister with sample. Commonly the sample is pressurized to 15 psig, effectively doubling the sample volume.

Although active sampling is very flexible, a drawback to using a pump is the need for additional quality assurance requirements for sample integrity (i.e., no artifacts or loss of analytes). Additionally, a pump requires a battery or line power source, which may be difficult in remote field-site sampling.

Grab vs. Integrated Sampling

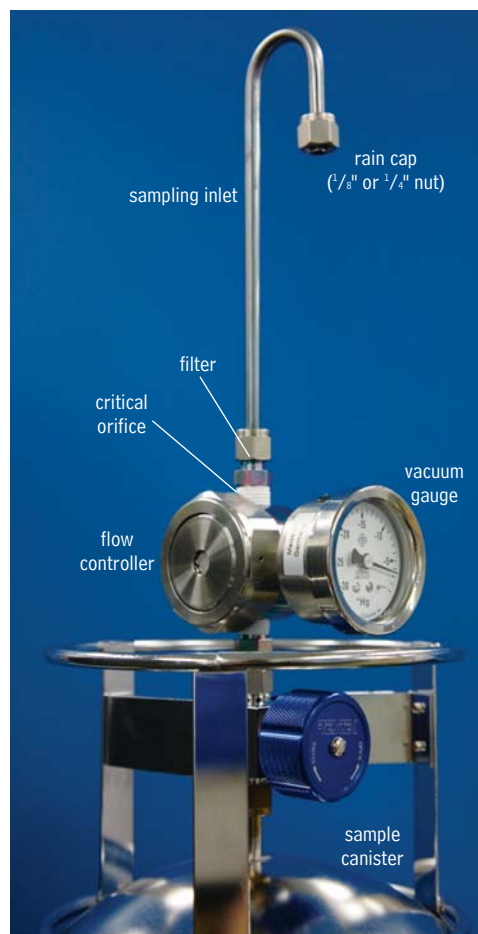
If the environment is not changing, or if only a qualitative sample is needed, a simple “grab” sample can be obtained. For example, an evacuated sample canister can be opened and sample rapidly collected at an uncontrolled rate, usually over several seconds, until the container reaches equilibrium with atmospheric pressure. Generally this qualitative approach is used when unknown analytes must be identified, when the air contains high concentrations of analytes at certain (short) times, or when an odor is noticed and a sample must be obtained quickly. Paired grab samples (before/after or smell/no smell) often are employed to qualitatively diagnose a perceived problem.

To obtain a more representative sample requires time-integrated sampling. A flow restrictor is used to spread the sample collection flow over a specific time period to ensure an “average” composited or time-weighted average (TWA) sample. A TWA sample will accurately reflect the mean conditions of the ambient air in the environment and is preferred when, for regulatory or health reasons, a typical exposure concentration is required for a situation that may have high variability, as in an occupational setting.

II. Equipment Used for Passive Air Sampling

To ensure a valid sample when using a passive sampling technique, it is important that the flow rate not change greatly during the time interval specified for the integrated sample. The proper sampling equipment helps accomplish this objective. A typical passive sampling train should include the following components, all constructed of stainless steel: a sampling inlet, a sintered metal particle filter, a critical orifice, a flow controlling device, a vacuum gauge, and a canister (Figures 1 and 2).

Figure 2 Integrated sampling kit.



Sampling Inlet

The sampling inlet—the entrance to the sampling train—typically is cleaned stainless steel tubing, either 1/4" OD or 1/8" OD. US EPA Compendium Method TO-14A/15 recommends sampling at a height of 2 meters above the ground. In a highly trafficked area, this would minimize the problem of dust particles entering the sampling train. This height is not mandatory, however, and it is common practice to use an inlet that is 12" (approximately 1/3 meter) high. The EPA also recommends having the entrance of the sampling inlet face downward to prevent raindrops from entering the inlet. In some sampling trains, a 1/8" or 1/4" nut at the entrance of the inlet keeps water droplets away from the edge of the inlet, where they could be drawn into the sampling train with the sample.

Particle Filter

The particle filter is installed in the sampling train prior to the flow-controlling device to prevent airborne particles from entering the sample flow path. Particles could partially obstruct the flow path and alter the flow rate during sampling. In extreme cases, particles could plug the flow path and stop the sample flow. The smallest orifice commonly used in a passive sampling train is 0.0012" (approximately 30 micrometers). Without a particle filter, dust particles could occlude this opening as they accumulate in the orifice fitting. Particles also can affect the leak integrity of the valve, and possibly cause damage to the valve.

Two types of filters are used for this application, frit filters and in-line filters (Figure 3). A variety of models of each type are available; most are of sintered stainless steel and have 2-, 5-, or 7-micron pores. Use of smaller pore filters reduces the likelihood of problems from airborne particles. EPA Compendium Method TO-14A/15 recommends using a particle filter with 2-micron pores.

Critical Orifice

The critical orifice (Figure 4) restricts the flow to a specified range (Table II). In conjunction with the flow controller, this allows the canister to fill at a certain rate over a specified time period. The most common critical orifice design is a series of interchangeable stainless steel 1/4" NPT to 1/4" compression unions, each fitted with a precisely bored ruby orifice. Each orifice provides a specific flow range (Table II). Stability over a wide range of temperatures makes ruby the construction material of choice. Typically during field sampling, the sampling train is subjected to temperature fluctuations that would cause metals to contract or expand, affecting the diameter of the aperture and thereby affecting flow. Ruby will not expand or contract across ambient temperature extremes incurred during sampling.

Table II Critical orifice diameter vs. flow rate.

Orifice Diameter (in.)	Flow Rate Range (mL/min.)	Canister Volume / Sampling Time			
		1L	3L	6L	15L
0.0008	0.5–2	24 hr.	48 hr.	125 hr.	—
0.0012	2–4	4 hr.	12 hr.	24 hr.	60 hr.
0.0016	4–8	2 hr.	6 hr.	12 hr.	30 hr.
0.0020	8–15	1 hr.	4 hr.	8 hr.	20 hr.
0.0030	15–30	—	2 hr.	3 hr.	8 hr.
0.0060	30–80	—	—	1.5 hr.	4 hr.
0.0090	80–340	—	—	0.5 hr.	1 hr.

A critical orifice can be used as the sole flow-restricting device, but it cannot ensure uniform flow. Since the source pressure of the flow changes during sampling, the flow rate through the orifice can also change, resulting in an invalid time-integrated sample. It is important that a highly consistent flow rate be maintained during passive sampling, and this is accomplished by the flow controller.

Flow Controller

The flow controller (Figure 4) maintains a constant sample flow over the integrated time period, despite changes in the vacuum in the canister, or in the environmental temperature (Figure 5). In the Veriflo® Model SC423 XL Flow Controller shown in Figure 4, the critical orifice acts as a flow restrictor,

Figure 3 Filters used in sampling trains.

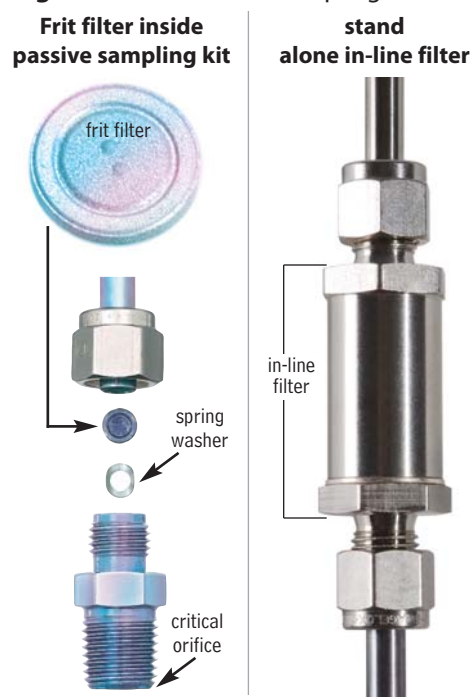


Figure 4 Flow controller & critical orifice.

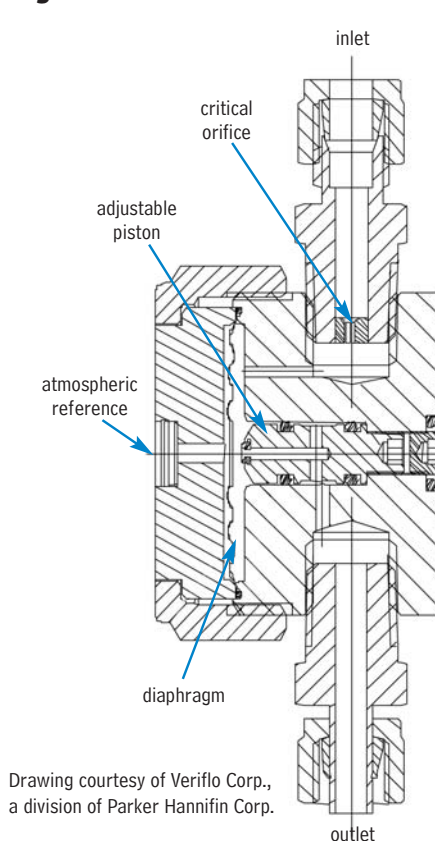
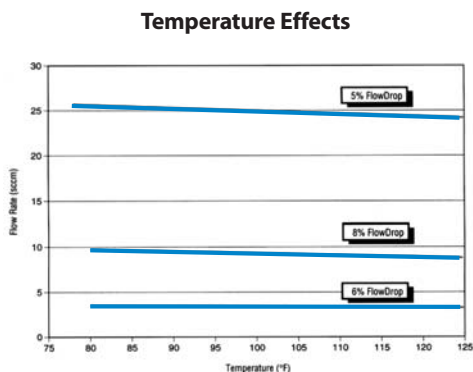


Figure 5 A flow controller will maintain a constant sample flow despite changes in canister pressure or environmental temperature.

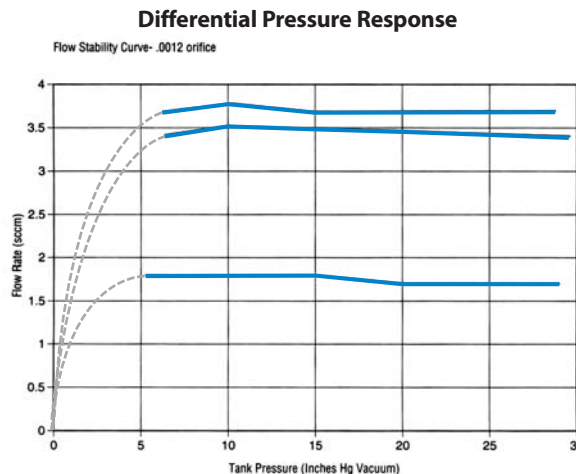


Courtesy of Veriflo Corp., a division of Parker Hannifin Corp.

upstream of a constant back pressure. This constant back pressure is established by the balance between the mechanical spring rate of the diaphragm and the pressure differential across the diaphragm. The latter is established by the pressure difference between the atmospheric pressure, the vacuum in the canister, and the flow through the critical orifice. The net result is a constant flow.

The critical orifice determines the flow range. The adjustable piston is used to set a specific, fixed flow rate within the flow range. An adjustment to the position of the piston changes the back pressure, which changes the pressure differential across the critical orifice. If the piston is lowered away from the diaphragm, the flow rate will increase. If the piston is raised toward the diaphragm, the flow rate will decrease. This flow controller will accurately maintain a constant flow despite changes in vacuum over a range of -30" Hg to -7" Hg. Flow is constant until the vacuum range of the device is exceeded, making the flow controller unable to maintain the constant pressure differential. In Figure 6, for example, the flow rate is constant from -29.9" Hg to approximately -7" Hg, at which point the flow rate decreases because the flow controller is unable to maintain the proper pressure differential. This control will allow the user to collect approximately 5 liters of sample in a 6-liter canister. This is an extremely important factor in obtaining valid time-integrated samples through passive sampling. We will discuss this point further in the **Field Sampling** (Section V) of this guide.

Figure 6 A flow controller will maintain a constant sample flow until it is unable to maintain a stable pressure differential across the critical orifice.



Courtesy of Veriflo Corp., a division of Parker Hannifin Corp.

Figure 7A Field Sampling Gauge



Figure 7B High Accuracy Laboratory Gauge



Field Sampling and Laboratory Vacuum Gauges

A vacuum gauge as shown in Figure 7A enables sampling personnel to visually monitor changes in the vacuum in the canister during sampling. If the flow rate changes unexpectedly (e.g., due to a leak or an incorrect setting), the vacuum gauge will indicate a disproportionately high or low vacuum in the canister, and corrective action can be taken (i.e., flow adjusted) in time to ensure a valid sample. This type of vacuum gauge is attached to the sampling train for use in the field. The vacuum gauge should be of high quality to ensure that it does not introduce contaminants into the sample. All wetted parts in the vacuum gauge are constructed of stainless steel; Restek gauges are accurate to within 1% of full scale. Once used for sampling, a gauge must be cleaned, and should be certified clean. Procedures are described later in this guide.

To monitor pressure in the canister before and after sampling, use a more accurate measuring device. For example, test gauges built by Ashcroft®, as shown in Figure 7B, are accurate to 0.25% of full scale. These sensitive gauges should not be used in the field—they typically are wall mounted in the lab.

Canister

The canister is a stainless steel vessel designed to hold vacuum to less than 10 mTorr or pressure to 40 psig. Canisters are available in a range of volumes: 400 mL, 1.0 liter, 3.0 liter, 6.0 liter, and 15 liter. The size of canister used usually depends on the concentration of the analytes in the sample, the sampling time, the flow rate, and the sample volume required for the sampling period (Table II, page 3). Typically, smaller canisters are used for more concentrated samples, such as soil gas collection, 3-liter and 6-liter canisters are used to obtain integrated (TWA) ambient air samples at sampling times of up to 24 hours, and large 15-liter canisters are used for reference standards. Sampling time will be limited by the combination of canister size and the flow rate at which the sample is to be collected.

A well-designed canister is essential to the success of the sampling project. First, the canister should be made of stainless steel, so the collected sample will not permeate through the vessel wall or degrade due to exposure to light during shipment to the analytical laboratory. Second, the interior surface of the canister should be inert, to reduce the potential for interactions with the analytes in the sample. Third, all canisters involved in a particular application should be of consistent volume, to simplify calculating sample volumes. Finally, the canister should have a high quality valve that resists abuse in the field (e.g., overtightening that potentially could cause leaks). An inferior valve can fail, causing sample loss and incurring replacement costs. It can be more expensive to sample again than to replace a valve.

Two types of canisters are available, the difference being the interior surface. The traditional canister is the stainless steel SUMMA® or TO-Can® canister. The interior of this type of canister is electropolished, using a polishing procedure (developed by Molecotrics) that enriches the nickel and chromium surface and makes it more inert than untreated stainless steel. The new generation of sampling canister is typified by the SilcoCan® canister. Like the SUMMA® or TO-Can® canisters, the SilcoCan® canister is made of stainless steel, and the interior is electropolished, but in an additional step—Siltek® treatment—an inert layer is chemically bonded to the interior surface. Siltek® treatment makes the surface inert not only for relatively inactive organic compounds, but also for compounds that are highly reactive with metal surfaces, such as sulfur-containing compounds. Thus, surface inertness for SilcoCan® canisters exceeds that for SUMMA® and TO-Can® canisters.

Canister Valve

The valve on a sampling canister must be of high quality, with the following characteristics: leak integrity, a metal seat, stainless steel wetted surfaces, and a packless design. A metal seat eliminates offgassing of seat components into the sample and memory effects in the seat material. A packless design provides a completely enclosed system, to ensure no contamination from lubricants or packing material occurs. Various valves are used, the most common being the Swagelok® SS4H bellows valve and the Parker Hannafin diaphragm valve with metal seat. Several valve options are available for Restek canisters.

The connection of the valve to the canister is critical. The connection must be leak tight, to ensure a correct sampling flow rate, but use extreme caution to prevent overtightening the tube compression fittings. To ensure a leak tight valve, always use a pre-filter (such as an inline filter) to prevent valve seat damage.



Ensure Accurate Sampling of Reactive Compounds with Siltek® Treatment

Siltek® treatment is a proprietary process, developed by Restek Corporation, through which an inert layer is chemically bonded to a metal surface. The surface produced by this treatment is virtually inert to active compounds. The stainless steel pathway described in this guide is sufficient for sampling atmospheres containing only nonreactive compounds, but for reactive compounds the entire sampling pathway should be Siltek® treated to eliminate contact between the reactive analytes and the metal surfaces. Siltek® treatment can be applied to the interior surfaces of the canister and valve, to ensure an inert sample pathway.



Table III Flow rates for integrated sampling, using a 6-liter canister and sampling on the flat portion of the flow curve for the flow controller (Figure 5).

Sampling Period (hours)	Flow Rate Range (mL/min.)
0.5	133–167
0.75	89–111
1	67–83
2	33–42
4	17–21
8	8–10
12	5.6–6.9
16	4.2–5.2
24	2.8–3.5
125	0.5–0.7

Collected volume is 4–5 liters
(flow = volume in mL / sampling time in min.).

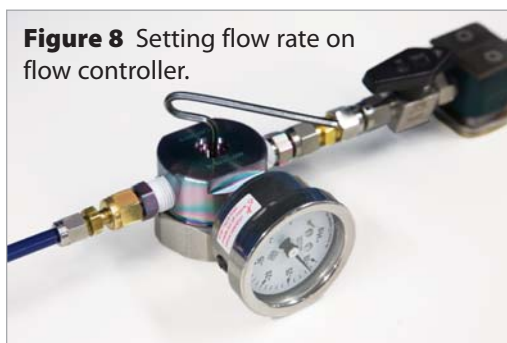


Figure 8 Setting flow rate on flow controller.

Important Precautions!

- Only hand tighten knob to close valve. Overtightening may damage seat causing leakage.
- Tighten compression fitting on valve inlet only $\frac{1}{4}$ turn past finger tight. Overtightening will cause leakage.
- Use prefilter during sampling to prevent particulate damage to valve.
- Do not disassemble valve—disassembly may void warranty.
- Protect valve inlet by replacing brass cap when not in use.
- Do not exceed canister maximum pressure of 40 psig.

III. Preparing the Sampling Train for Use

The sampling train must be prepared in the laboratory before it can be used in the field. The train must be assembled and leak tested, the flow rate must be set, and the train must be certified clean. All of the following information should be documented for the chain of custody for the passive sampling train and the sample collected with it.

Assemble, Leak Test, and Set the Flow Rate of the Passive Sampling Train

Choose the critical orifice (Table II, page 3) according to the sampling period and flow rate you anticipate using (Table III). This will ensure an accurate and valid sample. There should be a marking on the outside of the critical orifice fitting indicating the size of the orifice. In a clean environment, assemble the sampling train components as shown in Figure 2 (page 2). It is imperative that you leak test the assembled train. If the sampling train leaks during sampling, the final pressure in the canister will not be the desired final pressure, making the sample invalid. The most common reason for invalid samples is leaks within the sampling train. There are two ways to leak test the train:

1. Pass helium gas through the flow controller and use a sensitive helium leak detector to test for leaks (e.g., Restek Leak Detector).

or...

2. Cap the inlet, attach the sampling train to an evacuated canister, open the valve on the canister and evacuate the sampling train. Then, close the valve and monitor any pressure change in the static sampling train. Leaks of less than 1 mL/min. can be detected in 1–2 minutes.

This is a good practical test—the small internal volume of the passive sampling train, combined with even a small leak, will produce a large change in monitored pressure. According to EPA Method TO-15, the pressure change should be less than 2 psig (13.8 kPa) over a 24-hour period.

After you are certain the sampling train is leak-free, set the desired sampling flow rate.

To set the desired flow rate follow these steps:

1. Remove the protective cap from the back of the Veriflo® Flow Controller SC423XL body.
2. Connect either an evacuated canister or a vacuum source to the outlet of the sampling train.
3. Connect a high quality calibrated flow meter (i.e., mass flow meter, rotameter, GC-type flow sensor [e.g., Restek ProFLOW 6000 Electronic Flowmeter, cat.# 22656]) to the inlet of the train.
4. Apply vacuum by opening the canister or turning on the vacuum source.
5. With a 3 mm hex (Allen®) wrench, adjust the piston gap screw to achieve the desired flow rate (Table III). Between adjustments allow the flow to equilibrate for several minutes. See Figure 8.
6. Replace the protective cap onto the back of the Veriflo® Flow Controller body.

Cleanliness: Certifying the Sampling Train for Use

US EPA Compendium Method TO-14A/TO-15 requires that the sampling train be certified clean prior to use. Certify the train by passing a humidified, high-purity air stream through the train, concentrating the exit gas on a trap, and analyzing the gas by gas chromatography/mass spectrometry or other selective detector. For the sampling train to pass certification the analytical system should not detect greater than 0.2 ppbv of any target VOC.

The certified sampling train should be carefully packaged in aluminum foil or in a clean container for storage or for shipment into the field. Care in packaging is critical. Careless handling could affect the preset flow rate. When the sampling train is ready for sampling, prepare the canister.

IV. Preparing the Canister for Sampling

Preparing a canister for sampling involves certifying the canister clean, evacuating the canister to final pressure for use, and identifying the canister. All information acquired during these processes is needed for the chain of custody.

Certifying the cleanliness of the canister is important toward ensuring that results reported are solely from the site sampled, and not contaminated with residue from a previous site or volatiles in laboratory air. To certify a canister clean, fill the canister with humidified air, pass the air from the canister through an adsorbent trap and analyze the adsorbent for target VOCs by GC/MS or other selective detector. Two US EPA methods discuss canister certification: EPA Compendium TO-12 and EPA Compendium TO-14A/TO-15. To comply with EPA Compendium Methods TO-14A/TO-15, the analytical system should not detect greater than 0.2 ppbv of any target VOC. To comply with EPA Compendium Method TO-12 the analytical system, GC/FID, should not detect greater than 0.02 ppmC hydrocarbons. Although batch certification of canister cleanliness is a relatively common practice, we recommend certifying and documenting each canister individually. Detailed cleaning instructions are presented in Section VIII. Cleaning the Canister (page 11).

Some laboratories certify a canister for VOC stability by introducing a low concentration test mixture into the canister and measuring degradation over a specified time period. If the canister meets the specification, it is certified for use. We recommend using such studies to ensure the effectiveness of a canister or group of canisters for a proposed application.

Once the canister is certified clean, evacuate the canister to a final vacuum of 10-50 mtorr, using either the canister cleaning system or a clean final vacuum system. This vacuum is critical to ensure the correct amount of sample is collected. Use an accurate test gauge (shown in Figure 7b, page 4) or digital pressure tester to ensure final vacuum has been reached and to document the final vacuum reading for the chain of custody. Install a brass cap nut onto the canister valve to ensure no contamination can enter the sample pathway during shipment to the field.

Apply an individual identity to the canister, either with a label and serial number or with a bar code.

Some analysts prefer to introduce surrogate standards into the canister prior to sampling. Debate on this practice revolves around theories that there are potential loss issues due to low humidity and inadequate surface passivation by water. Neither Restek chemists nor our consulting experts recommend adding surrogates to the canisters. If you choose to introduce surrogates into your canisters prior to sampling, be sure to recheck and record the vacuum reading for each canister after adding the surrogates.

V. Field Sampling, Using a Passive Sampling Train and Canister

It is important to mention again that the sampling train and canister must be leak tested and certified clean prior to use. To properly begin field sampling, we recommend bringing a “practice” evacuated canister and a flow measuring device with you to the field. Use this canister to verify the flow rate through the passive sampling train prior to using the train to obtain samples of record. To verify the flow rate, connect the passive sampling train to the “practice” canister. Attach a flow meter to the inlet of the sampling train. Open the canister and measure the flow rate through the sampling train. If the flow rate is within $\pm 10\%$ of the flow rate set in the lab, the train is ready to be used on the formal sampling canister. If the flow rate is not within these limits, adjust the flow rate by adjusting the piston gap screw.

When the flow rate is confirmed, record the rate as the canister flow rate for the chain of custody form.

Pressure Conversion Table

Pressure	psi	atm	kg/cm ²	torr	kPa	bar	inches Hg
psi =	1	0.068	0.0703	51.713	6.8948	0.06895	2.0359
atm =	14.696	1	1.0332	760	101.32	1.0133	29.921
kg/cm ² =	14.223	0.967	1	735.5	98.06	0.9806	28.958
torr =	0.0193	0.00132	0.00136	1	0.1330	0.00133	0.0394
kPa =	0.1450	0.00987	0.0102	7.52	1	0.0100	0.2962
bar =	14.5038	0.9869	1.0197	751.88	100	1	29.5300
in Hg =	0.49612	0.0334	0.0345	25.400	3.376	0.03376	1

Multiply units in the left-most column by the conversion factors listed in the columns to the right.

e.g., 10PSI x 0.068 = 0.68atm, 10 bar x 29.5300 = 295.300 inches Hg

did you know?

Our light-weight tripod holds 2 canisters securely without any tools.



To begin sampling, using the formal sampling canister, follow these steps:

1. Remove the brass cap nut from the canister valve.
2. If you are using a test gauge, attach the gauge to the canister and record the vacuum reading. If you choose not to use a test gauge under field conditions, record the reading on the vacuum gauge that is part of the passive sampling train.
3. Attach the verified passive sampling train to the canister.
4. Record the sampling start time and necessary meteorological data.
5. Open the canister valve and begin sampling.
6. Periodically check the canister throughout the sampling period to ensure the pressure reading is accurate and sampling is proceeding as planned.
7. Once the sampling period is complete, close the valve and remove the sampling train. Check the final pressure within the canister, using the test gauge or the vacuum gauge in the sampling train.

There are four possible scenarios:

- A. Ideally there will be a vacuum of -7" to -4" Hg in the canister (e.g., Table IV).
 - B. If more than -7" Hg vacuum remains, less sample was collected than initially anticipated. The sample will be valid, but the detection limit may be higher than expected. You might have to pressurize the canister prior to the analysis, which will dilute the sample and require you to use a dilution factor to determine final concentrations of target compounds.
 - C. A vacuum of less than -4" Hg indicates the sample might be skewed toward the initial part of the sampling period. This assumption usually is valid because the flow rate through the flow controller will fall once the vacuum falls below -5" Hg (Figure 6, page 4), when the change in pressure across the flow controller diaphragm becomes too small and the flow controller is unable to maintain a constant flow. Although flow was not constant over the entire sampling period, the sample may be usable because sample was collected over the entire interval.
 - D. If the ending vacuum is less than -1" Hg the sample should be considered invalid because it will be impossible to tell when the sample flow stopped.
8. Record the final pressure in the canister and replace the cap nut.

Table IV Final vacuum and volume of sample collected in 6-liter canister.

Final Vacuum ("Hg)	Sample Volume (liters)
29	0
27	0.58
25	0.99
23	1.39
20	1.99
17	2.59
15	2.99
12	3.59
10	3.99
7	4.60
5	5.0
3	5.40
0	6

Information that should be acquired at the sampling site includes the start time and interval time, the stop time, atmospheric pressure and temperature and, for ambient sampling, wind direction. Include elevation if it is a factor. These parameters often prove very useful when interpreting results.

After sampling, the canisters are sent back to the laboratory where the final vacuum is measured again with a test gauge. Using the initial vacuum and final vacuum, the sample volume collected can be determined from Equation 1.

It is also good practice to recheck the flow rate after sampling, because this will affect the sample volume (Equation 2). Laboratories typically allow a maximum deviation of $\pm 10\%$ to $\pm 25\%$ between the initial flow rate and the post-sampling flow rate.

Equation 1:

$$\text{sample volume} = \frac{\text{pressure change}^*}{\text{initial pressure}} \times \text{canister volume}$$

*initial pressure – final pressure

Example: A sample is collected in a 6-liter canister. The initial gauge pressure reading when the canister left the lab was -29.92" Hg vacuum; the final gauge pressure reading when the canister was returned to the lab was -7" Hg vacuum.

$$\text{sample volume} = \left[\frac{-29.92'' \text{ Hg} - (-7'' \text{ Hg})}{-29.92'' \text{ Hg}} \right] \times 6 \text{ L} = 4.59 \text{ liters collected}$$

Equation 2:

$$\text{sample volume} = [(\text{initial flow rate} + \text{post-sampling flow rate})/2] \times \text{sampling time}$$

Example: A flow controller was set at 3.3 mL/min. After obtaining a 24-hour sample the flow rate was 3.0 mL/min.

$$\text{sample volume} = [(3.3 \text{ mL/min.} + 3.0 \text{ mL/min.})/2] \times 1,440 \text{ min.} = 4,536 \text{ mL}$$

VI. Analysis of Collected Samples

Once received by the lab, each canister is identified from the information in the chain of custody report. The final pressure is checked to ensure no leaks appeared during transport. It might be necessary to pressurize a canister prior to the analysis; do this by adding humidified nitrogen or air to the canister to a pressure greater than 5 psig or higher, depending on the sample volume needed for analysis or for suitably diluting the sample (e.g., Table V). The need to dilute is determined by the preconcentrator instrument. Some air preconcentrators can be operated while the canister is under slight vacuum. Check with your instrument manuals or with the manufacturer to determine if you must dilute your samples prior to analysis. Dilution factors can be calculated according to Equation 3.

Equation 3:

$$\text{dilution factor} = (P_{\text{after dilution}} + P_{\text{lab atmosphere}}) / (P_{\text{lab atmosphere}} - P_{\text{before dilution}})$$

The dilution factor is calculated from the post-sampling pressure (before dilution), the final pressure (after dilution), and the atmospheric pressure in the laboratory. The factor for converting "Hg to psi = 0.491.

Example: At the end of a sampling period the gauge pressure in a canister was -7 "Hg. The canister was pressurized with nitrogen to 14.7 psig (1 Atm.).

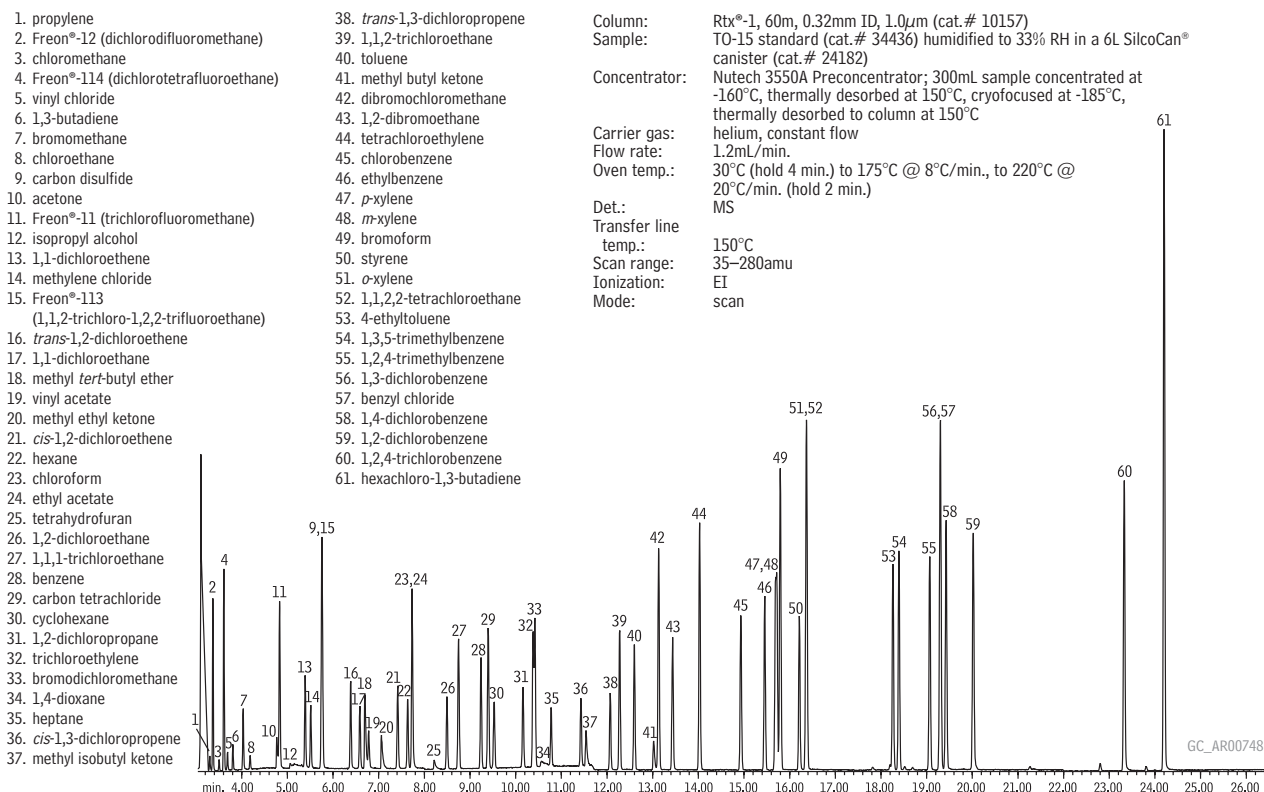
The dilution factor is $(14.7 + 14.7) / (14.7 - (7 \times 0.491)) = 2.61$

Table V Dilution factors to adjust final sampling pressure to **14.7 psig** for a 6-liter canister.

Final Vacuum ("Hg)	Sample Volume (liters)	Dilution Factor
29	0	63.77
27	0.58	20.37
25	0.99	12.12
23	1.39	8.63
20	1.99	6.02
17	2.59	4.63
15	2.99	4.01
12	3.59	3.34
10	3.99	3.00
7	4.60	2.61
5	5.0	2.40
3	5.40	2.22
0	6	2.00

To analyze the sample, withdraw an aliquot of the sample from the canister. For low level ambient air analysis, withdraw 250-500 mL of sample from the canister and concentrate the analytes by using a mass flow controller and a cryogenically cooled trap (e.g., glass beads and/or a solid sorbent). Desorb the concentrated analytes from the trap and deliver them to a cryofocuser to focus the sample bandwidth prior to introduction onto the GC column. A 60 m x 0.32 mm ID x 1.0 µm Rtx®-1 column typically is used for EPA Method TO-14A or Method TO-15 ambient air analysis; an MSD is a common detector. Figure 9 shows a typical TIC spectrum for a TO-15 ambient air analysis.

Figure 9 US EPA TO-15 ambient air analysis.



Procedures used in these chromatographic analyses generally include a multi-point calibration, using gas standards. Therefore calculations of organic compounds in collected samples are straightforward—only volumes analyzed and dilution rates are needed to determine sample concentrations. High concentration calibration gas standards are commercially available (e.g., 1 ppmv or 100 ppbv). To prepare analytical standards, introduce an aliquot of stock material into a canister and dilute with humidified air or nitrogen. After analyzing the calibration standards, determine the response factor for each analyte using the peak area counts per concentration.

After analyzing the multipoint calibration standards and calculating peak area/concentration response factors, analyze the “real world” samples. If an “unknown” sample has not been diluted, apply the corresponding response factor to each “unknown” analyte peak area to get the reporting limit concentration of the “unknown” in the analysis (typically in ppbv). If you have diluted the canister to get a positive pressure, you must apply the dilution factor to the concentration values. This is done by multiplying the reporting limit by the dilution factor.

VII. Cleaning the Passive Sampling Train

The cleanliness of the sampling train is critical to collecting accurate and representative samples. Practices followed for cleaning passive sampling equipment between uses range from purging the sampling pathway with humidified nitrogen or air for many hours, to heating the pathway during a purge, to disassembling each component, sonicating the pieces in solvent (**except for the critical orifice**), and oven baking the pieces prior to reassembly. The most suitable mode of cleaning depends on the concentrations of analytes of interest, and contaminants, in the previous sample collected.

The particle filter must be thoroughly cleaned between uses. Disassemble the filter, then remove the larger particles from the frit by blowing particle-free nitrogen through the frit from the outlet surface toward the inlet surface. After the larger particles are removed, sonicate or rinse the filter parts in methanol and then bake the parts in an oven at 130 °C to remove any residual organic vapors.

The critical orifice and flow controller can be cleaned in either of two ways. The first method is to disassemble the flow controller and clean all the metal parts with methanol. This will remove any high boiling point compounds that have condensed onto the wetted areas of the controller. Heat the cleaned parts in an oven at 130 °C to remove residual organic vapors. **Do not sonicate the critical orifice.** Do not sonicate in solvent or bake any of the nonmetallic parts, such as O-rings, or they will be damaged. Do not rinse the vacuum gauge with methanol. The vacuum gauge may be heated, but do not exceed 80 °C; higher temperatures will damage the face and the laminated safety glass lens. Heating to 80 °C will not affect the mechanical operation of the spiral bourdon tube in the vacuum gauge.

A less involved method of cleaning the flow controller is to use a heating jacket or heat gun to heat the components of the assembled sampling train, while purging the system with nitrogen. As organic compounds are heated and desorbed from the interior surfaces, the nitrogen gas sweeps them out of the sampling equipment.

Preparing the Clean Passive Sampling Train for Re-use

After the sampling train components have been cleaned, reassemble the system, check for leaks, set the desired flow rate, and certify the sampling system clean. Follow the procedures described previously in this guide. Package the clean sampling train to prevent contact with airborne contaminants.

frequently asked question

Where can I find EPA Air Toxic Methods?

pdf files of US EPA Air Toxic Methods are available at this web address:

www.epa.gov/ttn/amtic



for more info

ASTM Reference D5466 Standard Test Method for Determination of Volatile Organic Chemicals in Atmospheres (Canister Sampling Methodology)

available at www.astm.org

VIII. Cleaning the Canister

Every air sampling canister, whether new or used, must be cleaned and certified before it is used for sampling. Some laboratories batch test and certify canisters. This is done by testing and certifying one canister out of 10 following cleaning. We recommend certifying each canister clean prior to use—especially if there is potential for litigation.

For years there has been much discussion regarding what constitutes a proper procedure for cleaning canisters. US EPA Method TO-15 has provided guidance, and in the last 5–10 years automated commercially available canister cleaning systems have evolved. Because many of these systems are quite expensive, and some designs have limitations, analysts often design their own systems and methodologies for cleaning canisters. The cleaning procedure described in this section is a practical approach that will ensure canisters are suitably cleaned for ambient air sampling, whether you are using a commercially available cleaning system or a system of your own design. There are minor differences when cleaning SilcoCan® or TO-Can® (SUMMA®) canisters. We will discuss these differences in this procedure.

Air Versus Nitrogen

The two gases recommended for cleaning canisters are humidified ultra-high purity air and ultra-high purity nitrogen. The water in the humidified gas hydrolyzes impurities in the canister and, according to theory, will occupy the active sites on the interior surface, displacing the impurities and allowing them to be removed. Air is recommended when oxidation of the interior surface is desired. The oxygen content of air, 21%, is sufficient for this surface oxidation; it is not necessary to use pure oxygen gas. Nitrogen is equally effective for cleaning ambient air canisters, but will not oxidize the surface of the canister.

Heat or No Heat*

Some user-designed canister cleaning systems do not heat the canisters. Typically this does not create a problem when cleaning canisters that are used in ambient air collection, but as a safeguard we recommend heating the canisters during the cleaning process. Compounds collected in most ambient air samples are in the low ppbv range, and can be removed from a canister by multiple cycles of pressurization with humidified air or nitrogen followed by evacuation. If there are higher concentrations of contaminants in the canister, heat might be required to clean the canister satisfactorily. In addition, the cleaning cycle may be reduced when heat is applied.

Caution: Adding heat and humidified gas to a canister may create a steam pressure vessel. Some commercial cleaning systems incorporate a pressure release valve to ensure the pressure does not exceed the pressure rating of the canisters.

Cleaning Systems

- **Oven** Some canister cleaning systems are incorporated within an oven. Batch size is determined by the number of canisters that can fit inside the oven. The supply line for the humidified air or nitrogen stream and the line to the vacuum system are plumbed directly into the oven. A cold trap is employed to trap impurities. Accurate monitoring of vacuum and pressure is required. In this arrangement, the entire canister, including the valve, will be heated. This will help remove contaminants if both the valve and the canister are dirty. Typically, when using heat, it is helpful to create steam from the humidified air or nitrogen stream. An oven temperature of at least 120 °C is required, but higher temperatures often are used.

- **Heat Bands** A band heater placed around the equator of the canister typically is capable of heating the canister to approximately 130 °C. There is a heat gradient, and the valve might only receive radiant heat (approximately 70–100 °C). In most sampling situations, this lower temperature should be sufficient for effectively removing contaminants from the valve.

- **Insulated Heat Jackets** Insulated heat jackets surround and heat each canister. These jackets typically have a silicone or Teflon®-coated fiberglass fabric exterior and a fiberglass insulation interior. Some operate at a fixed temperature; others can provide variable temperature. Restek's heating jacket offers a significant advantage over alternatives because it encompasses the valve area.

- **Infrared Heat** An infrared heating system includes an infrared heat source and a reflective panel similar to the cylinder drying rack on a gas cylinder system. The infrared source and the reflective panel are placed on opposing sides of the canisters. Infrared rays from the source heat the canisters; rays that pass the canisters strike the reflective panel and heat the canisters from the opposing side.

- **User Designed** Figure 10 shows an example of a “homemade” system designed to clean 24 six-liter canisters. This design does not employ heat, but a heater can be added (see **Heat or No Heat**). It provides a humidified air or nitrogen stream to all canisters and the roughing pump on the bottom shelf is the vacuum source. This system is computer operated to automate the cleaning cycles.



Figure 10
User-designed system
for cleaning 24
six-liter canisters.

*If you are cleaning any fused silica lined canisters, and will be using heat, use humidified nitrogen, not air.

Cleaning any fused silica lined canisters with humidified air and heat above 80 °C may damage the fused silica surface, resulting in reduced recoveries of sulfur and other reactive compounds.



Cleaning Method

1. Connect all canisters to the cleaning system, then release any pressure within any of the canisters.

Apply vacuum to the system to evacuate the canisters. US EPA Method TO-14A/15 recommends evacuating the system to 50 mTorr for 1 hour, but a reduced pressure of -23 to -25 " Hg is sufficient for general cleaning.

2. After the canisters have been under vacuum for approximately 1 hour, pressurize the canisters with humidified air or nitrogen*. Pressurization will dilute the impurities and the moist air will hydrolyze them.

Pressurize canisters to 5 psig if they will be heated, or to 30 psig if they will not be heated.

Proceed to step 3 when the system has equilibrated at the designated pressure.

3. Heat the pressurized canisters to 120–250 °C, depending on the type of valve on the canister being cleaned. Different valves have different temperature limits; consult the manufacturer specifications for your valve type. Many commercial cleaning systems avoid this concern by ensuring the valve is not within the heated zone. The canister below the valve is heated but the valve receives only radiant heat.

Heat the canisters filled with humidified air/nitrogen for at least 1 hour.

4. Re-evacuate the canisters to remove the desorbed impurities.

Allow the canisters to equilibrate for 1 hour.

5. Determine if the canisters have been cleaned effectively by following the procedure in **Certifying the Canister** (p. 13). US EPA methods recommend testing every canister until a reliable procedure is developed.

Repeat steps 1–5 as necessary; the number of cycles will be determined by how dirty the canisters are and how easily they are cleaned.

We recommend developing a cleaning procedure that matches your specific sampling procedure, by testing the canisters for cleanliness after each cycle and determining the number of cycles necessary for proper cleaning.

If the canisters are not heated, the number of cycles required to clean the canisters might be higher.

6. Once a canister is clean, prepare it for collecting a sample by evacuating it to 10–50 mTorr. If your system is leak-tight, you can do this by using a roughing pump.

Many commercial systems include a molecular drag pump to reach final vacuum quickly.

*If you are cleaning any fused silica lined canisters, and will be using heat, use humidified nitrogen, not air.

Cleaning any fused silica lined canisters with humidified air and heat above 80 °C may damage the fused silica surface, resulting in reduced recoveries of sulfur and other reactive compounds.

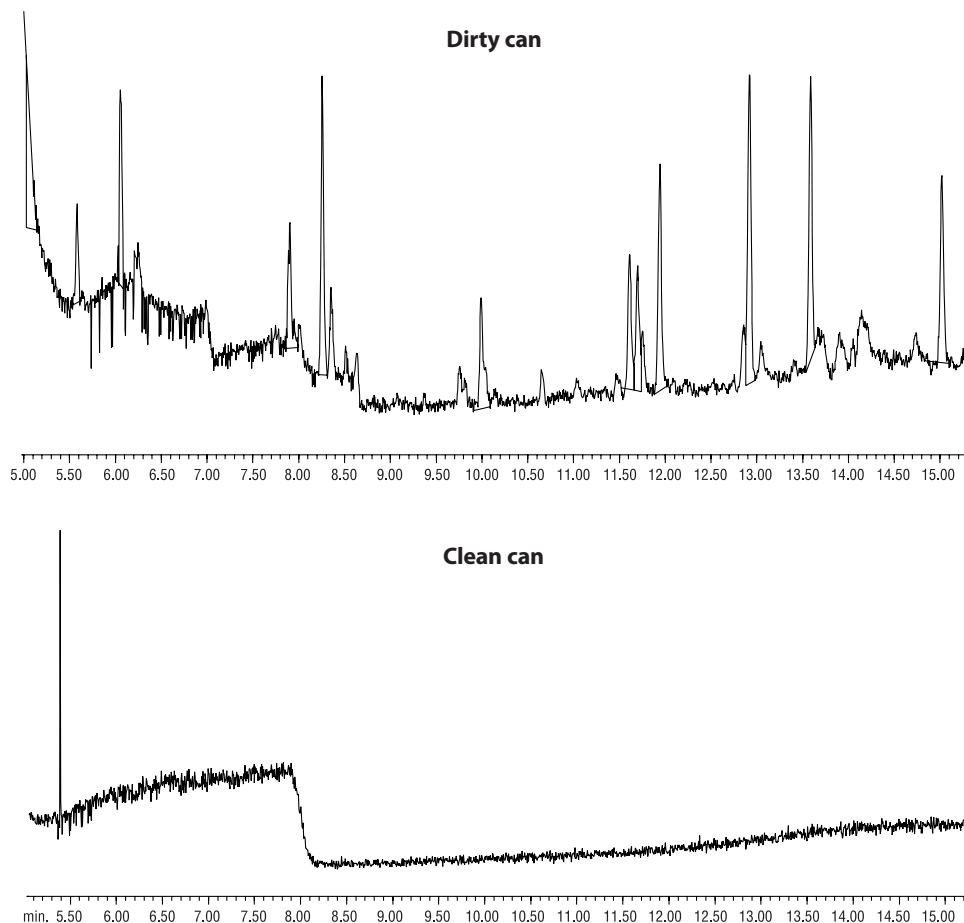
IX. Certifying the Canister

We recommend certifying canisters for both cleanliness and for analyte stability. To certify a canister clean, pressurize the canister to 14.7 psig with humidified ultra-high purity air or nitrogen after it has gone through the cleaning cycles. The humid air or nitrogen stream must be certified clean before it can be used for canister certification. Analyze an aliquot of the canister content by GC/MS or GC/FID/ECD. US EPA Method TO-14A/15 specifies a canister must contain less than 0.2 ppbv of any target VOC compound (Figure 11); EPA Method TO-12 specifies less than 0.02 ppmC, as detected by GC/FID. If a canister does not meet specification, it must be cleaned again and retested for certification.

To certify a canister for analyte stability, introduce a low working concentration of a characterized test mix into the canister. Analyze an aliquot of the contents of the canister immediately after introducing the test mixture and at periodic intervals. We recommend monitoring for changes for a minimum of 2 weeks or for a timeframe similar to your anticipated holding period. Responses should not decrease more than 20% over this period.

Commercial standards are available for stability testing, but we recommend you make your own test mixture that is comparable to the target compound list that the canister will hold. For example, if you are analyzing sulfur compound content in ambient air, prepare a sulfur-specific test mix and evaluate the canister's performance for sulfurs. Maintain a log sheet for each canister, and record the test results and certification. This will be a permanent record for each canister. Some labs certify canisters for certain compounds and use a canister only for this specific application.

Figure 11 Aliquots from a canister before and after cleaning with 2 cycles of humidified air while heated to 200 °C.



Rtx®-1 60m x 0.32mm ID x 1.0µm (cat. #10157); 50°C (hold 1 min) to 165°C @ 8°C/min. to 220°C @ 15°C/min. (hold 5 min.); flow rate = 1.4mL/min.; Nutech 3550 Preconcentrator conditions: sample = 300cc from canister, cryotrap = -160°C, desorb = 150°C, cryofocuser = -190°C, desorb = 150°C; MSD conditions: Instrument: HP5971 GC/MSD, 5 minute solvent delay, scan range = 25-260amu

X. Conclusion

A well designed and properly prepared passive sampling system helps ensure accurate, useful information is obtained from an air sampling project. In this guide, we describe the components of the system, procedures for assembling the system and preparing it for sampling, and the sampling procedure. Cleaning system options and procedures for cleaning a used sampling train and canister for certification prior to a subsequent sampling are also presented. The following section describes Restek products designed to help collect and analyze air samples.

How to Extend Canister Life

What reduces canister performance and longevity? Leakage is the most common reason for canister failure, but contamination and damage to the fused silica lining can also send canisters to the scrap yard prematurely. Here are some tips to protect your investment:

1. Prevent leaks

Use proper handling to avoid these 3 leading causes of leaks.

a. Particles in the valve

You can prevent particles from entering the valve by always using a 2 or 7 μm particulate filter during sampling and on your canister cleaning equipment. Also, protect the valve inlet by replacing the brass dust cap when not in use. The EPA-recommended metal-to-metal sealing valves provide the greatest inertness, but tend to be more sensitive to particulate damage than other valve types.

b. Galled thread fittings

Avoid galled thread fittings by using a gap gauge to prevent overtightening of compression fittings. Turning only $\frac{1}{4}$ turn past finger-tight is another rule of thumb to prevent overtightening. Use brass compression fittings on stainless steel, during nonsampling activities, such as cleaning or calibration, to minimize thread damage. Galled threads may also cause a poor connection to vacuum/pressure gauges, resulting in inaccurate measurement and misleading conclusion that canister leakage exists.

c. Overtightened valve

Canister valves are designed to close securely with hand tightening only. Overtightening a valve closure with a wrench may damage the valve seat where the seal is made.

2. Reduce contamination

a. Segregate high concentration (ppm) cans and trace concentration (ppb) cans. Use dedicated canisters, or gas sampling bags, for ppm level sampling, since it is extremely difficult to remove impurities from ppm sampling to a level suitable for trace sampling.

b. Clean the entire sampling train as you would the can to minimize introduction of contaminants into a clean can. Maximum temperature is 80 °C on the gauge and 90 °C on Restek's Veriflo® flow controller.

c. High temperature (>100 °C) humidified air (steam cleaning) provides the most effective way to remove contamination from electropolished cans (TO-Can® or SUMMA® canisters), but can damage fused silica lined cans. See #3 below for proper cleaning of fused silica lined cans.

3. Avoid damage to fused silica lined cans

Be sure to follow method recommendations when cleaning your canisters to avoid damaging the fused silica lining. Cleaning studies of SilcoCan® canisters using humidified air and heat at 80 °C and 125 °C have shown reduced recoveries of sulfur compounds, when compared to using nitrogen under the same conditions. This irreversible damage is due to oxidation of the surface, creating active sites that may affect the recovery of reactive or polar compounds. Strong acids and bases may also result in damage to the internal can surface.

XI. Air Sampling Products

Air Canisters for VOC Monitoring

SilcoCan® & TO-Can® Air Monitoring Canisters



24182

- Get high performance canisters from the innovators of fused silica coating technology.
- Variety of options available, including SUMMA can equivalent.
- Standard fittings compatible with all instrumentation and accessories.
- Exclusive manufacturer of 1L spherical canister.
- Repair service available to extend canister life.



22107

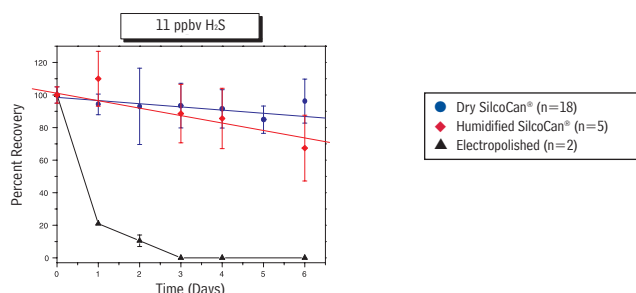
Canister Options

Sizes	1, 3, 6, 15L
Valves	Parker diaphragm, Swagelok bellows
Interior Coating	Electropolished, Siltek treated
Gauges	3 vacuum/pressure ranges

Applications

Ambient Air - US EPA TO-14A, TO-15, ASTM D5466
Indoor Air
Vapor Intrusion
Emergency Response

SilcoCan® canisters effectively store very low levels of sulfur compounds.



Quickly confirm vacuum or pressure.
Request a high-quality gauge mounted
on your SilcoCan® or TO-Can® canister.

also available

We also offer sampling kits, sampling bags, and a range of gas reference standards to meet your environmental gas sampling requirements.
See www.restek.com/air

Can Volume	Dimensions (height x sphere diameter)		Weight	
1 liter	8.5 x 5.25"	21.6 x 13.3cm	2.5 lbs	1.13kg
3 liter	11.5 x 7.25"	29.2 x 18.4cm	4 lbs	1.81kg
6 liter	12.5 x 9.25"	31.8 x 23.5cm	7 lbs	3.18kg
15 liter	17 x 12.25"	43.2 x 31.1cm	13 lbs*	5.90kg

*16 lbs shipped UPS Air, 22 lbs shipped Fed Ex (USA).

SilcoCan® Air Monitoring Canisters

Description	qty.	Restek Exclusive! 1L Volume	3L Volume	6L Volume	15L Volume
		cat.#	cat.#	cat.#	cat.#
SilcoCan Canister, 1/4" Valve	ea.	24180	24181	24182	24183
SilcoCan Canister, Siltek Treated 1/4" Valve	ea.	24180-650	24181-650	24182-650	24183-650
SilcoCan Canister with Gauge, 1/4" Valve	ea.	24140	24141	24142	24143
SilcoCan Canister with Gauge**, Siltek Treated 1/4" Valve	ea.	24140-650	24141-650	24142-650	24143-650
SilcoCan Canister without Valve	ea.	22090	22091	22092	22093

TO-Can® Air Monitoring Canisters

Description	qty.	1L Volume	3L Volume	6L Volume	15L Volume
		cat.#	cat.#	cat.#	cat.#
TO-Can Canister with 1/4" Valve	ea.	24172	24173	24174	24175
TO-Can Canister with Gauge**, 1/4" Valve	ea.	24176	24177	24178	24179
TO-Can Canister without Valve	ea.	22094	22095	22096	22097
TO-Can Canister with 1/4" Swagelok SS4H Bellows-Sealed Valve	ea.	22105	22106	22107	22108

**range of standard gauge is -30" Hg to 60 psi.



See our complete line of products for

Air Monitoring

visit www.restek.com/air

Simplify Air Sampling

Canister Air Sampling Timer

- Program up to 12 timed events!
- Capable of both manual and automated operation.
- Perfect for either grab or time-integrated sampling.
- Long battery life; recharges conveniently using the USB port on any PC.
- All stainless steel sample flow path ensures inertness, improving accuracy.

Description	qty.	cat.#
Canister Air Sampling Timer	ea.	24267

Whether automated or manual, Restek's Canister Timer has the features you need for easy, reliable sampling!



Dimensions:
44"H x 48"W x 27"L
Weight: 525 lbs

	Restek	Entech
Capacity	12-6L cans	6-6L cans
Software	Included	Separate

www.restek.com/air

TO-Clean Canister Cleaning System

High capacity, fully automated, easy to use canister cleaning oven dramatically increases lab efficiency.

- Twelve 6L canister capacity; custom-built trays for different canister sizes.
- Method TO-14A/15 compliant.
- Small footprint saves lab space.
- No computer needed—uses embedded touch screen controller.
- Save up to 10 user defined methods.
- Automated system leak test.
- Isothermal oven cleans entire can AND valve more completely than band heaters.
- Includes Edwards® RV-8 vacuum pump—no turbo pumps!
- One year limited warranty.

Description	qty.	cat.#
TO-Clean Oven, 120V, 60Hz	ea.	22916
TO-Clean Oven, 220/230V, 50/60Hz	ea.	22917
Optional Accessories (not included with TO-Clean Oven)	qty.	cat.#
Dewar, glass, 4300mL stainless steel u-tube trap	ea.	22918
Oven Cart, 29"H x 27"W x 49"D, 12 gauge steel, push handle and casters	ea.	22919
1L Option: includes tubing, fittings, and inserts for 24 1L canisters	ea.	22920
Humidification Chamber	ea.	24282

Shipping: FedEx Ground, unless otherwise requested. Costs vary depending on ship-to location.

Note: Ovens are built on demand, therefore, a ten week lead time is required on all orders. A limited cancellation and return policy applies to TO-Clean ovens; contact Restek Customer Service for details. Not available in countries requiring CE certification (Europe & Japan).



Air Canister Tripod conveniently holds 2 air canisters.

Air Canister Tripod

- Lightweight (9 pounds) and compact, for easy storage and transport.
- Extends from 6' to 9' high.
- Large base provides enhanced stability, without additional supports.
- Sturdy, rugged metal design, for outdoor sampling and transport.


Restek's Air Canister Tripod holds two canisters simultaneously for collocated ambient air sampling. The custom-designed bracket holds most 1 L, 3 L, and 6 L canisters securely, without any tools.*

Description	qty.	cat.#
Air Canister Tripod	ea.	24151

*Air sampling canisters sold separately.

Expand Air Sampling with Mini-Cans & Accessories

- Grab and integrated sampling without sampling pump.
- 8-hr integrated sample possible with 400 cc mini-can.
- Siltek® coating delivers high level of inertness for H₂S & other reactive compounds.
- Versatile enough for many applications:
 - Indoor air - Industrial hygiene
 - Soil gas - Emergency response



Get Mini!

Mini-Can Options

Sizes	400cc, 1000cc
Valves	Quick connect, diaphragm
Interior Coating	Electropolished, Siltek treated
Sample Inlets	Area, personal
Flow ranges	0.5-15 sccm

Miniature Air Sampling Kits

- Provide accurate integrated sampling without a sampling pump.
- Convenient smaller size connects easily to miniature canisters.
- Available in stainless steel or Siltek® treated components for greater inertness.

Restek's passive air sampling kit incorporates all the hardware necessary to collect air samples, and is easy to assemble for field sampling.* Kit includes flow controller, critical orifice, 2 µm frit filter, vacuum gauge, and sample inlet. The gauge (cat.# 24120) and sample inlet (cat.#s 26211, 26212) are downsized for partnering with smaller canisters.

Miniature Air Sampling Kits

Canister		Flow (sccm)	Orifice size	Siltek Treated Sampling Kits	Stainless Steel Sampling Kits
400cc	1 Liter				
8 hour	24 hour	0.5-2	0.0008"	26253	26252
2 hour	4 hour	2-4	0.0012"	26255	26254
1 hour	2 hour	4-8	0.0016"	26257	26256
—	1 hour	8-15	0.0020"	26259	26258

*Air sampling canisters sold separately.

Miniature Air Sampling Canisters

Description	qty.	400cc cat.#	1,000cc cat.#
Miniature Canister with Quick-Connect Stem Fittings			
Electro-Polished Stainless Steel	ea.	24188	24194
Siltek Treated	ea.	24189	24195
Siltek Treated, with Siltek Treated Quick-Connect Stem Fitting	ea.	24190	24196
Miniature Canister with Metal-Seated Diaphragm			
Electro-Polished Stainless Steel	ea.	24191	24197
Siltek Treated	ea.	24192	24198
Siltek Treated, with Siltek Treated Diaphragm Valve	ea.	24193	24199



Sampling Belt &
Personal Sample Inlet



Mini-Can Stand

Mini-Can Accessories

These accessories enhance the usage of the mini-can and provide flexibility in their application, from personal to area to vapor intrusion sampling.

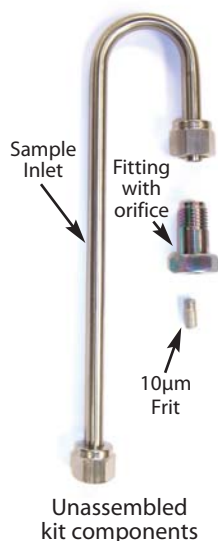
Description	qty.	cat.#
Sampling Belt	ea.	22122
Personal Sample Inlet (includes: 3" x 1/8" OD Teflon tubing, Clip, Teflon Reducing Ferrule, 1/4" SS nut)	ea.	22123
Mini-Can Stand	ea.	22124

For more information on Restek's Mini-Cans, sampling kits, and accessories, visit
www.restek.com/air



Includes:

- 1 Veriflo® SC423XL flow controller
- 2 Stainless steel vacuum gauge
- 3 1/4-inch Siltek® sample inlet
- 4 2-micron frit filter and spring washer (not visible in image)
- 5 Interchangeable critical orifice



Unassembled
kit components



Assembled
kit on canister
(canister sold separately)

Superior Performance—an Excellent Restek Value

Integrated Air Sampling Kits

- Provide accurate integrated sampling without a sampling pump.
- Inert Siltek® treated components ensure accurate sampling of active components.
- Excellent for sampling times from 0.5 hour to 125 hours.

Restek's passive air sampling kit incorporates all the hardware necessary to collect air samples, and is easy to assemble for field sampling.* The improved filter design greatly reduces the number of potential leak sites.

The passive air sampling kit is available in seven sampling flow ranges, and in stainless steel or Siltek® treated finish. The stainless steel kit is ideal to partner with the Restek TO-Can® air sampling canister for TO-14A and TO-15 methods. Use the Siltek® treated version with the Restek SilcoCan® air sampling canister when collecting low-level volatile sulfur compounds, or other active compounds.

Canister Volume*/Sampling Time					Flow	Orifice	Siltek Treated	Stainless Steel
400cc	1 Liter	3 Liter	6 Liter	15 Liter	(sccm)	size	Sampling Kits	Sampling Kits
8 hour	24 hour	48 hour	125 hour	—	0.5–2	0.0008"	24217	24216
2 hour	4 hour	12 hour	24 hour	60 hour	2–4	0.0012"	24160	24165
1 hour	2 hour	6 hour	12 hour	30 hour	4–8	0.0016"	24161	24166
—	1 hour	4 hour	8 hour	20 hour	8–15	0.0020"	24162	24167
—	—	2 hour	3 hour	8 hour	15–30	0.0030"	24163	24168
—	—	—	1.5 hour	4 hour	30–80	0.0060"	24164	24169
—	—	—	0.5 hour	1 hour	80–340	0.0090"	22101	22100

*Air sampling canisters sold separately.

Canister Grab Sampling Kit

- Use with 1, 3, or 6 L canisters, for qualitative grab air sampling.
- 1/4" compression fitting connects directly to canister valve inlet.
- Replaceable frit protects orifice and valve from particulates.
- Sample inlet design minimizes water entry into sampling train.
- Variety of orifice sizes, for fast sampling from 5 to 60 minutes.
- Individual replacement components available.

Canister Volume*/Sampling Time (min.)			Flow	Orifice Size	Siltek Treated	Stainless Steel
1 L Canister	3 L Canister	6 L Canister	(mL/min.)		Grab Sampling Kits	Grab Sampling Kits
					cat.#	cat.#
60	—	—	15	0.0018"	26280	26263
30	—	—	20	0.0020"	26281	26264
15	60	—	45	0.0030"	26282	26265
—	30	60	80	0.0040"	26283	26266
5	15	30	150	0.0055"	26284	26267
—	—	15	300	0.0080"	26285	26268
—	5	—	390	0.0090"	26286	26269
—	—	5	>1,000	0.0130"	26287	26270

*Air sampling canisters sold separately.

Replacement Fittings for Grab Sampling Kits

Orifice Size	Siltek Treated	Stainless Steel
	Replacement Fitting w/Orifice	Replacement Fitting w/Orifice
	cat.#	cat.#
0.0018"	26288	26271
0.0020"	26289	26272
0.0030"	26290	26273
0.0040"	26291	26274
0.0055"	26292	26275
0.0080"	26293	26276
0.0090"	26294	26277
0.0130"	26295	26278

Environmental Air Monitoring Gas Standards

TO-14A Internal Standard/Tuning Mix

bromochloromethane
1-bromo-4-fluorobenzene
(4-bromofluorobenzene)

chlorobenzene-d5
1,4-difluorobenzene

1ppm in nitrogen, 104 liters @ 1,800psi

cat. # 34408 (ea.)

100ppb in nitrogen, 104 liters @ 1,800psi

cat. # 34425 (ea.)

1ppm in nitrogen, 110 liters @ 1,800psi (Pi-marked Cylinder)

cat. # 34408-PI (ea.)

100ppb in nitrogen, 110 liters @ 1,800psi (Pi-marked Cylinder)

cat. # 34425-PI (ea.)

TO-15 Subset 25 Component Mix (25 components)

acetone
allyl chloride
benzyl chloride*
bromodichloromethane
bromoform
1,3-butadiene
2-butanone (MEK)
carbon disulfide*
cyclohexane
dibromochloromethane
trans-1,2-dichloroethene
1,4-dioxane
ethyl acetate

4-ethyltoluene
heptane
hexane
2-hexanone (MBK)
4-methyl-2-pentanone
methyl *tert*-butyl ether (MTBE)
2-propanol
propylene
tetrahydrofuran
2,2,4-trimethylpentane
vinyl acetate
vinyl bromide

1ppm in nitrogen, 104 liters @ 1,800psi

cat. # 34434 (ea.)

100ppb in nitrogen, 104 liters @ 1,800psi

cat. # 34435 (ea.)

1ppm in nitrogen, 110 liters @ 1,800psi (Pi-marked Cylinder)

cat. # 34434-PI (ea.)

100ppb in nitrogen, 110 liters @ 1,800psi (Pi-marked Cylinder)

cat. # 34435-PI (ea.)

*Stability of this compound cannot be guaranteed.

Massachusetts APH Mix (26 components)

benzene
1,3-butadiene
butylcyclohexane
cyclohexane
n-decane
2,3-dimethylheptane
2,3-dimethylpentane
n-dodecane
ethylbenzene
n-heptane
n-hexane
isopentane
isopropylbenzene

p-isopropyltoluene
methyl *tert*-butyl ether
1-methyl-3-ethylbenzene
naphthalene
n-nonane
n-octane
toluene
1,2,3-trimethylbenzene
1,3,5-trimethylbenzene
n-undecane
o-xylene
m/p-xylene (combined)

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1ppm in nitrogen, 21 liters @ 350psig (Pi-marked Cylinder)

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carbon disulfide*
carbon tetrachloride
chlorobenzene
chloroethane
chloroform
chloromethane
cyclohexane
dibromochloromethane
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethene
cis-1,2-dichloroethene
trans-1,2-dichloroethene

1,2-dichloropropane
cis-1,3-dichloropropene
trans-1,3-dichloropropene
1,4-dioxane
ethanol*
ethyl acetate
ethyl benzene
ethylene dibromide
(1,2-dibromoethane)
4-ethyltoluene
trichlorofluoromethane
(Freon 11)
dichlorodifluoromethane
(Freon 12)
1,1,2-trichloro-
1,2,2-trifluoroethane
(Freon 113)
1,2-dichlorotetra-
fluoroethane
(Freon 114)
heptane
hexachloro-1,3-butadiene
hexane
2-hexanone (MBK)

4-methyl-2-pentanone
(MIBK)
methylene chloride
methyl *tert*-butyl ether
(MTBE)
methyl methacrylate
naphthalene
2-propanol
propylene
styrene
1,1,2,2-tetrachloroethane
tetrachloroethene
tetrahydrofuran
toluene
1,2,4-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethene
1,2,4-trimethylbenzene
1,3,5-trimethylbenzene
vinyl acetate
vinyl chloride
m-xylene
o-xylene
p-xylene

1ppm in nitrogen, 104 liters @ 1,800psi

cat. # 34436 (ea.)

100ppb in nitrogen, 104 liters @ 1,800psi

cat. # 34437 (ea.)

1ppm in nitrogen, 110 liters @ 1,800psi (Pi-marked Cylinder)

cat. # 34436-PI (ea.)

100ppb in nitrogen, 110 liters @ 1,800psi (Pi-marked Cylinder)

cat. # 34437-PI (ea.)

*Stability of this compound cannot be guaranteed.

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Analyte	wide range of VOCs	wide range of VOCs & permanent gases	sorbent specific
Applications	ambient, IAQ, emergency response, IH	ambient, IAQ emission	IAQ, IH
Durability	reusable	one time use	one time use
Inertness	excellent	fair	fair
Stability	30 day	48 hrs	varies by analyte
Sample Volume	0.4–6 L	0.5–100 L	varies by analyte
Sampling Time	minutes to days	minutes to hours	minutes to hours

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Guide to Preparing and Analyzing Semivolatile Organic Compounds



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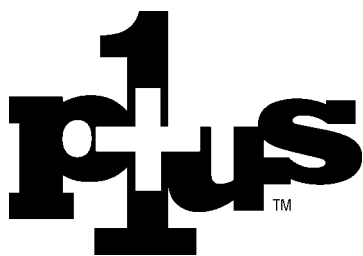
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Introduction

This technical guide addresses the preparation and gas chromatographic (GC) analysis of semivolatile organic compounds such as those listed in US Environmental Protection Agency (EPA) Methods 8270, 525, and 625; and polycyclic aromatic hydrocarbons (PAHs) such as those listed in US EPA Methods 610 and 8100. These analyses are some of the most common tests performed by environmental laboratories, yet there are many analytical challenges of which the analyst needs to be aware. For example, the samples often are highly contaminated with non-target compounds (e.g., hydrocarbons) and quality assurance/control (QA/QC) of the methods is rigorous. There are several procedures and techniques that can be employed, however, to make these analyses simpler to perform. Review this guide to learn these techniques and to troubleshoot analytical problems associated with the methods.

The compounds addressed in this guide are listed in Table I, but many additional compounds are also amenable to these semivolatile methods. Table I includes the compounds cited in the US EPA Methods, as well as some other compounds typically analyzed in environmental samples.

Extraction

The compounds listed in Table I may be difficult to extract because they fall into different chemical classes (i.e., acidic, basic, neutral, halogenated, oxygenated, polar, non-polar, low-boiling, and high-boiling compounds). Therefore, the extraction method will need to solvate a wide variety of compounds. It also must recover the analytes of interest while removing the interfering non-target contaminants. This limits the choices of cleanup options. A number of sample extraction methods can be applied to these compounds, but only the most common will be addressed in this guide.

Liquid Samples

For liquid samples, either separatory funnel extraction (US EPA Method 3510) or automated liquid-liquid extraction (US EPA Method 3520) may be used. Separatory funnel extraction is faster and less expensive to set up than the other methods, but it requires continuous operator attention. Automated liquid-liquid extractors run unattended, but are more expensive and, if analyte recovery is lower than allowed, re-extraction by separatory funnel may be required. Alternatively, if the sample forms an emulsion to the degree that acceptable solvent recovery is not possible using a separatory funnel, then some samples will require automated liquid-liquid extraction. Solid phase extraction (US EPA Method 3535) also is an option for aqueous samples.

For **separatory funnel extraction**, measure up to 1L of water into a 2L separatory funnel and adjust the pH to >11 using 10M NaOH; be careful not to add too much base. Then extract the sample by adding 60mL of dichloromethane and shaking for two minutes. It is critical to shake all samples consistently or variations in extraction efficiency will be observed. The best way to ensure consistency is to use a mechanical separatory funnel shaker, as there often is considerable variation with manual extractions. Allow the dichloromethane layer to settle to the bottom of the funnel and then decant that layer into a collection vessel (i.e., a Kurdena Danish [KD] concentrator, or a Turbo vap or Rapid vap® container if using automated concentrators). This extraction step is repeated twice more to get quantitative recovery of all analytes. Collect all three extractions into the same collection vessel and label as base/neutral.

Then adjust the water sample to a pH of slightly less than 2 using sulfuric acid (1:1, v/v). Avoid over-acidification because it can result in an acidic extract. Repeat extraction procedure on the water sample as described above, collecting extracts in a separate collection vessel and labeling it as acid fraction.

It is critical to remove water from the dichloromethane before you concentrate the extract to final volume. Dichloromethane can hold approximately 11mL of water per liter of dichloromethane. If this water remains in the extract, it will partition out when the volume is reduced. This will result in the dichloromethane boiling off first, leaving water in the collection vessel, and the formation of a two-layer extract. The analyte recoveries will be

Table I. Semivolatile organic compounds listed in US EPA Methods 8270, 525, and 625.

Peak Number	Compound	Peak Number	Compound
1	2-fluorophenol	50	fluorene
2	phenol-d6	51	4-chlorophenyl phenyl ether
3	phenol	52	4-nitroaniline
4	bis(2-chloroethyl) ether	53	4,6-dinitro-2-methylphenol
5	2-chlorophenol-d4	54	N-nitrosodiphenylamine
6	2-chlorophenol	55	2,4,6-tribromophenol
7	1,3-dichlorobenzene	56	4-bromophenyl phenyl ether
8	1,4-dichlorobenzene-d4 (ISTD)	57	α -HCH
9	1,4-dichlorobenzene	58	hexachlorobenzene
10	benzyl alcohol	59	β -HCH
11	1,2-dichlorobenzene-d4	60	pentachlorophenol
12	1,2-dichlorobenzene	61	γ -HCH (Lindane)
13	2-methylphenol (o-cresol)	62	phenanthrene-d10 (ISTD)
14	2,2'-oxybis-(1-chloropropane)	63	phenanthrene
15	4-methylphenol (p-cresol)	64	anthracene
16	N-nitrosodi- <i>n</i> -propylamine	65	δ -HCH
17	hexachloroethane	66	carbazole
18	nitrobenzene-d5	67	heptachlor
19	nitrobenzene	68	di- <i>n</i> -butyl phthalate
20	isophorone	69	aldrin
21	2-nitrophenol	70	heptachlor epoxide
22	2,4-dimethylphenol	71	fluoranthene
23	bis(2-chloroethoxy)methane	72	endosulfan I
24	benzoic acid	73	pyrene
25	2,4-dichlorophenol	74	4,4'-DDE
26	1,2,4-trichlorobenzene	75	<i>p</i> -terphenyl-d14
27	naphthalene-d8 (ISTD)	76	dieldrin
28	naphthalene	77	endrin
29	4-chloroaniline	78	endosulfan II
30	hexachlorobutadiene	79	4,4'-DDD
31	4-chloro-3-methylphenol	80	endrin aldehyde
32	2-methylnaphthalene	81	butyl benzyl phthalate
33	hexachlorocyclopentadiene	82	4,4'-DDT
34	2,4,6-trichlorophenol	83	endosulfan sulfate
35	2,4,5-trichlorophenol	84	endrin ketone
36	2-fluorobiphenyl	85	methoxychlor
37	2-chloronaphthalene	86	3,3'-dichlorobenzidine
38	2-nitroaniline	87	benzo(a)anthracene
39	dimethyl phthalate	88	chrysene-d12 (ISTD)
40	2,6-dinitrotoluene	89	chrysene
41	acenaphthylene	90	bis(2-ethylhexyl)phthalate
42	3-nitroaniline	91	di- <i>n</i> -octyl phthalate
43	acenaphthene-d10 (ISTD)	92	benzo(b)fluoranthene
44	acenaphthene	93	benzo(k)fluoranthene
45	2,4-dinitrophenol	94	benzo(a)pyrene
46	4-nitrophenol	95	perylene-d12 (ISTD)
47	dibenzofuran	96	ideno(1,2,3-cd)pyrene
48	2,4-dinitrotoluene	97	dibenz(a,h)anthracene
49	diethyl phthalate	98	benzo(g,h,i)perylene

The optimum way to remove the water is to decant the dichloromethane through granular sodium sulfate, which is held in a funnel with a high-quality grade filter paper (e.g., Whatman® 541). Approximately 30g of sodium sulfate are sufficient for most samples. This step must not be skipped. Methods may call for powdered sodium sulfate, but some analytes can be adsorbed onto the smaller particles so it is recommended that only 10-60 mesh or similar granular sodium sulfate be used. Also, it is important that this material be contaminant-free, so it should be purchased as American Chemical Society (ACS) pesticide residue-grade in glass containers or baked in a muffle furnace if purchased in bulk packages where exposure to plastic is an issue (see Restek Tip). If a muffle furnace is not available, the sodium sulfate can be washed or extracted with dichloromethane prior to use; however this technique uses large amounts of solvent.

**Restek
Tip**

How to Bake Sodium Sulfate

To bake the sodium sulfate, spread it into a glass pie plate no more than 1" thick and place into a muffle furnace at 400°C for a minimum of two hours. After this time, the sodium sulfate should be placed into a glass container while still hot and sealed with a Teflon®-lined cap to prevent the material from readsorbing contaminants from the atmosphere.



Restek Tip

Clean Glassware

It is important to properly clean glassware used during sample extraction. Contaminated glass surfaces can react with samples and cause breakdown or adsorption of active compounds. Verify cleanliness by running blanks through all glassware.

Automated liquid-liquid extraction can run unattended once the samples are ready and the solvent is added. This extraction is performed at a single pH. Generally, you will need to adjust the sample pH to 2, but some methods call for adjusting the pH to 4. In any event, it is critical to not let the pH go below 2 when using a liquid-liquid extractor. If this happens, an acidic extract will form and may cause damage to the GC column. Acidic extracts also will cause low recoveries for the late-eluting internal standards, possibly due to isotope exchange (e.g., perylene-d12).

Automated liquid-liquid extractors are available in two versions—conventional and accelerated. The conventional types use 1L of sample and extract using 100 to 500mL of dichloromethane. These extraction vessels typically are operated for 16 to 24 hours in order to achieve complete extraction. The accelerated extractor uses a hydrophobic membrane to separate the aqueous from the organic phases, and the extraction time can be reduced by 25 to 30% compared to the conventional extractor. However, the membranes are expensive, so it is important to analyze the cost versus the number of samples extracted to determine if there is a cost benefit to using this accelerated technique.

Finally, **solid phase extraction (SPE)** also may be used to extract semivolatile organic compounds from aqueous samples. When using SPE, it is extremely important to follow the manufacturer's recommendations for product use. There are several manufacturers of C18 cartridges and disks, which are the typical media for these compounds. The specific steps to extract these compounds will vary somewhat depending on the manufacturer. One of the biggest problems with SPE is plugging of the disk or tube with suspended solids, so this method only works reliably for drinking water samples. If contamination levels are low and the samples are free of solids, SPE provides very fast extraction times and low solvent usage. It is used easily for field extractions. And, generally, the disks are preferred for the extraction of 1L sample volumes, but recoveries are not uniform for all of the compounds in Table I. The compounds listed in US EPA Method 525.2 exhibit good extraction recoveries using this technique. For detailed information on this extraction, request the Applications Note "*SPE Extraction for US EPA Method 525.1*" (lit. cat.# 59557).

Soil Samples

Soxhlet and ultrasonic extraction are the most common extraction techniques for solid samples; although pressurized fluid, microwave, and supercritical fluid extraction (SFE) also can be used.

Because the soil and biota samples essentially are wet particles, acetone and dichloromethane (1:1) usually are used as the extraction solvents. Acetone is needed to adequately penetrate the soil particle so that compounds in the particle can be extracted. Several other solvent systems are used for more specialized extractions, but for most applications this combination works well.

All solvents used for extractions must be ACS pesticide-residue grade, and a solvent assay should be performed to verify purity prior to use. To perform a solvent assay, evaporate 300 to 400mL of solvent to a final volume of 1mL and analyze by GC/mass spectrometry (MS). The resulting chromatogram should have no compounds quantitated above $1/2$ the detection limit for any target compound.

Soxhlet and ultrasonic extraction work well for the semivolatile compounds listed in Table I. Sonication is a faster technique but requires constant operator attention. In both techniques, problems usually are caused by contaminated reagents (especially sodium sulfate) or by inconsistent handling from sample to sample. Sodium sulfate must be treated to remove water as described in the Restek Tip on page 3, and the sample must be mixed with the sodium sulfate to achieve a sandy consistency.

Pressurized fluid extraction (US EPA Method 3545A) can be run in an unattended fashion with multiple samples across a wide sample size range. Extraction vessels with volumes of 1 to 100 mL are available. Instruments like the Dionex ASE 200 accommodate wet samples from 1 to 15 grams, and the Dionex ASE 300 will accommodate wet samples from 15 to 50 grams. The volume of the cell needed for wet samples is generally twice the gram weight of the sample being used. For example, if 30-g wet samples are needed, the 66-mL and 100-mL vessels will be adequate for these extractions. This is necessary because a drying agent such

as diatomaceous earth is added to the sample prior to being loaded into the extraction vessels. The type of samples being extracted as well as the required method detection limits should be considered as part of the evaluation of pressurized fluid extraction.

Microwave extraction (US EPA Method 3546) can be useful for automated extraction as well. This method typically performs the extraction of 12 samples simultaneously, but requires slightly more operator handling than the pressurized fluid extraction instruments. Microwave extraction instrumentation is less expensive, but can suffer from the same sample size limitations.

Supercritical fluid extraction (SFE) has been promoted for a number of years as a means of “solventless” extraction for environmental samples. SFE has been added to SW-846 as Methods 3560, 3561, and 3562 but its application is limited. SFE suffers from severe matrix-related variation, requiring modification of its conditions depending on soil type, water content, sample size, and type of analytes. Doing so ultimately requires additional sample preparation prior to the actual extraction. These requirements, added to the high cost of the instrument, have virtually precluded the use of SFE for environmental sample preparation.

Cleanup

Sample extract cleanup may be the most important step in maintaining long-term instrument performance. Many times, when instrument problems arise, they are caused by exposure of the injection port and the column to material in the sample extracts other than the target compounds. While all contaminants cannot be eliminated, reducing them will minimize injection port and column maintenance. Most semivolatile extracts, especially those extracts from soil and biota samples, contain high-boiling hydrocarbons and lipids. The difficulty in attempting to remove these compounds using one of the common solid-liquid cleanup techniques (e.g., Florisil® and silica gel) is that the cleanup technique also removes some of the target compounds. In addition, because the analytical method usually calls for the reporting of several tentatively identified compounds (TICs), it is not desirable to clean the extracts of compounds that would normally elute in the range of the target compounds. For these reasons, gel permeation chromatography (GPC) is the only universal cleanup technique for semivolatile extracts.

Gel Permeation Chromatography

Gel permeation chromatography (GPC) is a preparative scale chromatographic method of separation based on molecular size. Because the target compounds are similar in molecular size, they elute as a band of material and are easily separated from lighter and heavier contaminants. However, GPC systems are expensive and the processing time per sample is between 30 to 70 minutes. For these reasons many laboratories choose not to use GPC. However, it is very efficient for removing sulfur, high molecular weight hydrocarbons, and lipids from semivolatile extracts; and may be prudent for soil and biota samples.

Although sulfur can be removed using other techniques such as mercury or activated copper powder, these procedures, especially copper powder, may degrade some of the target compounds and will not remove the high-boiling hydrocarbons or lipids. The lipid content of biota extracts can be significant and may overload most SPE clean-up techniques. If a sample extract with a high lipid content is injected into the GC, the injection port and front of the column will become contaminated quickly. This will result in failure of check standards and the loss of active compounds such as nitroanilines, nitrophenols, carbazole, and pentachlorophenol (PCP). In spite of the added expense and time required for GPC, it is the best alternative for extract cleanup.

US EPA Method 3640 details the requirements for GPC cleanup of extracts for semivolatile analysis. One of the important steps of GPC cleanup is to ensure each day that the instrument is within its retention time calibration. Although not required by the method, it is good practice to run a daily calibration check standard before processing the next batch of samples. If a number of samples have been processed that contain large amounts of contamination, the front of the GPC column can become reactive. This typically is observed in the loss of 2,4,6-tribromophenol for semivolatile extracts. If the column becomes reactive, injecting blanks may return the system to control and save the time required to change the column.

**Restek
Tip**

Stabilizing Dichloromethane

Dichloromethane requires a stabilizer to prevent the formation of hydrochloric acid (HCl). Without a stabilizer, HCl will form and injection of acidic dichloromethane will cause inlet liners and columns to become reactive. There are two types of stabilizers: stabilizers that keep HCl from forming, and stabilizers that eliminate HCl upon formation. Methanol is a stabilizer that prevents HCl from forming; whereas cyclohexane, cyclohexene, 2-methylbutene, and amylene scavenge the HCl after its formation.

Dichloromethane used in liquid extractors should contain both types of stabilizers. Methanol is a better stabilizer, acting as a free radical inhibitor, but methanol partitions into the water phase. This could leave an unstabilized extract unless a scavenger stabilizer also is used.

Obtaining consistent GPC results begins with the extraction and concentration procedures because slight changes in mobile phase and sample solvent composition can result in some target compounds being uncollected. Because the typical sample solvent for GPC is pure dichloromethane, it is critical that all extracts be reduced to as small a volume as possible before reconstitution in dichloromethane to avoid large amounts of acetone being applied to the column. Soil and biota samples typically are extracted with a solvent mixture of acetone and dichloromethane. It is critical that all extracts be reduced to as small a volume as possible before reconstitution in dichloromethane to avoid large amounts of acetone being applied to the column. Dichloromethane has a lower boiling point than acetone, so it will evaporate first during sample concentration, which will leave nearly 100% acetone in the concentration vessel. If dichloromethane is then added to adjust the extract to volume, there will be significant amounts of acetone introduced to the GPC column. This will lead to “solvent shock” and the formation of a void will be observed at the front of the column. This, in turn, will affect the retention times of the compounds eluting from the GPC column and ultimately will result in some target compounds being uncollected. Table II lists the common semivolatile compound elution volumes using GPC.

Analysis

Calibration Standards

Calibration standards are purchased as mixtures and usually are divided among three to seven separate ampuls due to the cross-reactivity of several compounds. It is important when making the actual working standard that the solution be stored under refrigerated conditions in a Mininert™ vial (Restek cat.# 21050 and 21051) due to the volatility of some of the compounds. Failure to properly store the calibration standards will result in evaporative loss of the early-eluting compounds and the solvent. This will, in effect, concentrate the late-eluting compounds and cause continuing calibration failure and quantitation errors. Even when stored under the correct conditions, there still will be degradation of some compounds due to cross-reactivity. This is observed as a loss of the target compound and commonly occurs with benzidine, 3,3'-dichlorobenzidine, 4-chloroaniline, N-nitrosodiphenylamine, and to a lesser extent with the phenols and other anilines. These standards are stable in the separate ampuls supplied from the manufacturer, but problems arise when all of the compounds are mixed together to make the working calibration standard. Therefore, it is important to monitor the response of the more active compounds and make fresh mixtures when the calibration standards degrade.

Restek Tip

Mixing Calibration Standards

When blending several ampuls to produce a calibration standard, it is important that all the compounds are completely dissolved in the solvent. This is particularly important with some of the high molecular weight polycyclic aromatic hydrocarbons (PAHs) and pesticides that can separate from solution during refrigerated storage. Before opening ampuls containing semivolatile compounds, allow them to warm to room temperature. Some mixtures may require sonication to ensure complete solubility. Follow the manufacturer's recommendations for proper handling of the standard mixture. Because some semivolatile compounds are light sensitive, it is recommended that calibration standards be stored in amber vials.

Injection Port Configuration

Several of the compounds listed in Table I are prone to breakdown or adsorption on active surfaces. Typically this will occur in the injection port; therefore, careful attention must be given to the configuration and maintenance of the injection system.

On-column injection techniques can eliminate breakdown or adsorption in the injection system and improve chromatographic analysis for drinking water extracts or extracts with little or no non-volatile residues. However, we do not recommend on-column injections for soil and biota extracts or extracts that contain large amounts of non-volatile residue, because the analytical column can be contaminated quickly.

The preferred injection technique for analyzing highly contaminated extracts is **direct injection**, but direct injection can cause solvent peak tailing and result in some of the target compounds eluting close to the solvent peak.

To reduce solvent peak tailing, **splitless injection** is most commonly used for GC/MS analysis of semivolatile compounds. There are some drawbacks to splitless injection including molecular weight discrimination, incomplete sample transfer, and reactivity. These problems can be minimized if the technique is properly optimized. Splitless injection requires an injection system that is equipped with a solenoid valve controlling the flow to a split vent. The solenoid valve is closed during the injection process, so the majority of the vaporized sample moves to the front of

Table II. Mobile phase volumes for elution of semivolatile compounds by GPC.

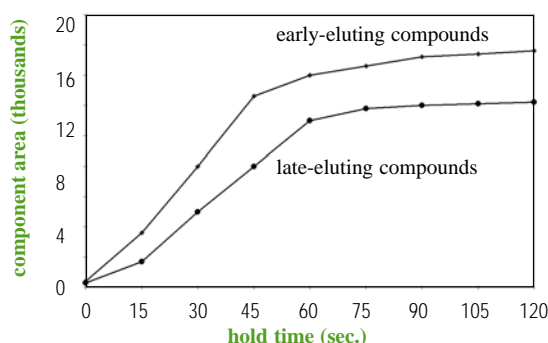
Compound	Elution Volumes (mL)		Compound	Elution Volumes (mL)	
	Start	End		Start	End
2-fluorophenol	201	241	acenaphthene-d10	225	249
phenol-d5	201	249	3-nitroaniline	209	225
phenol	209	225	acenaphthene	225	249
bis(2-chloroethyl)ether	201	225	2,4-dinitrophenol	201	225
2-chlorophenol-d4	209	249	dibenzofuran	225	249
2-chlorophenol	209	241	4-nitrophenol	201	217
1,3-dichlorobenzene	225	257	2,4-dinitrotoluene	201	225
1,4-dichlorobenzene-d4	225	257	diethylphthalate	186	209
1,4-dichlorobenzene	225	257	fluorene	225	241
1,2,-dichlorobenzene-d4	225	257	4-chlorophenyl-phenylether	217	241
1,2-dichlorobenzene	225	257	4-nitroaniline	201	225
2-methylphenol	201	241	4,6-dinitro-2-methylphenol	201	225
2,2'-oxybis(1-chloropropane)	201	225	N-nitrosodiphenylamine	201	233
4-methylphenol	201	241	2,4,6-tribromophenol	217	249
N-nitroso-di-n-propylamine	186	217	4-bromophenyl-phenylether	217	241
hexachloroethane	233	257	hexachlorobenzene	233	257
nitrobenzene-d5	209	233	pentachlorophenol	209	249
nitrobenzene	209	233	phenanthrene-d10	225	257
isophorone	193	217	phenanthrene	225	257
2-nitrophenol	217	233	anthracene	225	257
2,4-dimethylphenol	201	225	carbazole	225	257
bis(2-chloroethoxy)methane	193	225	di-n-butylphthalate	178	201
2,4-dichlorophenol	201	241	fluoranthene	225	257
1,2,4-trichlorobenzene	225	257	pyrene	225	257
naphthalene-d8	225	249	terphenyl-d14	217	233
naphthalene	225	249	butylbenzylphthalate	178	201
4-chloroaniline	217	241	benzo(a)anthracene	225	257
hexachlorobutadiene	225	249	3,3'-dichlorobenzidine	209	241
4-chloro-3-methylphenol	201	225	chrysene-d12	225	257
2-methylnaphthalene	225	249	chrysene	225	257
hexachlorocyclopentadiene	225	241	bis(2-ethylhexyl)phthalate	162	186
2,4,6-trichlorophenol	201	249	di-n-octylphthalate	162	186
2,4,5-trichlorophenol	201	249	benzo(b)fluoranthene	225	265
2-fluorobiphenyl	217	241	benzo(k)fluoranthene	225	265
2-chloronaphthalene	233	249	benzo(a)pyrene	225	265
2-nitroaniline	209	233	perylene-d12	249	273
dimethylphthalate	193	217	indeno(1,2,3-cd)pyrene	241	273
acenaphthylene	225	257	dibenz(a,h)anthracene	225	257
2,6-dinitrotoluene	193	225	benzo(g,h,i)perylene	233	273

Standard prepared and loaded as 1/4 acetone, dichloromethane; 5mL sample loop;
Column: 70g SX-3 silica size exclusion packing; **Guard column:** 5g same packing;
Flow rate: dichloromethane at 5.3mL/min. at 13 psi; Samples analyzed by GC/MS.
 (US EPA 8270)

the column. After a short time the solenoid valve is opened to allow excess solvent vapor to exit the split vent. The process of transferring the sample onto the column is relatively slow during splitless injection, so the sample must recondense at the front of the column through solvent or analyte focusing. This is accomplished by having the starting oven temperature 20°C lower than the boiling point of the solvent or the first eluting compound.

The time period that the solenoid valve is closed is referred to as the splitless hold-time. The hold-time must be optimized to obtain the best performance from the analytical system. If the solenoid valve is opened too quickly, some of the sample will be lost causing reduced response. If the solenoid valve is open too long, the solvent peak will tail. The splitless hold-time will vary depending on column flow rate, injection port geometry, injection port temperature, and volatility of the analytes. It is impossible to predict the optimum splitless hold-time without performing some experimentation under the exact conditions of your analysis.

Figure 1. Optimizing splitless hold time.



To optimize the splitless hold time for a particular instrument, prepare a standard that contains both an early- and a late-eluting compound (e.g., fluorophenol and benzo(g,h,i)perylene). Inject this standard over a range of splitless hold times from 0.1 to 2.0 minutes and plot the data. An example of this optimization is shown in Figure 1.

In this example the optimum splitless hold time is 60 seconds. This is the point on the graph where the response of the late-eluting compound levels off. Holding the solenoid valve closed longer will not appreciably increase the response of this compound, but will greatly increase the size of the solvent peak. Because the lower boiling compound will transfer onto the column faster, its response will level off sooner (in this example ~45 seconds). Once this data has been plotted, it is possible to observe the correct splitless hold-time (once again, the point at which the response of the late-eluting compounds levels off). The net effect of this optimization is to maximize response of late-eluting compounds while minimizing solvent tailing.

In addition to optimizing the splitless hold-time, fused-silica wool should be used in the injection port liner to improve vaporization of higher molecular weight compounds. While there are different theories regarding the placement of fused-silica wool, consistency in the amount of packing and location of the packing is most important. Restek recommends placing the plug of wool below the point that the syringe needle reaches, but above the inlet of the column. We also recommend using a gooseneck liner to minimize contact between the injected sample and the bottom of the injection port. This will help improve the response of the more reactive compounds such as 2,4-dinitrophenol, PCP, and the nitroanilines. The gooseneck liner also makes the greatest improvement in response and minimization of endrin breakdown for US EPA Method 525.

Another technique to minimize molecular weight discrimination is to perform the splitless injection under a higher column head pressure. A high inlet pressure is advantageous during injection to control the rapidly expanding vapor cloud in the inlet. By using a momentary pressure pulse for the time that the split vent line is closed, the sample vapor cloud is controlled and sample backflash into the gas lines entering and exiting the injection port is minimized. The effect of the pressure pulse is to increase the amount of analyte transferred to the column, especially the late-eluting components. This can lead to stationary phase overload, however, so it may be necessary to increase the capacity of the column when using

Any injection technique can suffer from reactivity (i.e., breakdown) and splitless injection is no exception. The splitless technique has two primary mechanisms for compound reactivity: sample backflash into the gas lines that enter and exit from the injector; and exposure of the sample extract to active sites on the wool, liner, and tip of the column. In general, the same set of compounds break down regardless of which mechanism is occurring: 2,4-dinitrophenol, PCP, 4-nitrophenol, carbazole, and 3-nitroaniline.

Daily maintenance of the injection port will help decrease this problem. Replace the inlet liner and fused silica wool plug, and the septum every day. Weekly, or more often depending on the extract contamination level, replace the inlet seal and remove a short section from the front of the column. The length of column removed will vary depending on the level of contamination in the extracts, generally 6 to 12 inches is adequate. When cutting the column and re-installing it into the injection port, be sure to make a square cut and be consistent with the installation distance. The installation distance varies by manufacturer. Refer to Table III for a list of recommended insertion distances.

Table III. Recommended installation distances.

Agilent (HP):	5-7mm from tip of ferrule
Varian 1075/1077:	5.7cm from back of nut
PerkinElmer Autosystem:	4.5 - 5.0cm from back of nut
Shimadzu 14A:	4.0cm from back of nut
Shimadzu 17A:	35mm from tip of ferrule
split:	40mm from back of nut
splitless:	64mm from back of nut

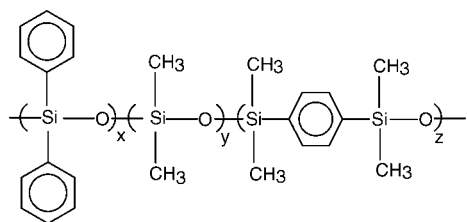
Column Selection

Due to the wide variation in functionality, volatility, and polarity of semivolatile compounds, it is not possible to select a column that is highly selective for all of them. As a result, this analysis is performed on a general-purpose stationary phase. The Rtx®-5Sil MS column has the best combination of low bleed, high inertness, and efficiency for semivolatile applications. (The Rtx®-5MS column also has been successfully used for analysis of semivolatile compounds.) The Rtx®-5Sil MS column features a silarylene phenyl/methyl phase that was developed to provide lower bleed and greater efficiency than other “5-type” phases for improved separation of the PAHs (Figure 2).

Low-bleed columns are necessary for the more sensitive instruments. For laboratories using the Agilent 5973 GC/MS or ion trap MS, column bleed can be a very important issue. As these instruments have become more sensitive, the higher-bleed columns produce a larger

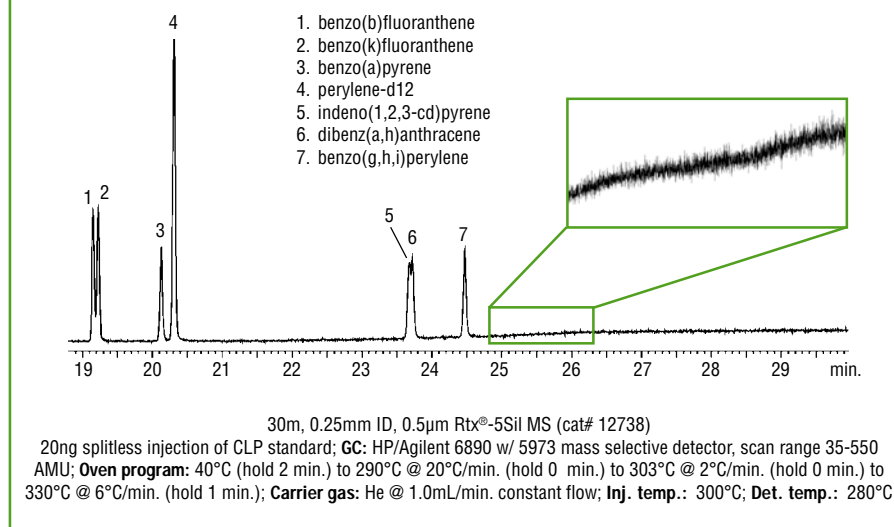
signal on the detector and can cause electron multiplier saturation. If this occurs, calibration curves may show non-linearity at higher concentrations. This is sometimes referred to as high-end roll-off, when the signal for a given concentration is lower than expected due to detector saturation. (See *Quantitation* section for more information on high-end roll-off.)

Figure 2. The Rtx®-5Sil MS column structure.



To diagnose column bleed problems, make an injection that allows the bleed to be measured relative to the concentration of the analyte in the method. Many data systems will normalize the display to the largest peak in the analysis. If no compounds are injected, the display will falsely indicate a high background. The Rtx®-5Sil MS column shows a minimal bleed level for a 20ng per component standard (Figure 3).

Figure 3. The Rtx®-5Sil MS column exhibits low bleed at 20ng concentration level.



Detector saturation also can be caused by the concentration of the analytes. It was common practice on older, less sensitive GC/MS systems to increase the multiplier voltage above the tune value to improve sensitivity of low-concentration standards. This technique can lead to problems with the newer, more sensitive instruments. It is much more likely the higher concentration calibration standards will saturate the new GC/MS systems. It may be necessary to reduce the multiplier voltage below the tune value if high-end roll-off is observed. High-end roll-off also may be observed when using pressure-pulsing injection techniques to minimize high molecular weight discrimination. If this is observed, you may either increase the stationary phase film thickness, or increase the column diameter. Alternatively, you may modify the injection conditions to eliminate the source of the overload.

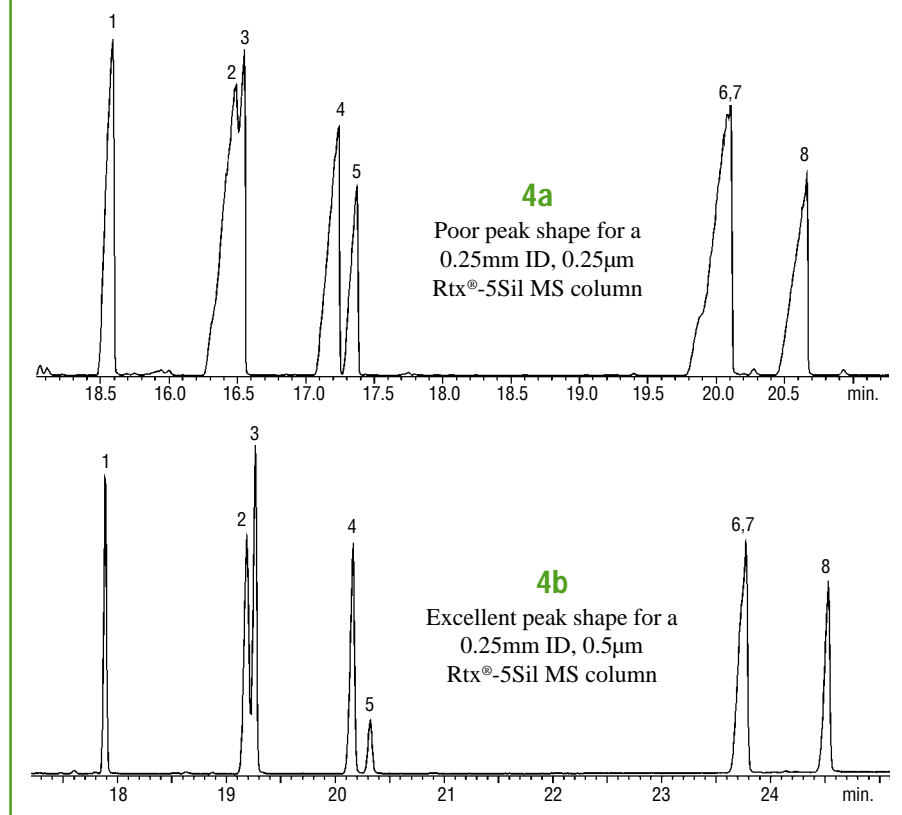
Column capacity also must be addressed when optimizing the analysis. The typical calibration range for many of these methods is 20 to 160ng per compound. This requires a column stationary phase and diameter that will not overload with a 160ng or larger injection. Because there is a loss of analyte in any splitless injection, calculation of the necessary column capacity is not simple. If the injection has been optimized for splitless hold-time and fused silica wool is being used in the liner to minimize high molecular weight discrimination, then it is easier to overload the analytical column. Possibly the biggest cause of overload is from pressure-pulsing the injection port, as this improves the transfer of all compounds to the column. The required capacity for your system will be a function of the specific calibration standards and, more importantly, the injection port.

From a capacity consideration, a 0.25mm ID column with 0.25µm film thickness does not have sufficient capacity for a 160ng per component standard. Figure 4a shows the poor peak shape observed when a column is overloaded. Increased capacity can be achieved by increasing column diameter or film thickness.

When increasing column diameter, the flow rate of the column can be a concern with bench-top GC/MS systems. Many bench-top GC/MS systems do not have the pumping capacity for the carrier gas flow that is needed with a 0.32mm ID column. A 0.28mm ID column can increase sample capacity without exceeding the pumping capacity of most bench-top GC/MS systems, making it ideal for calibrating semivolatile compounds from 20 to 160ng without overload. Alternatively, a 0.25mm ID column with a 0.5µm film thickness also has sufficient capacity to handle a calibration from 20 to 160ng without exhibiting overload. Figure 4b shows excellent peak shape for a 160ng-per-component standard on a 30m, 0.25mm ID, 0.5µm Rtx®-5Sil MS column.

The total **analysis time** should be as short as possible without sacrificing separation or resolution between compounds with similar mass spectra. Pay particular attention to the separation between benzo-b- and benzo-k-fluoranthrene—they tend to be the most difficult-

Figure 4a & 4b. Avoid overload by selecting a column with the proper capacity.



Peak List for Fig. 4a & 4b

1. di-*n*-octyl phthalate
2. benzo(b)fluoranthene
3. benzo(k)fluoranthene
4. benzo(a)pyrene
5. perylene-d12
6. indeno(1,2,3-cd)pyrene
7. dibenz(a,h)anthracene
8. benzo(ghi)perylene

Conditions for Fig. 4a

30m, 0.25mm ID, 0.5µm Rtx®-5Sil MS (cat.# 12738)
160ng splitless injection of CLP standard; GC: HP/Agilent 6890 w/ 5973 mass selective detector, scan range 35-550 AMU; **Oven program:** 40°C(hold 2 min.) to 290°C @ 20°C/min. (hold 0 min.) to 303°C @ 2°C/min. (hold 0 min.) to 330°C @ 6°C/min. (hold 1 min.); **Carrier gas:** He @ 1.0 mL/min. constant flow; **Inj. temp.:** 300°C; **Det. temp.:** 280°C

Conditions for Fig. 4b

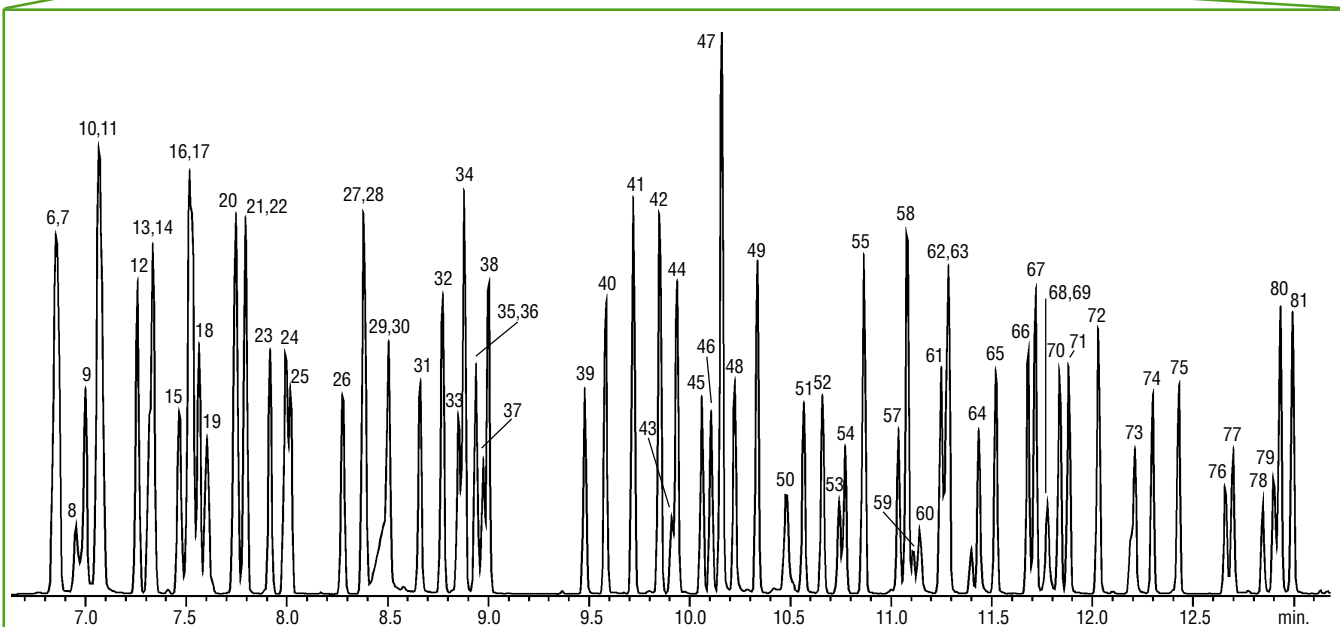
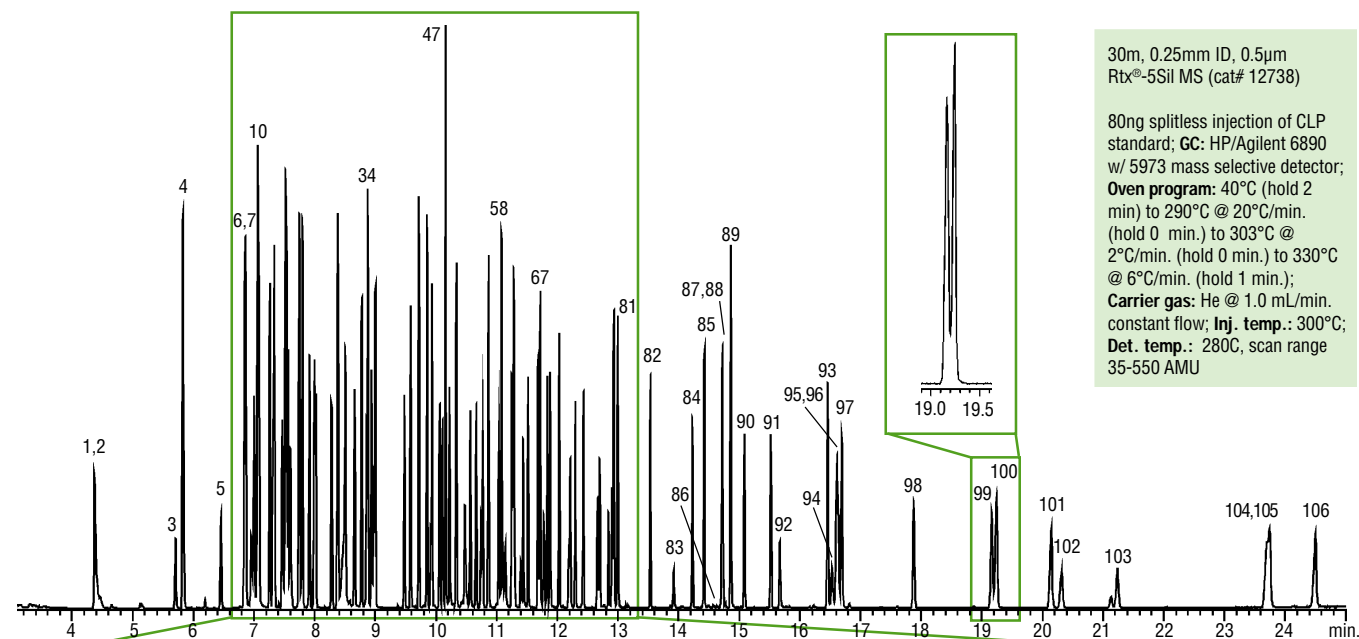
30m, 0.25mm ID, 0.25µm Rtx®-5Sil MS (cat.# 12723)
unknown concentration (>160ng) splitless injection of CLP standard; GC: HP/Agilent 6890 w/ 5973 mass selective detector, scan range 35-550 AMU; **Oven program:** 40°C (hold 2 min.) to 245°C @ 25°C/min. (hold 0 min.) to 330°C @ 6°C/min. (hold 5 min.); **Carrier gas:** He @ 1.0 mL/min. constant flow; **Inj. temp.:** 300°C; **Det. temp.:** 280°C

to-separate analytes, sharing common mass spectra and quantitation ions. Figure 5 shows a 80ng per component injection of the compounds listed in Table I with an analysis time under 30 minutes. The expanded sections of the chromatogram show the excellent resolution that can be achieved with the Rtx®-5Sil MS column.

In the past, the GC/MS systems used for semivolatile analysis did not have the sensitivity for split injections, so laboratories were limited to splitless injection. Newer systems such as the Agilent 5973 and ion trap GC/MS have greatly improved sensitivity, which allow the use of **split injection** and still meet the detection limits required by most semivolatile methods. Figure 6 shows the 20ng per component standard injected in split mode using a 20:1 split ratio on a 30m, 0.25mm ID, 0.25µm Rtx®-5Sil MS column. The low bleed exhibited by this column is critical when working with these more sensitive GC/MS systems. A benefit of split injection is narrower peak widths for improved separations between closely eluting compounds. Also, split injections usually result in less reactive compound breakdown because the residence time in the injection port is much shorter than in splitless injection.

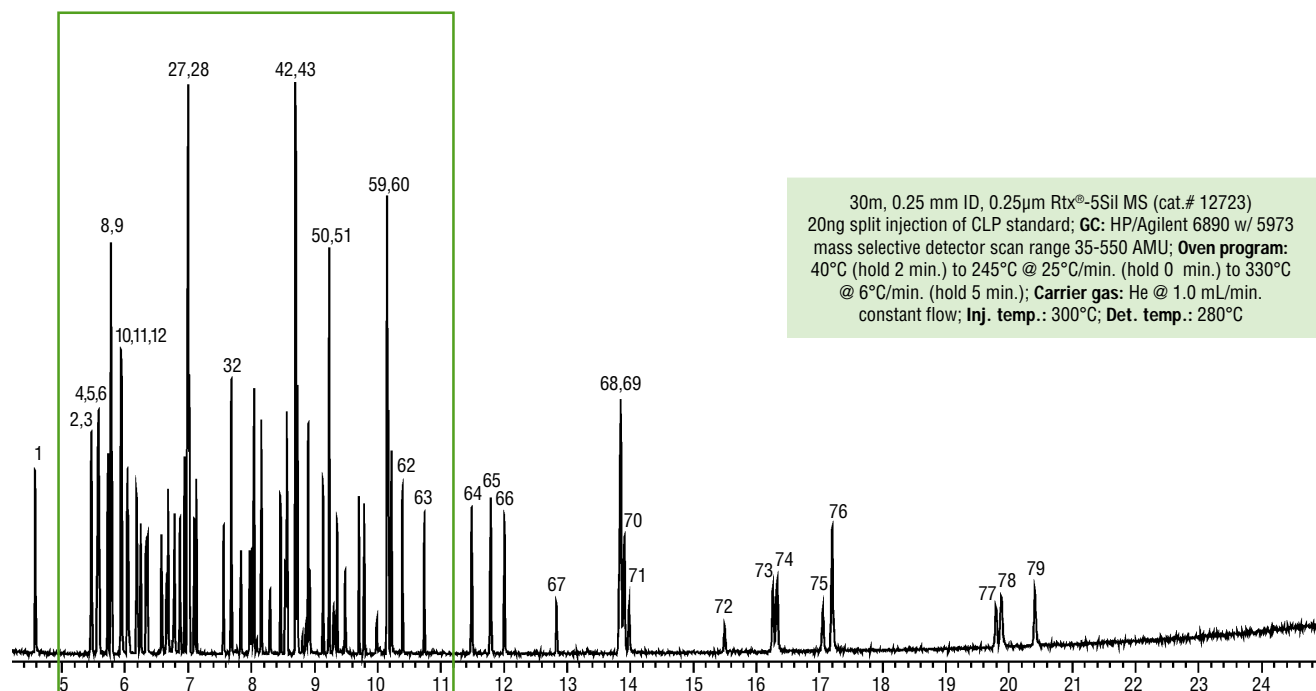
If the sensitivity of an instrument allows for split injection, then column capacity is not nearly the issue it is for splitless injection. Figure 7 shows a 160ng-per-component standard injected under the same conditions as shown in Figure 6. A column with a thinner film can be used because the concentration reaching the column is reduced by 20-fold. The analytical system using split injection will be able to handle higher concentrations of contaminants and possibly stay calibrated longer, but there will be a sacrifice in method detection limits (MDLs). Therefore, it is important to ensure that the MDLs specified in a particular method still can be met if split injection is used.

Figure 5. A 30m, 0.25mm ID, 0.5µm Rtx®-5Sil MS column offers excellent resolution of 106 compounds listed in less than 25 minutes.

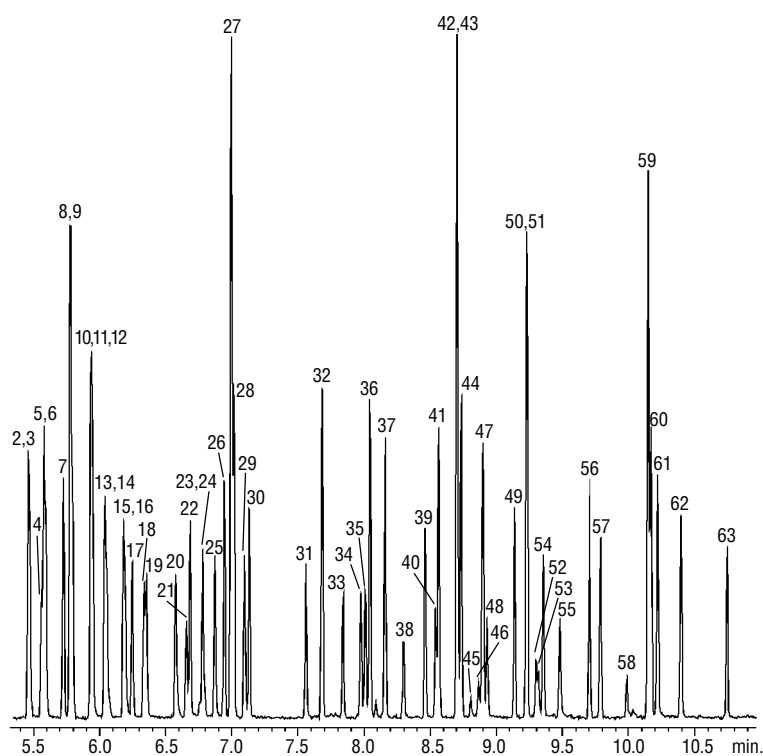


- | | | | | |
|-----------------------------------|--------------------------------|--------------------------------|---------------------------------|--------------------------------|
| 1. N-nitrosodimethylamine | 22. acetophenone | 43. hexachlorocyclopentadiene | 64. 2,3,4,6-tetrachlorophenol | 85. fluoranthene |
| 2. pyridine | 23. hexachloroethane | 44. 1,2,4,5-tetrachlorobenzene | 65. diethyl phthalate | 86. benzidine |
| 3. methyl methanesulfonate | 24. nitrobenzene-d5 | 45. 2,4,6-trichlorophenol | 66. 4-chlorophenyl phenyl ether | 87. pyrene |
| 4. 2-fluorophenol | 25. nitrobenzene | 46. 2,4,5-trichlorophenol | 67. fluorene | 88. aromite |
| 5. ethyl methanesulfonate | 26. isophorone | 47. 2-fluorobiphenyl | 68. 4-nitroaniline | 89. p-terphenyl-d14 |
| 6. phenol-d6 | 27. 2,4-dimethylphenol | 48. safrole | 69. 4,6-dinitro-2-methylphenol | 90. chlorbenzilate |
| 7. phenol | 28. 2-nitrophenol | 49. 2-chloronaphthalene | 70. diphenylamine | 91. butyl benzyl phthalate |
| 8. aniline | 29. benzoic acid | 50. 2-nitroaniline | 71. azobenzene | 92. kepone |
| 9. bis(2-chloroethyl)ether | 30. bis(2-chloroethoxy)methane | 51. 1,4-naphthoquinone | 72. 2,4,6-tribromophenol | 93. bis(2-ethylhexyl)phthalate |
| 10. 2-chlorophenol | 31. 2,4-dichlorophenol | 52. dimethylphthalate | 73. phenacetin | 94. 3,3'-dichlorobenzidine |
| 11. 2-chlorophenol | 32. 1,2,4-trichlorobenzene | 53. 1,3-dinitrobenzene | 74. 4-bromophenyl-phenyl ether | 95. benzo(a)anthracene |
| 12. 1,3-dichlorobenzene | 33. naphthalene-d8 | 54. 2,6-dinitrotoluene | 75. hexachlorobenzene | 96. chrysene-d12 |
| 13. 1,4-dichlorobenzene-d4 | 34. naphthalene | 55. acenaphthylene | 76. pentachlorophenol | 97. chrysene |
| 14. 1,4-dichlorobenzene | 35. 2,6-dichlorophenol | 56. 3-nitroaniline | 77. pentachloronitrobenzene | 98. di-n-octyl phthalate |
| 15. benzyl alcohol | 36. 4-chloroaniline | 57. acenaphthene-d10 | 78. dinoseb | 99. benzo(b)fluoranthene |
| 16. 1,2-dichlorobenzene-d4 | 37. hexachloropropene | 58. acenaphthene | 79. phenanthrene-d10 | 100. benzo(k)fluoranthene |
| 17. 1,2-dichlorobenzene | 38. hexachlorobutadiene | 59. 2,4-dinitrophenol | 80. phenanthrene | 101. benzo(a)pyrene |
| 18. 2-methylphenol | 39. 4-chloro-3-methylphenol | 60. 4-nitrophenol | 81. anthracene | 102. perylene-d12 |
| 19. bis(2-chloroisopropyl)ether | 40. isosafrole | 61. pentachlorobenzene | 82. di-n-butylphthalate | 103. 3-methylcholanthrene |
| 20. 4-methylphenol/3-methylphenol | 41. 2-methylnaphthalene | 62. 2,4-dinitrotoluene | 83. 4-nitroquinoline-1-oxide | 104. indeno(1,2,3-cd)pyrene |
| 21. N-nitroso-di-n-propylamine | 42. 1-methylnaphthalene | 63. dibenzofuran | 84. isodrin | 105. dibenz(a,h)anthracene |

Figure 6. A 20ng split injection shows the extremely low bleed of the Rtx®-5Sil MS column.



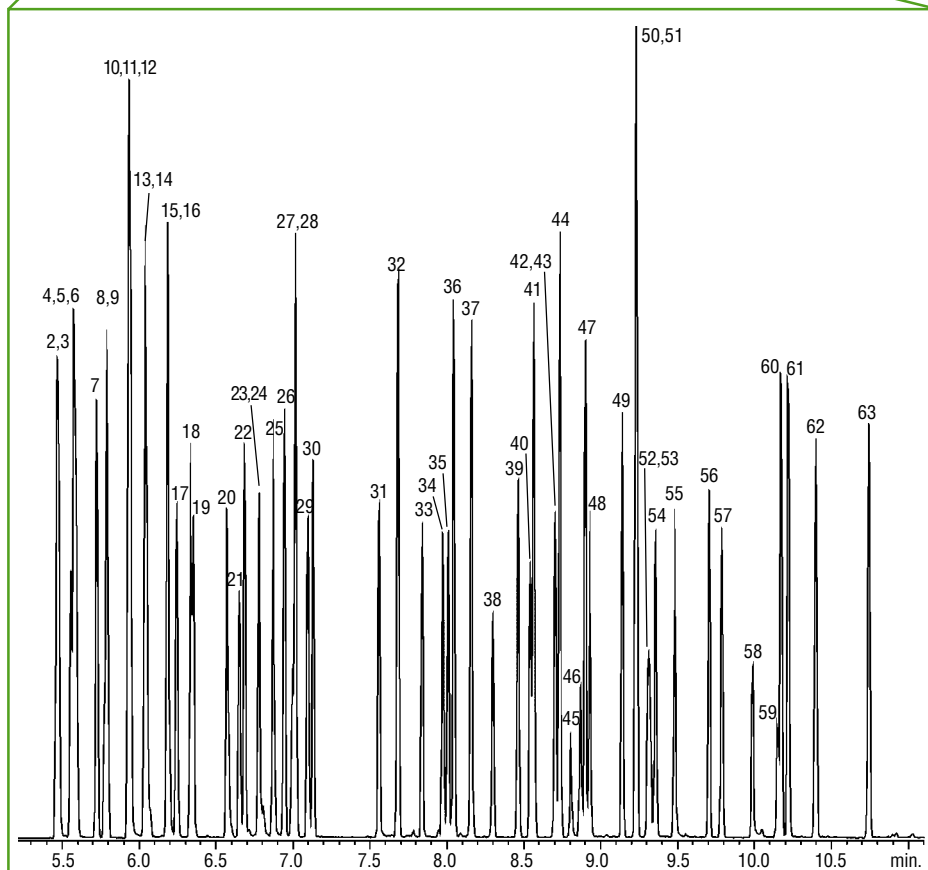
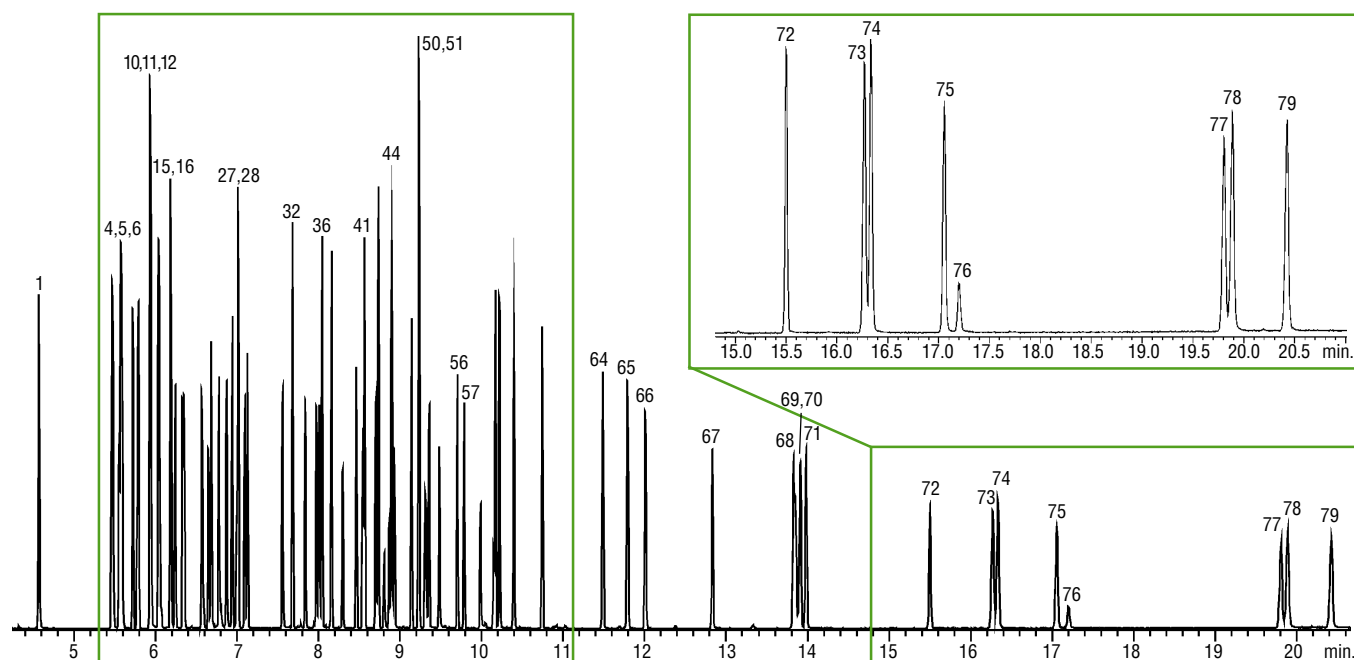
30m, 0.25 mm ID, 0.25µm Rtx®-5Sil MS (cat.# 12723)
20ng split injection of CLP standard; GC: HP/Agilent 6890 w/ 5973
mass selective detector scan range 35-550 AMU; **Oven program:**
40°C (hold 2 min.) to 245°C @ 25°C/min. (hold 0 min.) to 330°C
@ 6°C/min. (hold 5 min.); **Carrier gas:** He @ 1.0 mL/min.
constant flow; **Inj. temp.:** 300°C; **Det. temp.:** 280°C



Peak List for Figures 6 & 7

- | | |
|-----------------------------------|---------------------------------|
| 1. 2-fluorophenol | 41. acenaphthylene |
| 2. phenol-d6 | 42. acenaphthene-d10 |
| 3. phenol | 43. 3-nitroaniline |
| 4. bis(2-chloroethyl)ether | 44. acenaphthene |
| 5. 2-chlorophenol | 45. 2,4-dinitrophenol |
| 6. 2-chlorophenol | 46. 4-nitrophenol |
| 7. 1,3-dichlorobenzene | 47. dibenzofuran |
| 8. 1,4-dichlorobenzene-d4 | 48. 2,4-dinitrotoluene |
| 9. 1,4-dichlorobenzene | 49. diethyl phthalate |
| 10. benzyl alcohol | 50. 4-chlorophenyl phenyl ether |
| 11. 1,2-dichlorobenzene-d4 | 51. fluorene |
| 12. 1,2-dichlorobenzene | 52. 4-nitroaniline |
| 13. 2-methylphenol | 53. 4,6-dinitro-2-methylphenol |
| 14. bis(2-chloroisopropyl)ether | 54. diphenylamine |
| 15. 4-methylphenol/3-methylphenol | 55. 2,4,6-tribromophenol |
| 16. N-nitroso-di-n-propylamine | 56. 4-bromophenyl-phenyl ether |
| 17. hexachloroethane | 57. hexachlorobenzene |
| 18. nitrobenzene-d5 | 58. pentachlorophenol |
| 19. nitrobenzene | 59. phenanthrene-d10 |
| 20. isophorone | 60. phenanthrene |
| 21. 2-nitrophenol | 61. anthracene |
| 22. 2,4-dimethylphenol | 62. carbazole |
| 23. benzoic acid | 63. di-n-butylphthalate |
| 24. bis(2-chloroethoxy)methane | 64. fluoranthene |
| 25. 2,4-dichlorophenol | 65. pyrene |
| 26. 1,2,4-trichlorobenzene | 66. p-terphenyl-d14 |
| 27. naphthalene-d8 | 67. butyl benzyl phthalate |
| 28. naphthalene | 68. benzo(a)anthracene |
| 29. 4-chloroaniline | 69. chrysene-d12 |
| 30. hexachlorobutadiene | 70. chrysene |
| 31. 4-chloro-3-methylphenol | 71. bis(2-ethylhexyl)phthalate |
| 32. 2-methylnaphthalene | 72. di-n-octyl phthalate |
| 33. hexachlorocyclopentadiene | 73. benzo(b)fluoranthene |
| 34. 2,4,6-trichlorophenol | 74. benzo(k)fluoranthene |
| 35. 2,4,5-trichlorophenol | 75. benzo(a)pyrene |
| 36. 2-fluorobiphenyl | 76. perylene-d12 |
| 37. 2-chloronaphthalene | 77. indeno(1,2,3-cd)pyrene |
| 38. 2-nitroaniline | 78. dibenz(a,h)anthracene |
| 39. dimethylphthalate | 79. benzo(ghi)perylene |
| 40. 2,6-dinitrotoluene | |

Figure 7. Analyzing semivolatile compounds in the split injection mode can improve peak shape and eliminate column overload.



30m, 0.25mm ID, 0.25µm
Rtx®-5Sil MS (cat.# 12723)

Sample: 160ng split injection of CLP standard;
GC: HP/Agilent 6890 w/ 5973 mass selective
detector, scan range 35-550 AMU; **Oven program:**
40°C(hold 2 min.) to 245°C @ 25°C/min. (hold 0
min.) to 330°C @ 6°C/min. (hold 5 min.); **Carrier**
gas: He @ 1.0mL/min. constant flow; **Inj. temp.:**
300°C; **Det. temp.:** 280°C

Peak list on page 13

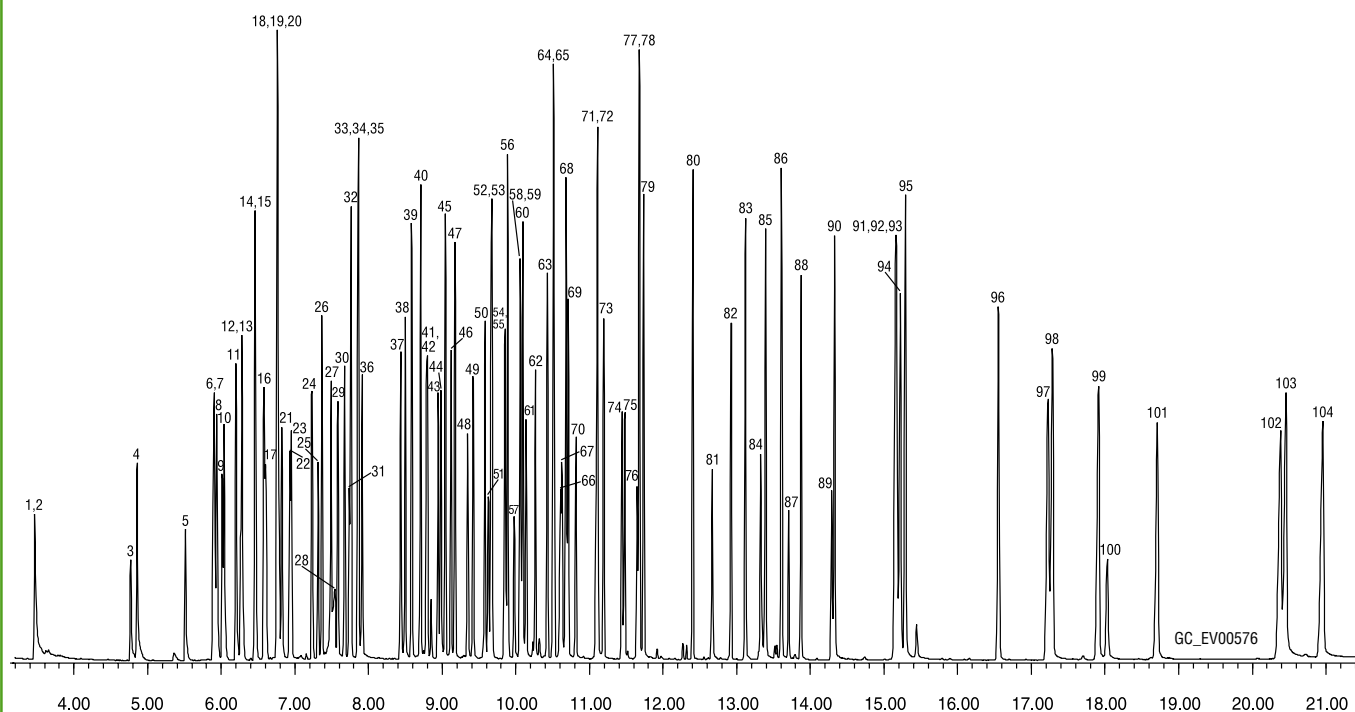
Questions?

Restek's Technical Service
Team is always here to help.
Call us at 800-356-1688 or
814-353-1300, ext. 4,
or email us at
support@restekcorp.com

Reducing Discrimination

Reduced response of the higher molecular weight semivolatile compounds can be caused by discrimination in the injection port. In extreme cases response for the last three PAH compounds may be lost completely at lower calibration levels. To reduce the effects of discrimination in the injection port, we recommend using a drilled Uniliner® inlet liner. Because the column seals into the taper at the bottom of the liner, there is reduced loss of high molecular weight compounds and improved response. The drilled Uniliner® inlet liner has a small hole drilled at the entrance that allows it to work with small diameter columns and Electronic Pressure Control (EPC) injection systems. When using the drilled Uniliner® liner (Figure 8), the response of the last three PAHs is significantly higher compared to the same analysis done with a normal splitless sleeve (see Figure 5). The drilled Uniliner® is available for Agilent 5890 and 6890 GCs (see page 19).

Figure 8. The Rtx®-5Sil MS combined with the Siltek™ drilled Uniliner® liner exhibits excellent peak shape and response for the semivolatile compounds listed in US EPA Method 8270.



1. N-nitrosodimethylamine	27. bis(2-chloroethoxy)methane	53. acenaphthylene	79. anthracene
2. pyridine	28. benzoic acid	54. acenaphthene-d10	80. di-n-butylphthalate
3. methyl methanesulfonate	29. 2,4-dichlorophenol	55. 3-nitroaniline	81. 4-nitroquinoline-1-oxide
4. 2-fluorophenol	30. 1,2,4-trichlorobenzene	56. acenaphthene	82. isodrin
5. ethyl methanesulfonate	31. naphthalene-d8	57. 2,4-dinitrophenol	83. fluoranthene
6. phenol-d6	32. naphthalene	58. pentachlorobenzene	84. benzidine
7. phenol	33. 2,6-dichlorophenol	59. 4-nitrophenol	85. pyrene
8. aniline	34. 4-chloroaniline	60. dibenzofuran	86. p-terphenyl-d14
9. bis(2-chloroethyl)ether	35. hexachloropropene	61. 2,4-dinitrotoluene	87. aramite
10. 2-chlorophenol	36. hexachlorobutadiene	62. 2,3,4,6-tetrachlorophenol	88. chlorbenzilate
11. 1,3-dichlorobenzene	37. 4-chloro-3-methylphenol	63. diethyl phthalate	89. kepone
12. 1,4-dichlorobenzene-d4	38. isosafrole	64. fluorene	90. butyl benzyl phthalate
13. 1,4-dichlorobenzene	39. 2-methylnaphthalene	65. 4-chlorophenyl phenyl ether	91. benzo(a)anthracene
14. 1,2-dichlorobenzene	40. 1-methylnaphthalene	66. 4-nitroaniline	92. 3,3'-dichlorobenzidine
15. benzyl alcohol	41. hexachlorocyclopentadiene	67. 4,6-dinitro-2-methylphenol	93. chrysene-d12
16. 2-methylphenol	42. 1,2,4,5-tetrachlorobenzene	68. diphenylamine	94. chrysene
17. bis(2-chloroisopropyl)ether	43. 2,4,6-trichlorophenol	69. azobenzene	95. bis(2-ethylhexyl)phthalate
18. acetophenone	44. 2,4,5-trichlorophenol	70. 2,4,6-tribromophenol	96. di-n-octyl phthalate
19. 4-methylphenol/3-methylphenol	45. 2-fluorobiphenyl	71. phenacetin	97. benzo(b)fluoranthene
20. N-nitroso-di-n-propylamine	46. safrole	72. 4-bromophenyl phenyl ether	98. benzo(k)fluoranthene
21. hexachloroethane	47. 2-chloronaphthalene	73. hexachlorobenzene	99. benzo(a)pyrene
22. nitrobenzene-d5	48. 2-nitroaniline	74. pentachlorophenol	100. perylene-d12
23. nitrobenzene	49. 1,4-naphthoquinone	75. pentachloronitrobenzene	101. 3-methylcholanthrene
24. isophorone	50. dimethylphthalate	76. phenanthrene-d10	102. indeno(1,2,3-cd)pyrene
25. 2-nitrophenol	51. 1,3-dinitrobenzene	77. dinoseb	103. dibenzo(a,h)anthracene
26. 2,4-dimethylphenol	52. 2,6-dinitrotoluene	78. phenanthrene	104. benzo(g,h,i)perylene

30m, 0.25mm ID, 0.25µm Rtx®-5Sil MS (cat.# 12723)
 Conc.: 24µg/mL in methylene chloride
 (cat.#s: 31618, 31619, 31620, 31621, 31622, 31626,
 31062, 31063)
 Note: Internal standards at 8 ppm

Inj. vol.: 1µL
Inj type: splitless
Hold time: 0.4 min.
Inlet liner: drilled Uniliner® liner, Siltek™
 deactivation (cat# 21054-214.1)
Inj. temp.: 300°C

Carrier gas: helium (1mL/min. constant flow)
Linear velocity: 34cm/sec.
Oven temp.: 35°C (hold 2 min.) to 260°C @
 20°C/min. (hold 0 min.), to 330°C @
 6°C/min. (hold 1 min.)

Det. type: MS
Transfer line temp.: 280°C
Scan range: 35 to 550amu
Ionization: EI
Mode: full scan

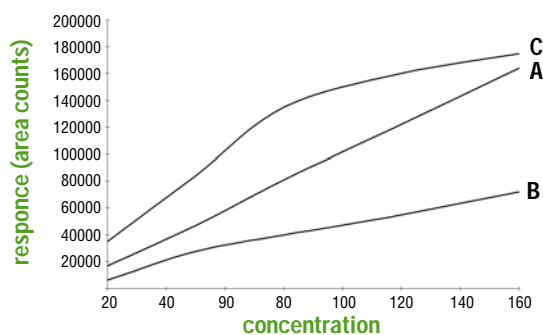
Quantitation

Because splitless injections suffer from irreproducibility, quantitation for semivolatile compounds is by internal standard, using a single ion for each analyte. Internal standards at known concentrations are used to correct for variances in the amount of material transferred to the column with different injections, and also to track MS sensitivity.

All sample analyte concentrations are calculated using response factors obtained from the calibration curve. This method uses single ions (i.e., extracted ions) for each compound so that chromatographic resolution of each compound is not necessary. It is acceptable for compounds to co-elute, as long as they do not have any common ions used for quantitation. Typically, only isomers have similar spectra, so chromatographic resolution is required only for these compounds.

Figure 9 shows the possibilities for calibration curves for this analysis. Curve A is the desired result, indicating a proportional response with increasing concentration. This implies that there is no detector saturation or reactivity for this compound. Curve B is indicative of a compound that undergoes a reaction, usually in the injection port or on the head of the column. If a calibration curve like Curve B is observed, injection port or column maintenance is required. Finally, Curve C shows high-end roll-off, indicating saturation. If a calibration

Figure 9. Calibration curves showing linear response, adsorption, and high-end roll-off.



curve like Curve C is observed, select a column with higher capacity or reduce detector sensitivity by lowering the multiplier voltage.

Summary

Although the analysis of semivolatile organic compounds is one of the more difficult tests performed by environmental laboratories, using Restek's Rtx®-5Sil MS or Rtx®-5MS columns and following the tips presented in this guide can make it easier to perform. Correct sample preparation, extract cleanup, injection technique, analytical columns, standards, and quantitation can help minimize problems normally associated with semivolatile organic analyses. When problems occur, use the most appropriate troubleshooting and maintenance procedures to quickly optimize your analytical system. When faced with difficulties in your semivolatile analysis, remember that the majority of problems occur in the sample preparation and cleanup step, or in the GC injection port. If you still are having difficulties after reading through this guide, contact Restek's Technical Service Team via email at support@restekcorp.com or via phone at 800-356-1688 or 814-353-1300, ext. 4.

Rtx®-5MS and Rtx®-5Sil MS Columns

Conventional capillary gas chromatography (GC) columns use liquid stationary phases, many of which are crossbonded to yield a higher working temperature. Even with crossbonding, however, the liquid stationary phase will slowly elute. This elution of the stationary phase, also termed column bleed, is more detectable at higher temperatures and is typically observed as an increasing baseline that follows the oven temperature program. Depending on the method of detection, column bleed may not be an issue for certain separations. If the capillary column is connected to a sensitive detector like a mass spectrometer (MS), then column bleed can cause a number of problems—specifically misidentification of analytes, loss of sensitivity, and inaccurate quantitation.

The level of column bleed will affect the sensitivity of any MS, especially ion trap instruments, which use automatic gain control. As the level of column bleed increases, so does the signal from bleed ions in the mass spectra of analytes and unknowns. Also, sensitivity (or detection limit) severely degrades. The contribution of bleed ions to the mass spectra can result in misidentification of compounds, requiring laboratory personnel to subtract these ions before performing library searches. Doing so can add considerable time to their analyses. Finally, if bleed ions contribute to the signal of the quantitation mass, quantitation of analytes and unknowns will be miscalculated. For these reasons, it is critical that analysts choose the lowest-bleed column designed for GC/MS applications.

Many manufacturers offer “MS” phases for applications requiring low bleed. In many cases, these represent nothing more than the reporting of the bleed signal when the column was tested for a single analysis at the manufacturer. Restek has developed true low-bleed MS phases. These columns exhibit a much lower column bleed than was previously available. The Rtx®-5MS column is a low-bleed, dimethyl/diphenyl polysiloxane phase. The Rtx®-5Sil MS is a low-bleed silarylene methyl/phenyl phase. The combination of Restek’s polymer chemistry and rigorous QA testing ensures that each MS column exceeds requirements of the most sensitive mass spectrometers.

online
ordering
now available!
for U.S. customers only
www.restekcorp.com

ID	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.25mm	0.10	-60 to 330/350°C	12605	12608	12611
	0.25	-60 to 330/350°C	12620	12623	12626
	0.50	-60 to 330/350°C	12635	12638	12641
	1.00	-60 to 325/350°C	12650	12653	
0.32mm	0.10	-60 to 330/350°C	12606	12609	12612
	0.25	-60 to 330/350°C	12621	12624	12627
	0.50	-60 to 330/350°C	12636	12639	12642
	1.00	-60 to 325/350°C	12651	12654	
0.53mm	0.50	-60 to 320/340°C	12637	12640	
	1.00	-60 to 320/340°C	12652	12655	
	1.50	-60 to 310/330°C	12667	12670	

Rtx®-5MS

Fused Silica (Crossbond® 5% diphenyl - 95% dimethyl polysiloxane) Stable to 360°C

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.10	-60 to 330/350°C	12705	12708
	0.25	-60 to 330/350°C	12720	12723
	0.50	-60 to 330/350°C	12735	12738
	1.00	-60 to 325/350°C	12750	12753
0.28mm	0.25	-60 to 330/350°C	12790	12793
	0.50	-60 to 330/350°C	12791	12794
	1.00	-60 to 325/350°C	12792	12795
0.32mm	0.10	-60 to 330/350°C	12706	12709
	0.25	-60 to 330/350°C	12721	12724
	0.50	-60 to 330/350°C	12736	12739
	1.00	-60 to 325/350°C	12751	12754
0.53mm	0.50	-60 to 320/340°C	12737	12740
	1.00	-60 to 320/340°C	12752	12755
	1.50	-60 to 310/330°C	12767	12770

Rtx®-5Sil MS

Fused Silica (equivalent selectivity of Crossbond® 5% diphenyl/95% dimethyl polysiloxane) Stable to 360°C



**Phases currently available as
Integra-Guard™ columns:**

Rtx®-1	Rtx®-1701
Rtx®-1MS	Rtx®-Volatiles
Rtx®-5	Rtx®-20
Rtx®-5MS	Rtx®-35
Rtx®-5Sil MS	Rtx®-35MS
XTT®-5	Rtx®-BAC 1 & 2
Rtx®-1301	Stabilwax®
Rtx®-624	

Innovative Integra-Guard™ Columns

Some people swear by press-fit connectors, and others swear at them. For many analysts, the art of attaching a guard column to an analytical column is a mystery. Restek's chemists have discovered the solution to this mystery—the easiest, most reliable connection is no connection at all! No guard column system is more permanent than one continuous length of tubing containing both the guard column and the analytical column.

Restek offers a wide variety of Integra-Guard™ capillary columns with a guaranteed leak-free connection! The guard column is tied separately from the analytical column, using high-temperature string. The transition area between the columns is the point at which the guard column ends and the analytical column begins. The entire setup is suspended in our unique “crush-free” cage, which prevents the column from coming in contact with anything that could damage it.

Ordering is simple. Just add the appropriate suffix number and price to the analytical column's catalog number and price. For example, a 30m, 0.25mm ID, 0.25µm Rtx®-5MS with a 5m Integra-Guard™ column is cat.# 12623-124.

ID (mm)	Length (m)	Suffix #
0.25	5	-124
	10	-127
0.28	5	-243
	10	-244

ID (mm)	Length (m)	Suffix #
0.32	5	-125
	10	-128
0.53	5	-126
	10	-129

For analysts who prefer to attach a guard column to the analytical column themselves, Restek offers deactivated guard columns and Press-Tight® connectors.

formoreinfo

For detailed information on types of guard columns, their uses, and a complete product listing, request Restek's **Guard Column Fast Facts** flyer (lit. cat.# 59319)

Intermediate-Polarity Deactivated Guard Columns

Nominal ID (mm)	Nominal OD (mm)	1-Meter	5-Meter
0.15	0.363 ± 0.012	10101	10042
0.18	0.37 ± 0.04	10102	10046
0.25	0.37 ± 0.04	—	10043
0.28	0.37 ± 0.04	—	10003
0.32	0.45 ± 0.04	—	10044
0.45	0.69 ± 0.05	—	10005
0.53	0.69 ± 0.05	—	10045

Press-Tight® Connectors

- Seals all common sizes (0.18 to 0.53mm ID, outside diameters from 0.3 to 0.75mm) of fused silica tubing.
- Connect guard columns to analytical columns, repair broken columns, or connect column outlets to transfer lines.
- Angled connectors are designed to approximate the curvature of a capillary column and reduce strain on column-end connections.
- Made from inert fused silica.

Universal Angled Press-Tight® Connectors:

cat.# 20446 (5-pk.); cat.# 20447 (25-pk.); cat.# 20448 (100-pk.)

Universal Press-Tight® Connectors:

cat.# 20400 (5-pk.); cat.# 20401 (25-pk.); cat.# 20402 (100-pk.)

Deactivated Universal Press-Tight® Connectors:

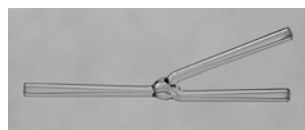
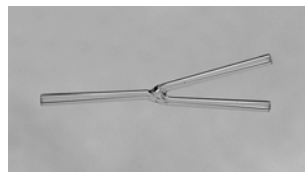
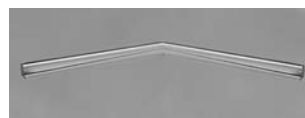
cat.# 20429 (5-pk.); cat.# 20430 (25-pk.); cat.# 20431 (100-pk.)

Universal “Y” Press-Tight® Connectors:

cat.# 20405 (ea.); cat.# 20406 (3-pk.)



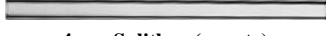
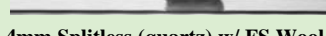


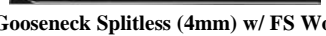


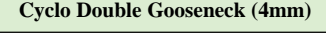
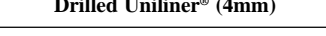
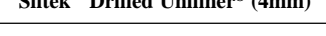
Universal Angled “Y” Press-Tight® Connectors:

cat.# 20403 (ea.); cat.# 20404 (3-pk.)




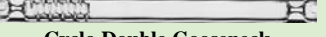



Inlet Liners

For Agilent/HP & Finnigan GCs

COLUMN INSTALLS THIS END	Splitless Liners for Agilent/HP & Finnigan GCs	Benefits/Uses:	ID*/OD & Length (mm)	ea.	cat.# 5-pk.	25-pk.	Similar to Agilent part #
	 4mm Splitless	trace samples >2µL	4.0 ID 6.5 OD x 78.5	20772	20773	20774	—
	 4mm Splitless w/ FS Wool	trace samples >2µL	4.0 ID 6.5 OD x 78.5	22400	22401	22402	19251-60540
	 4mm Splitless (quartz)	trace samples >2µL	4.0 ID 6.5 OD x 78.5	20912	20913	—	—
	 4mm Splitless (quartz) w/ FS Wool	trace samples >2µL	4.0 ID 6.5 OD x 78.5	22403	22404	—	18740-80220 5181-8818
	 Gooseneck Splitless (2mm)	trace samples <2µL	2.0 ID 6.5 OD x 78.5	20795	20796	20797	—
	 Gooseneck Splitless (4mm)	trace samples >2µL	4.0 ID 6.5 OD x 78.5	20798	20799	20800	5181-3316
	 Gooseneck Splitless (4mm) w/ FS Wool	trace samples >2µL	4.0 ID 6.5 OD x 78.5	22405	22406	22407	5062-3587
	 Double Gooseneck Splitless (4mm)	trace, active samples >2µL	4.0 ID 6.5 OD x 78.5	20784	20785	20786	5181-3315
	 Cyclo Double Gooseneck (2mm)	trace, active, dirty samples <2µL	2.0 ID 6.5 OD x 78.5	20907	20908	—	—
	 Cyclo Double Gooseneck (4mm)	trace, active, dirty samples >2µL	4.0 ID 6.5 OD x 78.5	20895	20896	20997	—
	 Drilled Uniliner® (4mm)	allows direct injection when using EPC-equipped GC	4.0 ID 6.3 OD x 78.5	21054	21055	—	—
	 Siltek™ Drilled Uniliner® (4mm)	allows direct injection when using EPC-equipped GC	4.0 ID 6.3 OD x 78.5	21054-214.1	21055-214.5	—	—




For Varian GCs

COLUMN INSTALLS THIS END	Splitless Liners for Varian 1075/1077GCs	Benefits/Uses:	ID*/OD & Length (mm)	ea.	cat.# 5-pk.	25-pk.	Similar to Varian Part #
	 2mm Splitless	trace samples <2µL	2.0 ID 6.3 OD x 74	20721	20722	20723	01-900109-05
	 4mm Splitless	trace samples >2µL	4.0 ID 6.3 OD x 74	20904	20905	20906	01-900109-05
	 Double Gooseneck	trace, active samples up to 4µL	4.0 ID 6.3 OD x 74	20847	20848	20849	—
	 Cyclo Double Gooseneck	trace, dirty, active samples up to 4µL	4.0 ID 6.3 OD x 74	20897	20898	—	—
COLUMN INSTALLS THIS END	1078/1079 Liners for Varian GCs	Benefits/Uses:	ID*/OD & length (mm)	ea.	cat.# 5-pk.	25-pk.	Similar to Varian Part #
	 1078/1079 Splitless	trace samples <2µL	2.0 ID 5.0 OD x 54	21711	21712	—	03-918466-00


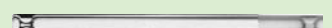
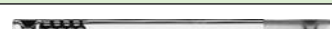

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Inlet Liners



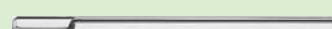

For Shimadzu GCs

Splitless Liners for Shimadzu GCs	Benefits/Uses:	ID**/OD & Length (mm)	ea.	cat.# 5-pk. 25-pk.	Similar to Shimadzu Part #
 94mm Splitless with Wool*	trace samples	3.5 ID 5.0 OD x 94	20955	20956 20957	221-09145
 94mm Double Gooseneck	reduces backflash and catalytic decomposition	3.5 ID 5.0 OD x 94	20958	20959 20960	—
 94mm Single Gooseneck	reduces backflash, also operates in DI mode	3.5 ID 5.0 OD x 94	20961	20962 20963	221-41599-00

For PerkinElmer GCs

Splitless Liners for PerkinElmer GCs	Benefits/Uses:	ID**/OD & Length (mm)	ea.	cat.# 5-pk. 25-pk.	Similar to PE Part #
 Auto SYS Splitless w/Wool (2mm ID)*	trace samples	2.0 ID 6.2 OD x 92.1	20829	20830 20831	N6101372
 Auto SYS Double Gooseneck	trace, active samples up to 4µL	3.5 ID 6.2 OD x 92.1	20853	20854 —	—
 Auto SYS Cyclo Double Gooseneck	trace, dirty, active samples up to 4µL	3.5 ID 6.2 OD x 92.1	20899	20900 —	—
 Auto SYS XL Split/Splitless	most common analyses	2.0 ID 4.0 OD x 86.2	21717	21718 —	N612-1004

For Thermo Orion GCs

Splitless Liners for 5000-6000 Series GCs	Benefits/Uses:	ID**/OD & Length (mm)	ea.	cat.# 5-pk. 25-pk.	Similar to TO Part #
 Splitless (4mm ID)	trace samples	4.0 ID 5.5 OD x 79.5	20814	20815 20816	—
Splitless Liners for 8000 & TRACE™ Series GCs	Benefits/Uses:	ID**/OD & Length (mm)	ea.	cat.# 5-pk. 25-pk.	Similar to TO Part #
 Splitless (3mm ID)	trace samples	3.0 ID 8.0 OD x 105	20942	20943 20944	453 20032
 Splitless (5mm ID)	trace samples	5.0 ID 8.0 OD x 105	20945	20946 20947	453 20033
 Double Gooseneck	trace, active samples up to 4µL	4.0 ID 8.0 OD x 105	20952	20953 —	—

O-Rings

Viton®

Viton® o-rings are universal. One size fits both split (6.3mm ID) and splitless (6.5mm ID) sleeves.

	Max. temp.	Similar to Agilent Part #	cat.#	Qty.
Viton® (fluorocarbon)	350°C	5180-4182	20377	25-pk.

Graphite

Graphite o-rings have excellent thermal stability and can be used at injection port temperatures up to 450°C!

	Similar to Agilent Part #	10-pk.	50-pk.
6.35mm ID for split liners	5180-4168	20296	20297
6.5mm ID for splitless liners	5180-4173	20298	20299



*Liner is
**Nomini

Deactivated Fused Silica Wool

- Ensure uniform vaporization in split or splitless liners.
- Prolong column life by trapping septum particles.
- Recommended for autosamplers with fast injection rates.
- Inertness tested for endrin breakdown.

cat.# 20790, (10 grams)

free
guide

Request the handy,
pocket-sized, *Inlet
Supplies Guide*
(lit. cat.# 59893A).



Replacement Inlet Seals

- Special grade of stainless steel deforms easily, ensuring a completely leak-free seal.
- Available in stainless steel, gold-plated, and Silcosteel®-treated.
- Cross-Disk ideal for high-flow split applications on EPC-equipped GCs.
- Shipped with washers.

For Agilent 5890/6890/6850 Split/Splitless Injection Ports

Single-Column Installation, Opening Size 0.8mm ID	
Stainless Steel Inlet Seal*	
21315, 2-pk.	21316, 10-pk.
Gold-Plated Inlet Seal**	
21317, 2-pk.	21318, 10-pk.
Silcosteel® Inlet Seal	
21319, 2-pk.	21320, 10-pk.

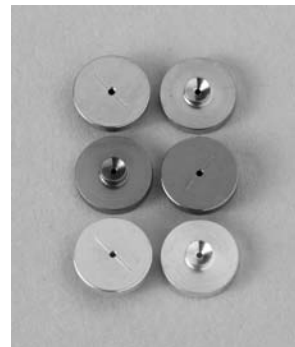
*0.8mm ID stainless steel inlet seal is equivalent to Agilent part #18740-20880.

**0.8mm ID gold-plated inlet seal is equivalent to Agilent part #18740-20885.

Cross-Disk for Agilent GCs†

Cross-Disk, Opening Size 0.8mm ID	
Gold-Plated Inlet Seal	
20477, 2-pk.	20476, 10-pk.
Silcosteel® Inlet Seal	
20475, 2-pk.	20474, 10-pk.
Cross-Disk, Opening Size 1.2mm ID	
Gold-Plated Inlet Seal	
21009, 2-pk.	21010, 10-pk.
Silcosteel® Inlet Seal	
21011, 2-pk.	21012, 10-pk.

†Similar to Agilent part #5182-9652.



Thermolite® Septa

- Each batch tested on FIDs, ECDs, & MSDs to ensure lowest bleed.
- Excellent puncturability.
- Preconditioned and ready to use.
- Do not adhere to hot metal surfaces.
- Usable to 340°C inlet temperatures.
- Packaged in non-contaminating tins.

Septum Diameter	25-pk.	50-pk.	100-pk.
5mm (3/16")	20351	20352	20353
6mm (1/4")	20355	20356	20357
7mm	20381	20382	20383
8mm	20370	20371	—
9.5mm (3/8")	20359	20360	20361
10mm	20378	20379	20380
11mm (7/16")	20363	20364	20365
11.5mm	22385	22386	22387
12.5mm (1/2")	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Blue	20372	20373	20374



Call our literature hotline
at 800-356-1688
or 814-353-1300, ext. 5,
or your local Restek
representative for
Restek's 20-page bulletin,
*A Guide to Minimizing
Septa Problems*
(lit. cat.# 59886).

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guide

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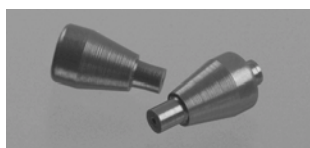
Website : www.chromtech.net.au E-mail : info@chromtech.net.au TelNo : 03 9762 2034 . . . in AUSTRALIA



EZ-Vent™ 2000 for Agilent GCs



EZ-Vent™ 2000 for Varian GCs



EZ-Vent™ 2000 ferrules

EZ-Vent™ 2000

- Change GC/MS columns in minutes without venting.
- Silcosteel® treated for greater inertness.
- Deactivated transfer line minimizes bleed into the source.
- Stainless steel body and high-temperature polyimide ferrules minimize leaks at the problematic transfer line fitting.
- Less expensive than other “no-vent” fittings.
- 100µm transfer line throttles vacuum and prevents column pump down.
- Available for Agilent GCs with 5971/5972 or 5973 MS and Varian 3400, 3600, or 3800 GCs with Saturn 2000 MS.
- Precision-machined orifice.



Kits

EZ-Vent™ 2000 for Agilent GCs with 5971/5972 or 5973 MS

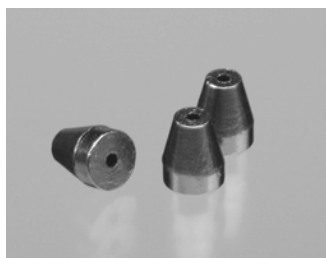
Includes EZ-Vent™ 2000, 1/16" SS nut, 0.4mm ID ferrules for connecting capillary column, 0.4mm ID ferrules for connecting transfer line, 100µm deactivated transfer line (3 ft.), and EZ-Vent™ column plug; cat.# 21013, (kit)

EZ-Vent™ 2000 for Varian Saturn 2000 MS systems with 3400, 3600, or 3800 GCs

Includes EZ-Vent™ 2000, 1/16" SS nut, 0.4mm ferrules for connecting capillary column, 0.4mm ID ferrules for connecting transfer line, 100µm deactivated transfer line (3 ft.), and EZ-Vent™ column plug; cat.# 21014, (kit)

Ferrules

Capillary Ferrules (for 1/16" compression-type fittings)



Ferrule ID (mm)	Fits Column ID (mm)	Qty.	Graphite	Vespe®/ Graphite
0.4	0.25	50-pk.	20227	20229
0.4	0.25	10-pk.	20200	20211
0.5	0.32	10-pk.	20201	20212
0.5	0.32	50-pk.	20228	20231
0.6	0.28	10-pk.	—	20232
0.8	0.53	10-pk.	20202	20213
0.8	0.53	50-pk.	20224	20230

Compact Ferrules for Agilent GCs

Ferrule ID (mm)	Fits Column ID (mm)	Qty.	Graphite	Vespe®/ Graphite
0.4	0.25	10-pk.	20250	20238
0.4	0.25	50-pk.	20251	20239
0.5	0.32	10-pk.	21007	20248
0.5	0.32	50-pk.	21008	20249
0.8	0.53	10-pk.	20252	20263
0.8	0.53	50-pk.	20253	20264

Encapsulated Ferrules

- Will not deform and stick in fittings.
- Reusable.
- For 1/16" compression fittings.

Ferrule ID	Fits column ID	cat.#/10-pk.
0.4mm	0.25mm	21036
0.5mm	0.32mm	21037
0.8mm	0.53mm	21038

Direct Replacement Split/Splitless Injection Ports for Agilent GCs

Would you like better performance from your injector? Restek's Silcosteel®-coated split/splitless injector is a **direct replacement** for Agilent 5890 and 6890/6850 GCs. The injector is manufactured from high-quality stainless steel and meets or exceeds Agilent original equipment specifications. Silcosteel® passivates the metal surface to ensure an inert pathway for the sample, delivering increased performance.

SILCOSTEEL®
version available

For Agilent 5890 GCs

Description	cat.#, (ea.)
Replacement Weldment (Similar to Agilent part# 19251-60575)	20265
Replacement Shell Weldment (Similar to Agilent part# 19251-80570)	20266
Silcosteel® Weldment	20267
Silcosteel® Shell Weldment	20268

For Agilent 6890/6850 GCs

Description	cat.#, (ea.)
Replacement Weldment for Agilent 6890/6850 GCs with EPC	22674
Replacement Weldment for Agilent 6890/6850 GCs with manual flow	20265
Replacement Shell Weldment for Agilent 6890/6850 GCs	22673



Weldment for Agilent 5890 GCs



Shell weldment for Agilent 5890 GCs



Weldment and shell weldment for Agilent 6890/6850 GCs

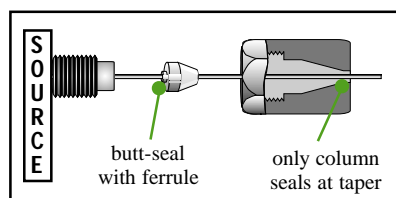
MSD Conversion Fitting—Improved

- Uses a flat, soft aluminum sealing ring to deform and butt-seal against the MSD interface (see figure below).
- A standard Vespel® ferrule seals the column and 1/16-inch stainless steel nut.
- Fitting is constructed of nickel-plated brass for longevity and softness.
- Can use any standard Vespel® or Vespel®/graphite 1/16-inch ferrule.
- Includes a 1/16-inch stainless steel nut and two replacement sealing rings. Order ferrules separately.

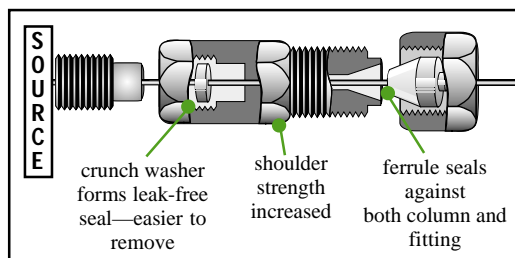
MSD Conversion Fitting: cat.# 21314, (ea.)

MSD Conversion Fitting Replacement Ring Seal: cat.# 21313, (2-pk.)

Original Agilent Design



Restek MSD Conversion Fitting



MSD Source Nut

The nut bore has been changed from 0.8mm to 1.2mm to permit easy removal of ferrules with a standard tapered-needle file (cat.# 20106). The nuts still match the manufacturer's original part specifications and are made of brass to prevent thread-stripping on the transfer line. (Similar to Agilent part #05988-20066.)

(Detector) MSD Source Nut: cat.# 20643, (2-pk.)

 **check it out**



Agilent's MSD interface requires a butt-seal at the base of a Vespel® ferrule, which is prone to leakage. Restek's version uses a standard ferrule design that simultaneously seals the fitting and capillary tubing with compressive forces.





US EPA Method 8270D outlines the analysis of semivolatile organic pollutants in solid waste, soil, water, and air matrices, using GC/MS. Update IVA of the third edition of SW-846—*Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*—includes EPA Method 8270D, in which there were no major revisions from EPA Method 8270C.

Method 8270 Calibration Kits

8270 Calibration Kit

31618: 8270 Calibration Mix #1
31619: 8270 Calibration Mix #2
31620: 8270 Calibration Mix #3
31621: 8270 Calibration Mix #4
31622: 8270 Calibration Mix #5



Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31626	31626-500

8270/Appendix IX Calibration Kit

31618: 8270 Calibration Mix #1
31619: 8270 Calibration Mix #2
31620: 8270 Calibration Mix #3
31621: 8270 Calibration Mix #4
31622: 8270 Calibration Mix #5
31623: 8270 Calibration Mix #6
31625: Appendix IX Mix #1



Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31627	31627-500

Aramite Mix

2,000ppm each in hexane, 1mL/ampul

	Each	5-pk.	10-pk.
	31624	31624-510	
w/data pack	31624-500	31624-520	31724

US EPA Methods 8270C & 8270D Analytical Reference Materials

Internal Standard Mixtures

SV Internal Standard Mix

acenaphthene-d10 naphthalene-d8
crysene-d12 perylene-d12
1,4-dichlorobenzene-d4 phenanthrene-d10
4,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31006	31006-510	
w/data pack	31006-500	31006-520	31106

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31206	31206-510	
w/data pack	31206-500	31206-520	31306

Surrogate Mixtures

B/N Surrogate Mix (4/89 SOW)

2-fluorobiphenyl p-terphenyl-d14
nitrobenzene-d5
1,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31024	31024-510	
w/data pack	31024-500	31024-520	31124

5,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31062	31062-510	
w/data pack	31062-500	31062-520	31162

5,000µg/mL each in methylene chloride, 5mL/ampul

	Each	5-pk.	10-pk.
	31086	31086-510	
w/data pack	31086-500	31086-520	31186

Acid Surrogate Mix (4/89 SOW)

2-fluorophenol 2,4,6-tribromophenol
phenol-d6
2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31025	31025-510	
w/data pack	31025-500	31025-520	31125

10,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31063	31063-510	
w/data pack	31063-500	31063-520	31163

10,000µg/mL each in methylene chloride, 5mL/ampul

	Each	5-pk.	10-pk.
	31087	31087-510	
w/data pack	31087-500	31087-520	31187

Matrix Spiking Mixtures

B/N Matrix Spike Mix

acenaphthene N-nitroso-di-n-propylamine
1,4-dichlorobenzene pyrene
2,4-dinitrotoluene 1,2,4-trichlorobenzene
1,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31004	31004-510	
w/data pack	31004-500	31004-520	31104

5,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31074	31074-510	
w/data pack	31074-500	31074-520	31174

5,000µg/mL each in methylene chloride, 5mL/ampul

	Each	5-pk.	10-pk.
	31084	31084-510	
w/data pack	31084-500	31084-520	31184

Acid Matrix Spike Mix

4-chloro-3-methylphenol pentachlorophenol
2-chlorophenol phenol
4-nitrophenol

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31014	31014-510	
w/data pack	31014-500	31014-520	31114

10,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31061	31061-510	
w/data pack	31061-500	31061-520	31161

10,000µg/mL each in methylene chloride, 5mL/ampul

	Each	5-pk.	10-pk.
	31071	31071-510	
w/data pack	31071-500	31071-520	31171

GC/MS Tuning Mixture

GC/MS Tuning Mixture

benzidine DFTPP
4,4'-DDT pentachlorophenol

1,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31615	31615-510	
w/data pack	31615-500	31615-520	31715

US EPA Methods 8270C & 8270D Analytical Reference Materials

Calibration Check Compound Mixtures

8270 B/N Calibration Check Mix

acenaphthene
benzo(a)pyrene
1,4-dichlorobenzene
di-*n*-octyl phthalate

diphenylamine
fluoranthene
hexachlorobutadiene

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31616	31616-510	
w/data pack	31616-500	31616-520	31716

8270 Acid Calibration Check Mix

4-chloro-3-methylphenol
2,4-dichlorophenol
2-nitrophenol

pentachlorophenol
phenol
2,4,6-trichlorophenol

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31617	31617-510	
w/data pack	31617-500	31617-520	31717

Calibration Mixtures

8270 Calibration Mix #1

benzoic acid
4-chloro-3-methylphenol
2-chlorophenol
2,4-dichlorophenol
2,6-dichlorophenol
2,4-dimethylphenol
4,6-dinitro-2-methylphenol
2,4-dinitrophenol
dinoseb
2-methylphenol

3-methylphenol
4-methylphenol
2-nitrophenol
4-nitrophenol
pentachlorophenol
phenol
2,3,4,6-tetrachlorophenol
2,4,5-trichlorophenol
2,4,6-trichlorophenol

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31618	31618-510	
w/data pack	31618-500	31618-520	31718

8270 Calibration Mix #2

aniline
benzidine
4-chloroaniline
3,3'-dichlorobenzidine
diphenylamine
2-nitroaniline

3-nitroaniline
4-nitroaniline
N-nitrosodimethylamine
N-nitrosodi-*n*-propylamine
pyridine

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31619	31619-510	
w/data pack	31619-500	31619-520	31719

Calibration Mixtures

8270 Calibration Mix #3

aramite
bis (2-chloroethyl) ether
bis (2-chloroethoxy) methane

bis (2-chloroisopropyl) ether
4-bromophenyl phenyl ether
chlorobenzilate
2-chloronaphthalene
4-chlorophenyl phenyl ether
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
1,3-dinitrobenzene

hexachlorobenzene
hexachlorobutadiene
hexachloro-cyclopentadiene
hexachloroethane
hexachloropropene
isodrin
kepone
pentachlorobenzene
pentachloronitrobenzene
1,2,4,5-tetrachlorobenzene
1,2,4-trichlorobenzene

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31620	31620-510	
w/data pack	31620-500	31620-520	31720

8270 Calibration Mix #4

acetophenone
azobenzene
benzyl alcohol
bis (2-ethylhexyl) phthalate
butyl benzyl phthalate
dibenzofuran
diethyl phthalate
dimethyl phthalate
di-*n*-butyl phthalate
di-*n*-octyl phthalate
2,4-dinitrotoluene

2,6-dinitrotoluene
ethyl methanesulfonate
isophorone
isosafrole (*cis* & *trans*)
methyl methanesulfonate
1,4-naphthoquinone
nitrobenzene
4-nitroquinoline-1-oxide
phenacetin
safrole

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31621	31621-510	
w/data pack	31621-500	31621-520	31721

8270 Calibration Mix #5

acenaphthene
acenaphthylene
anthracene
benzo(a)anthracene
benzo(a)pyrene
benzo(b)fluoranthene
benzo(ghi)perylene
benzo(k)fluoranthene
chrysene
dibenz(a,h)anthracene

fluoranthene
fluorene
ideno(1,2,3-cd)pyrene
3-methylcholanthrene
1-methylnaphthalene
2-methylnaphthalene
naphthalene
phenanthrene
pyrene

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31622	31622-510	
w/data pack	31622-500	31622-520	31722

Calibration Mixtures

8270 Calibration Mix #6

diallate (*cis* & *trans*)
dimethoate
disulfoton
famphur
methyl parathion
phosphorothioate

parathion
phorate
pronamide
thionazine
0,0,0-triethyl

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31623	31623-510	
w/data pack	31623-500	31623-520	31723

Organochlorine Pesticide Mix AB #1

aldrin
α-BHC
α-chlordane
β-BHC
4,4'-DDD
4,4'-DDE
4,4'-DDT
δ-BHC
dieldrin
endosulfan I

endosulfan II
endosulfan sulfate
endrin
endrin aldehyde
endrin ketone
γ-BHC (lindane)
γ-chlordane
heptachlor
heptachlor epoxide (B)
methoxychlor

200µg/mL each in hexane/toluene (1:1), 1mL/ampul

	Each	5-pk.	10-pk.
	32291	32291-510	
w/data pack	32291-500	32291-520	32391

Appendix IX Mix #1

2-acetylaminofluorene
4-aminobiphenyl
p-dimethylaminoazobenzene
3,3'-dimethylbenzidine
α,α,-dimethylphenethylamine (free base)
methapyrilene (free base)
1-naphthylamine
2-naphthylamine
5-nitro-*o*-toluidine
N-nitrosodibutylamine

N-nitrosodiethylamine
N-nitrosomethylethylamine
N-nitrosomorpholine
N-nitrosopiperidine
N-nitrosopyrrolidine
1,4-phenylenediamine
2-picoline
o-toluidine

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31625	31625-510	
w/data pack	31625-500	31625-520	31725

CLP GPC Calibration Mix

Qualitative mixture useful for determining GPC dump/collect times. Data packs are not available. The compounds are dissolved in methylene chloride at the concentrations listed.

CLP GPC Calibration Mix

bis(2-ethylhexyl)phthalate	10mg/mL
corn oil	250
methoxychlor	2.0
perylene	0.2
sulfur	0.8

In methylene chloride, 1mL/ampul

1mL/ampul	Each	5-pk.	10-pk.
	32019	32019-510	32119
5mL/ampul	Each	5-pk.	10-pk.
	32023	32023-510	32123

Revised GPC Calibration Mix

bis(2-ethylhexyl)phthalate	5mg/mL
corn oil	250
methoxychlor	1.0
perylene	0.2
sulfur	0.8

In methylene chloride, 1mL/ampul

1mL/ampul	Each	5-pk.	10-pk.
	32041	32041-510	32141
5mL/ampul	Each	5-pk.	10-pk.
	32042	32042-510	32142

**Restek
Tip**

CLP OLM 04.1, 04.2 Semivolatile Dilution

Benzaldehyde and atrazine will react quickly and directly with the methanol stabilizer used in most brands and grades of methylene chloride. This reaction will prevent you from obtaining stable, working-level calibration standards. Therefore, Restek has prepared the CLP OLM 04.1 Semivolatile B/N Mega- Mix™ from methylene chloride that is stabilized with amylene and is completely free of methanol. Restek strongly recommends screening the methylene chloride you use to dilute these mixtures to confirm that it is free of methanol.

EPA CLP—Semivolatiles Calibration Mixtures

04.1 SOW, 04.2 OSW

Restek chemists carefully reviewed the 04.2 Statement of Work and determined that the identical products listed in 04.1 will also be required for the 04.2 revision. The products listed here are a result of this work.

CLP 04.1 Phenols Calibration Mix

4-chloro-3-methylphenol	2-methylphenol
2-chlorophenol	4-methylphenol
4-nitrophenol	2-nitrophenol
2,4-dichlorophenol	pentachlorophenol
2,4-dimethylphenol	phenol
2,4-dinitrophenol	2,4,5-trichlorophenol
2-methyl-4,6-dinitrophenol	2,4,6-trichlorophenol

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31494	31494-510	—
w/data pack	31494-500	31494-520	31594

CLP 04.1 B/N MegaMix™

Note: This product is provided as a two ampul set:

CLP 04.1 B/N MegaMix™ Mix A

acenaphthene	di-n-octyl phthalate
acenaphthylene	dibenz(a,h)anthracene
acetophenone	dibenzofuran
anthracene	3,3'-dichlorobenzidine
atrazine	diethyl phthalate
benzo(a)anthracene	dimethyl phthalate
benzaldehyde	2,4-dinitrotoluene
benzo(a)pyrene	2,6-dinitrotoluene
benzo(b)fluoranthene	fluoranthene
benzo(k)fluoranthene	fluorene
benzo(ghi)perylene	hexachlorobenzene
1,1'-biphenyl	hexachlorobutadiene
bis(2-chloroethoxy)methane	
hexachlorocyclopentadiene	hexachloroethane
bis(2-chloroethyl)ether	ideno(1,2,3-cd)pyrene
bis-(2-chloroisopropyl)ether	isophorone
bis(2-ethylhexyl)phthalate	2-methylnaphthalene
4-bromophenyl phenyl ether	naphthalene
butyl benzyl phthalate	nitrobenzene
caprolactam	n-nitrosodi-n-propylamine
carbazole	n-nitrosodiphenylamine
2-chloronaphthalene	phenanthrene
4-chlorophenyl phenyl ether	pyrene
chrysene	
di-n-butyl phthalate	

1,000µg/mL each in methylene chloride/benzene (75:25)

CLP 04.1 B/N MegaMix™ Mix B

4-chloroaniline	3-nitroaniline
2-nitroaniline	4-nitroaniline

1,000µg/mL each in methylene chloride

	Each	5-pk.	10-pk.
	31495	31495-510	—
w/data pack	31495-500	31495-520	31595

CLP OLM 04.1 SV Kit #1

31000: SV Screening Mix
31001: SV Tuning Mix
31493: CLP 04.1 BNA Surrogate Mix
31492: CLP 04.1 B/N Matrix Spike Mix
31005: Acid Matrix Spike Mix
31006: SV Internal Standard Mix
31494: CLP 04.1 Phenols Calibration Mix
31495: CLP 04.1 B/N MegaMix™
31012: SV Calibration Mix #6 (pesticides)

kit

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31603	31603-500

CLP OLM 04.1 SV Kit #2

31494: CLP 04.1 Phenols Calibration Mix
31495: CLP 04.1 B/N MegaMix™
31012: SV Calibration Mix #6 (pesticides)

kit

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31604	31604-500

CLP OLM 04.1 SV Kit #3

31494: CLP 04.1 Phenols Calibration Mix
31495: CLP 04.1 B/N MegaMix™

kit

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31605	31605-500

Questions?

Restek's Technical Service Team is always here to help. Call us at 800-356-1688 or 814-353-1300, ext. 4, or email us at support@restekcorp.com

EPA CLP—Semivolatiles Calibration Mixtures and Kits

3/90 and 4/89 SOW

SV Calibration Mix #1

benzyl alcohol 3-nitroaniline
4-chloroaniline 4-nitroaniline
2-nitroaniline

2,000µg/mL each in CH₂Cl₂, 1mL/ampul

	Each	5-pk.	10-pk.
	31007	31007-510	
w/data pack	31007-500	31007-520	31107

SV Calibration Mix #2

benzoic acid 4-methylphenol
4-chloro-3-methylphenol 2-nitrophenol
2-chlorophenol 4-nitrophenol
2,4-dichlorophenol pentachlorophenol
2,4-dimethylphenol phenol
2,4-dinitrophenol 2,4,5-trichlorophenol
2-methyl-4,6-dinitrophenol 2,4,6-trichlorophenol
2-methylphenol

2,000µg/mL each in CH₂Cl₂, 1mL/ampul

	Each	5-pk.	10-pk.
	31008	31008-510	
w/data pack	31008-500	31008-520	31108

SV Calibration Mix #3

bis(2-chloroethoxy)methane 4-chlorophenyl phenyl ether
bis(2-chloroethyl)ether dimethyl phthalate
bis(2-chloroisopropyl)ether di-*n*-butyl phthalate
bis(2-ethylhexyl)phthalate di-*n*-octyl phthalate
4-bromophenyl phenyl ether N-nitrosodimethylamine
butyl benzyl phthalate N-nitroso-di-*n*-propylamine
2-chloronaphthalene N-nitrosodiphenylamine

2,000µg/mL each in CH₂Cl₂, 1mL/ampul

	Each	5-pk.	10-pk.
	31009	31009-510	
w/data pack	31009-500	31009-520	31109

SV Calibration Mix #4

carbazolehexachlorocyclopentadiene
dibenzofuran hexachloroethane
diethyl phthalate isophorone
2,4-dinitrotoluene 2-methylnaphthalene
2,6-dinitrotoluene nitrobenzene
hexachlorobenzene 1,2,4-trichlorobenzene
hexachlorobutadiene

2,000µg/mL each in CH₂Cl₂, 1mL/ampul

	Each	5-pk.	10-pk.
	31010	31010-510	
w/data pack	31010-500	31010-520	31110

SV Calibration Mix #5

acenaphthene chrysene
acenaphthylene dibenzo(a,h)anthracene
anthracene fluoranthene
benzo(a)anthracene fluorene
benzo(a)pyrene indeno(1,2,3-cd)pyrene
benzo(b)fluoranthene naphthalene
benzo(k)fluoranthene phenanthrene
benzo(ghi)perylene pyrene

2,000µg/mL each in CH₂Cl₂, 1mL/ampul

	Each	5-pk.	10-pk.
	31011	31011-510	
w/data pack	31011-500	31011-520	31111

SV Calibration Mix #6

aldrin endosulfan I
α-BHC endosulfan II
β-BHC endosulfan sulfate
δ-BHC endrin
γ-BHC (lindane) endrin aldehyde
4,4'-DDD endrin ketone
4,4'-DDE heptachlor
4,4'-DDT heptachlor epoxide (B)
dieldrin methoxychlor

2,000µg/mL each in toluene/hexane (1:1), 1mL/ampul

	Each	5-pk.	10-pk.
	31012	31012-510	
w/data pack	31012-500	31012-520	31112

SV Calibration Mix #7

1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene

2,000µg/mL each in CH₂Cl₂, 1mL/ampul

	Each	5-pk.	10-pk.
	31013	31013-510	
w/data pack	31013-500	31013-520	31113

3,3'-Dichlorobenzidine

2,000µg/mL each in methanol, 1mL/ampul

	Each	5-pk.	10-pk.
	31026	31026-510	
w/data pack	31026-500	31026-520	31126

CLP Semivolatile Calibration Kit #1

(with pesticides)

kit

31007: SV Calibration Mix #1 (anilines)
31008: SV Calibration Mix #2 (phenols)
31009: SV Calibration Mix #3 (base neutrals)
31010: SV Calibration Mix #4 (base neutrals)
31011: SV Calibration Mix #5 (PAHs)
31012: SV Calibration Mix #6 (pesticides)
31013: SV Calibration Mix #7 (dichlorobenzenes)
31026: 3,3'-dichlorobenzidine

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31461	31461-500

CLP Semivolatile Calibration Kit #2

(without pesticides)

kit

31007: SV Calibration Mix #1 (anilines)
31008: SV Calibration Mix #2 (phenols)
31009: SV Calibration Mix #3 (base neutrals)
31010: SV Calibration Mix #4 (base neutrals)
31011: SV Calibration Mix #5 (PAHs)
31013: SV Calibration Mix #7 (dichlorobenzenes)
31026: 3,3'-dichlorobenzidine

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31462	31462-500

Semivolatile Calibration Kit #3

(with benzidine)

kit

31007: SV Calibration Mix #1 (anilines)
31008: SV Calibration Mix #2 (phenols)
31009: SV Calibration Mix #3 (base neutrals)
31010: SV Calibration Mix #4 (base neutrals)
31011: SV Calibration Mix #5 (PAHs)
31013: SV Calibration Mix #7 (dichlorobenzenes)
31030: 605 Benzidines Calibration Mix
(benzidine & 3,3'-dichlorobenzidine)

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31463	31463-500

Semivolatile Organics Kit (3/90 SOW)

kit

31000: SV Screening Mix
31001: SV Tuning Compound
31002: B/N Surrogate Std. Mix (3/90 SOW)
31003: Acid Surrogate Std. Mix (3/90 SOW)
31004: B/N Matrix Spike Mix
31005: Acid Matrix Spike Mix
31006: SV Internal Standard Mix
31007: SV Calibration Mix #1
31008: SV Calibration Mix #2
31009: SV Calibration Mix #3
31010: SV Calibration Mix #4
31011: SV Calibration Mix #5
31012: SV Calibration Mix #6
31013: SV Calibration Mix #7
31026: 3,3'-dichlorobenzidine

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31051	31051-500

Reach for Restek!



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