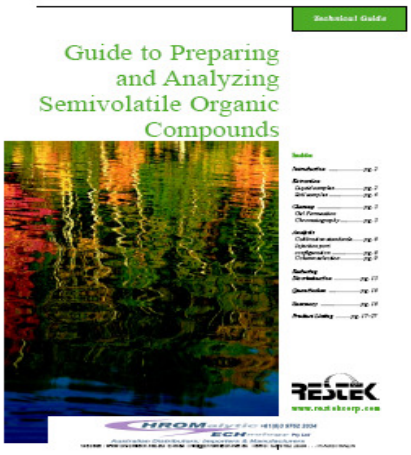
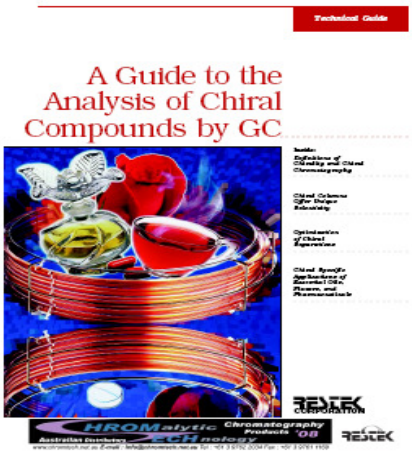
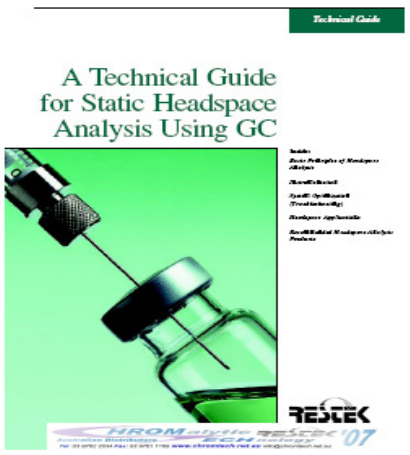
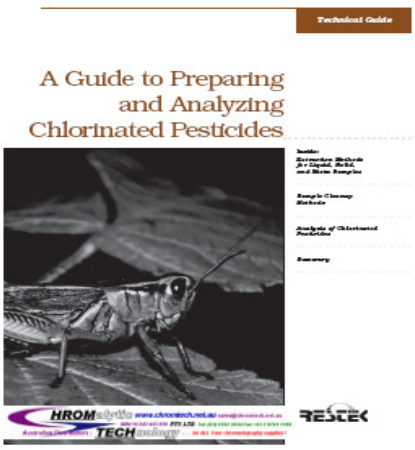
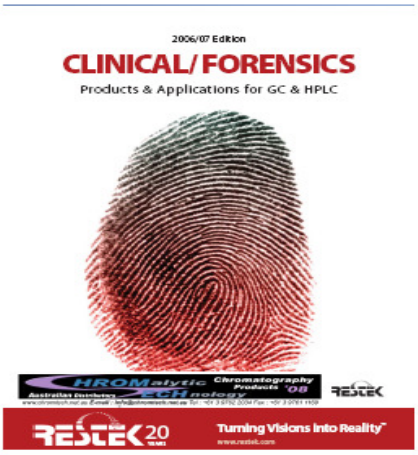
Restek APPLICATIONS Technical Guides

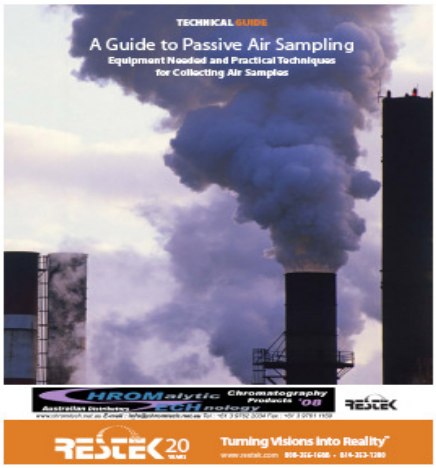


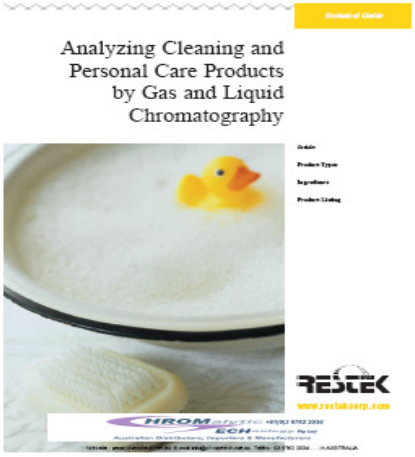


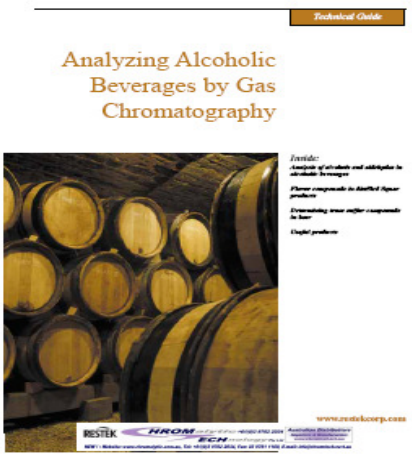


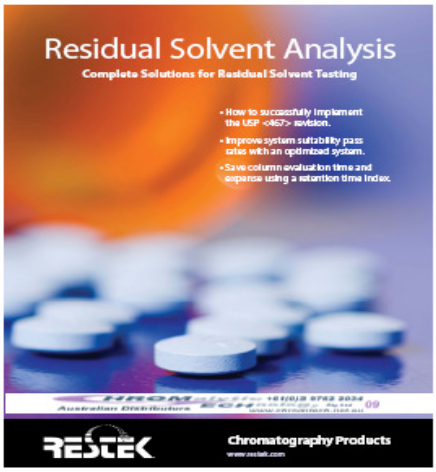


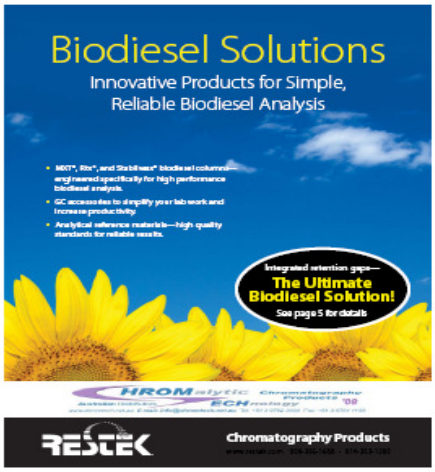












Technical Guide

Guide to Preparing and Analyzing Semivolatile Organic Compounds

Inside:

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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-*n*-propylamine	65	δ-HCH
17	hexachloroethane	66	carbazole
18	nitrobenzene-d5	67	heptachlor
19	nitrobenzene	68	di-*n*-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	*p*-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-*n*-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.

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.

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.

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 cyclohehane, 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 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-chloroanaline, 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.

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.

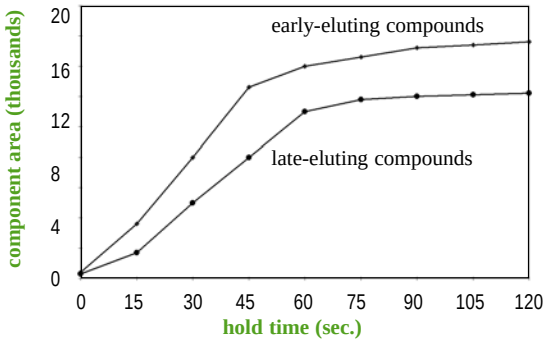
Compound	Elution Volumes (mL)		Compound	Elution Volumes (mL)	
	Start	End		Start	End
2-fluorophenol	201	241	acenaphthene-d10	225	249
phenol-d5	201	249	3-nitroaniline	209	225
phenol	209	225	acenaphthene	225	249
bis(2-chloroethyl)ether	201	225	2,4-dinitrophenol	201	225
2-chlorophenol-d4	209	249	dibenzofuran	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-flurorbiphenyl	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 nitroanalines. 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.

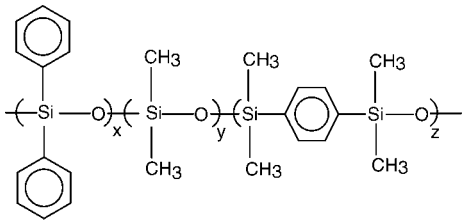
Agilent (HP):	5-7mm from tip of ferrule
Varian 1075/1077:	5.7cm from back of nut
PerkinElmer Autosystem:	4.5 - 5.0cm from back of nut
Shimadzu 14A:	4.0cm from back of nut
Shimadzu 17A:	35mm from tip of ferrule
split:	40mm from back of nut
splitless:	64mm from back of nut

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).

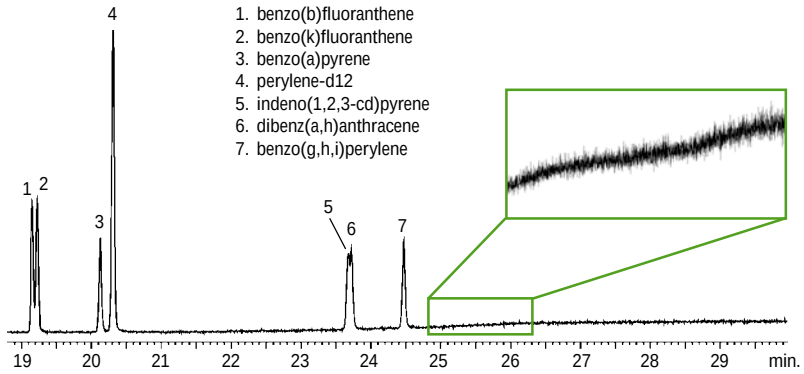
Low-bleed columns are necessary for the more sensitive instruments. For laboratories using the Agilent 5973 GC/MS or ion trap MS, column bleed can be a very important issue. As these instruments have become more sensitive, the higher-bleed columns produce a larger signal on the detector and can cause 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.



30m, 0.25mm ID, 0.5µm Rtx®-5Sil MS (cat# 12738)
20ng 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 @ 1.0mL/min. constant flow; **Inj. temp.:** 300°C; **Det. temp.:** 280°C

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.

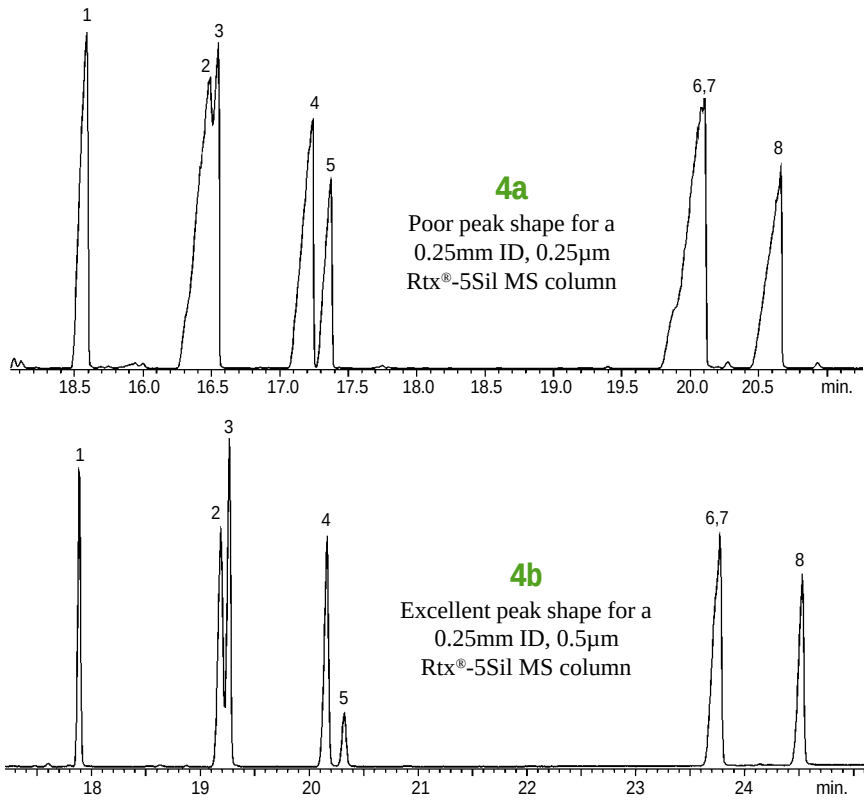
Column capacity also must be addressed when optimizing the analysis. The typical calibration range for many of these methods is 20 to 160ng per compound. This requires a column stationary phase and diameter that will not overload with a 160ng or larger injection. Because there is a loss of analyte in 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

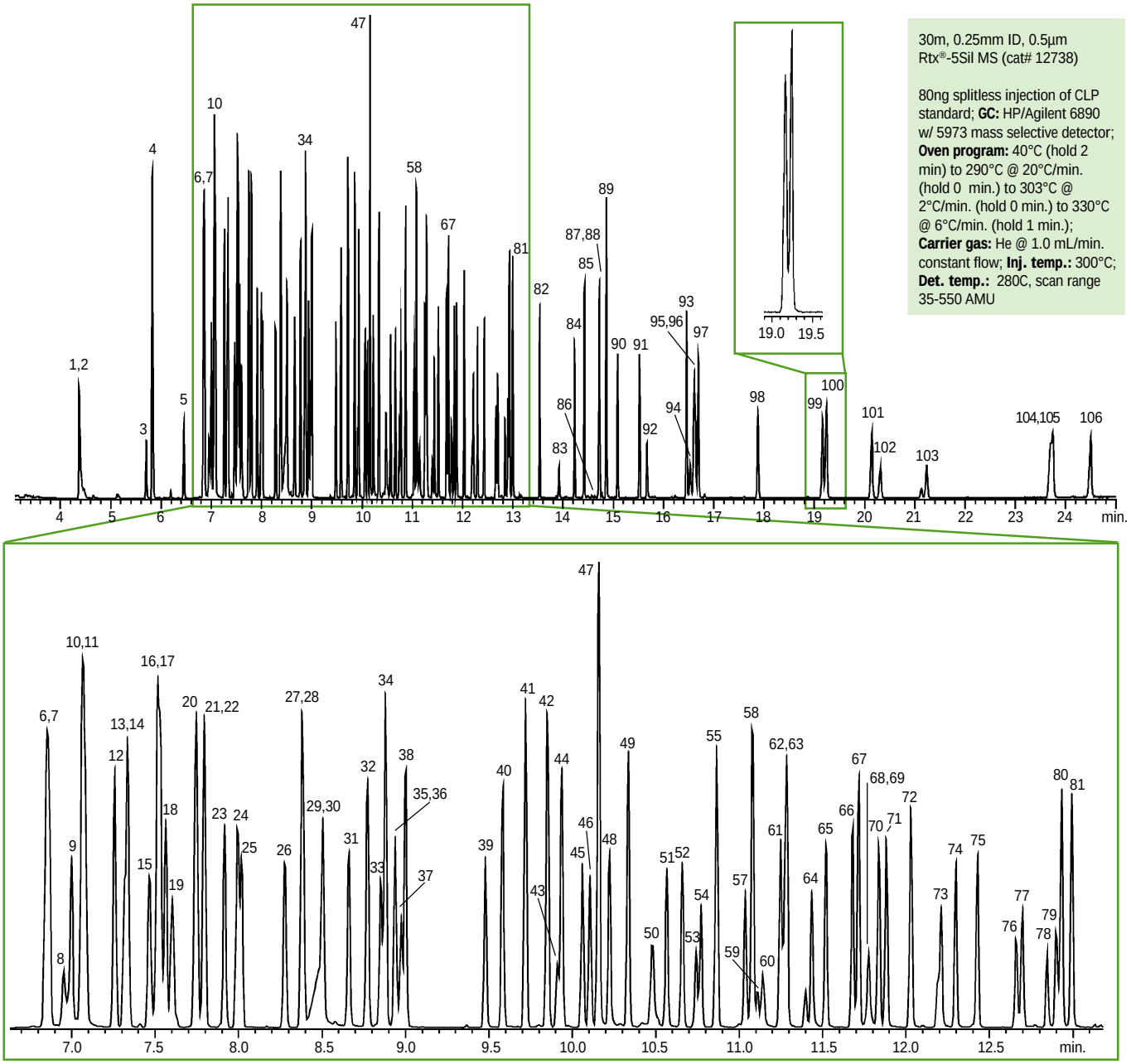
30m, 0.25mm ID, 0.25µm Rtx®-5Sil MS (cat.# 12723)
unknown concentration (>160ng) splitless injection of CLP standard; **GC:** HP/Agilent 6890 w/ 5973 mass selective detector, scan range 35-550 AMU; **Oven program:** 40°C (hold 2 min.) to 245°C @ 25°C/min. (hold 0 min.) to 330°C @ 6°C/min. (hold 5 min.); **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.

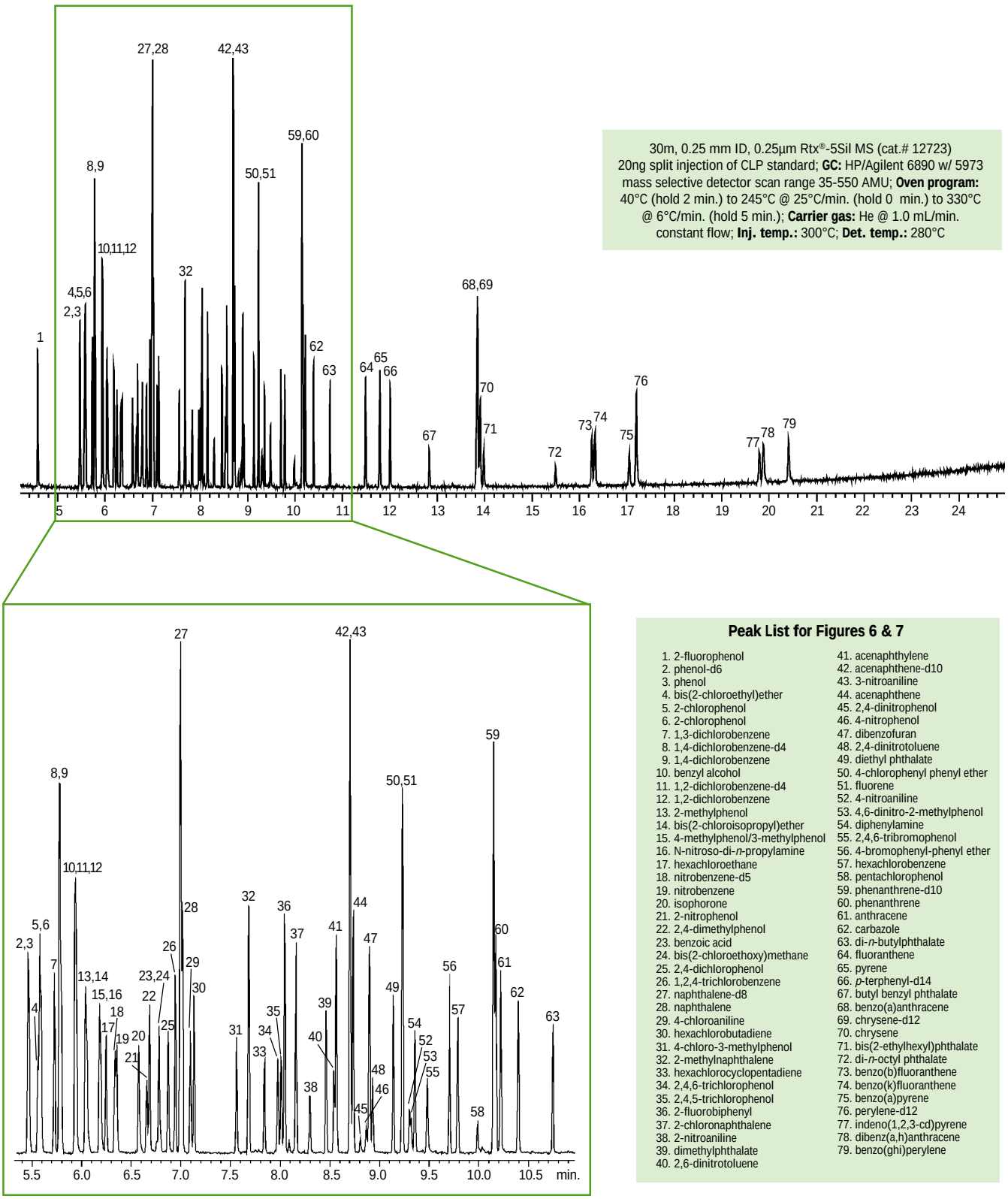
If the sensitivity of an instrument allows for split injection, then column capacity is not nearly the issue it is for splitless injection. Figure 7 shows a 160ng-per-component standard injected under the same conditions as shown in Figure 6. A column with a thinner film can be used because the concentration 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
2. pyridine
3. methyl methanesulfonate
4. 2-fluorophenol
5. ethyl methanesulfonate
6. phenol-d6
7. phenol
8. aniline
9. bis(2-chloroethyl)ether
10. 2-chlorophenol
11. 2-chlorophenol
12. 1,3-dichlorobenzene
13. 1,4-dichlorobenzene-d4
14. 1,4-dichlorobenzene
15. benzyl alcohol
16. 1,2-dichlorobenzene-d4
17. 1,2-dichlorobenzene
18. 2-methylphenol
19. bis(2-chloroisopropyl)ether
20. 4-methylphenol/3-methylphenol
21. N-nitroso-di-*n*-propylamine
22. acetophenone
23. hexachloroethane
24. nitrobenzene-d5
25. nitrobenzene
26. isophorone
27. 2,4-dimethylphenol
28. 2-nitrophenol
29. benzoic acid
30. bis(2-chloroethoxy)methane
31. 2,4-dichlorophenol
32. 1,2,4-trichlorobenzene
33. naphthalene-d8
34. naphthalene
35. 2,6-dichlorophenol
36. 4-chloroaniline
37. hexachloropropene
38. hexachlorobutadiene
39. 4-chloro-3-methylphenol
40. isosafrole
41. 2-methylnaphthalene
42. 1-methylnaphthalene
43. hexachlorocyclopentadiene
44. 1,2,4,5-tetrachlorobenzene
45. 2,4,6-trichlorophenol
46. 2,4,5-trichlorophenol
47. 2-fluorobiphenyl
48. safrole
49. 2-chloronaphthalene
50. 2-nitroaniline
51. 1,4-naphthoquinone
52. dimethylphthalate
53. 1,3-dinitrobenzene
54. 2,6-dinitrotoluene
55. acenaphthylene
56. 3-nitroaniline
57. acenaphthene-d10
58. acenaphthene
59. 2,4-dinitrophenol
60. 4-nitrophenol
61. pentachlorobenzene
62. 2,4-dinitrotoluene
63. dibenzofuran
64. 2,3,4,6-tetrachlorophenol
65. diethyl phthalate
66. 4-chlorophenyl phenyl ether
67. fluorene
68. 4-nitroaniline
69. 4,6-dinitro-2-methylphenol
70. diphenylamine
71. azobenzene
72. 2,4,6-tribromophenol
73. phenacetin
74. 4-bromophenyl-phenyl ether
75. hexachlorobenzene
76. pentachlorophenol
77. pentachloronitrobenzene
78. dinoseb
79. phenanthrene-d10
80. phenanthrene
81. anthracene
82. di-*n*-butylphthalate
83. 4-nitroquinoline-1-oxide
84. isodrin
85. fluoranthene
86. benzidine
87. pyrene
88. aromite
89. *p*-terphenyl-d14
90. chlorbenzilate
91. butyl benzyl phthalate
92. kepone
93. bis(2-ethylhexyl)phthalate
94. 3,3'-dichlorobenzidine
95. benzo(a)anthracene
96. chrysene-d12
97. chrysene
98. di-*n*-octyl phthalate
99. benzo(b)fluoranthene
100. benzo(k)fluoranthene
101. benzo(a)pyrene
102. perylene-d12
103. 3-methylcholanthrene
104. indeno(1,2,3-cd)pyrene
105. dibenz(a,h)anthracene

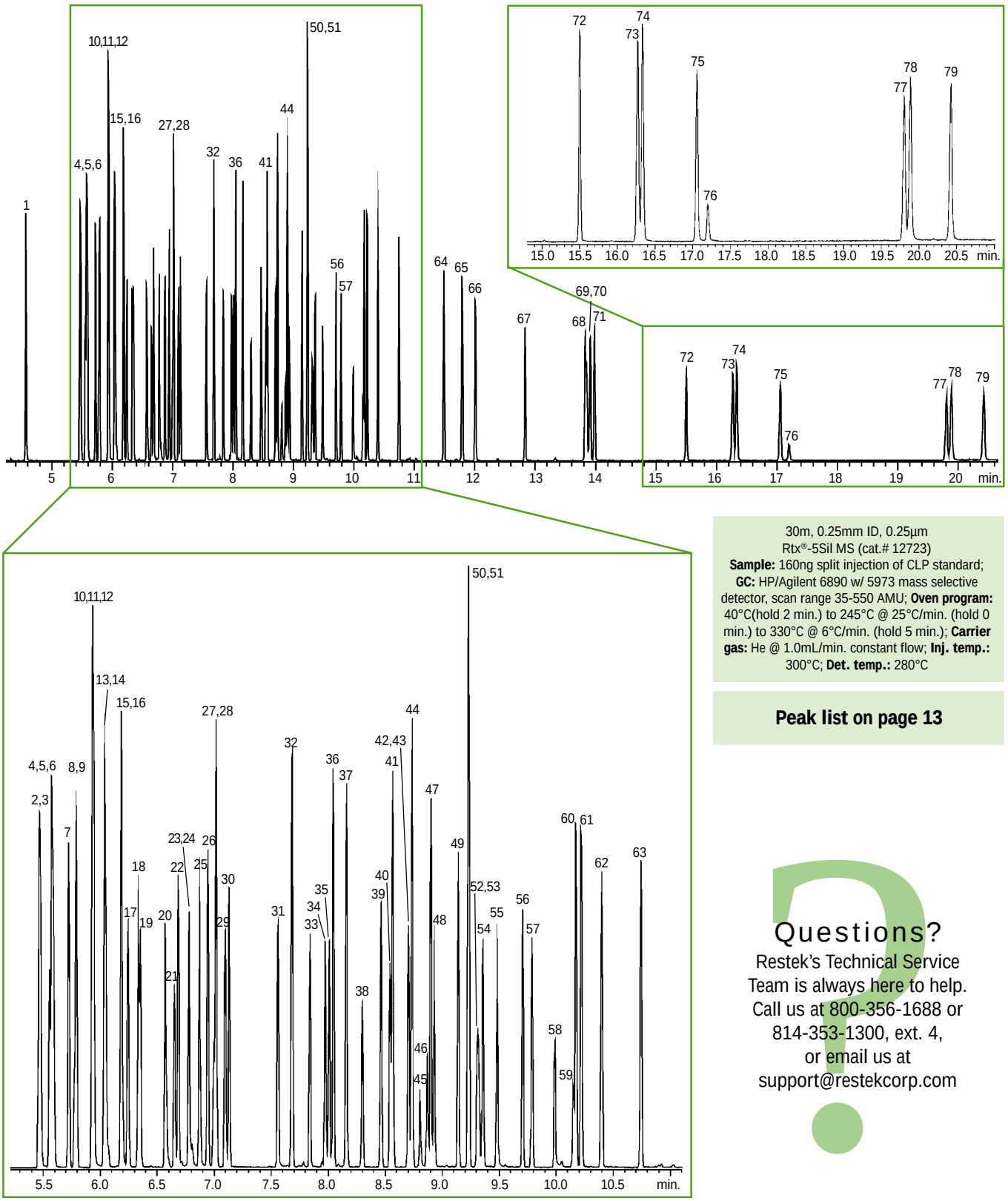
Figure 6. A 20ng split injection shows the extremely low bleed of the Rtx®-5Sil MS column.



Peak List for Figures 6 & 7

1. 2-fluorophenol
2. phenol-d6
3. phenol
4. bis(2-chloroethyl)ether
5. 2-chlorophenol
6. 2-chlorophenol
7. 1,3-dichlorobenzene
8. 1,4-dichlorobenzene-d4
9. 1,4-dichlorobenzene
10. benzyl alcohol
11. 1,2-dichlorobenzene-d4
12. 1,2-dichlorobenzene
13. 2-methylphenol
14. bis(2-chloroisopropyl)ether
15. 4-methylphenol/3-methylphenol
16. N-nitroso-di-*n*-propylamine
17. hexachloroethane
18. nitrobenzene-d5
19. nitrobenzene
20. isophorone
21. 2-nitrophenol
22. 2,4-dimethylphenol
23. benzoic acid
24. bis(2-chloroethoxy)methane
25. 2,4-dichlorophenol
26. 1,2,4-trichlorobenzene
27. naphthalene-d8
28. naphthalene
29. 4-chloroaniline
30. hexachlorobutadiene
31. 4-chloro-3-methylphenol
32. 2-methylnaphthalene
33. hexachlorocyclopentadiene
34. 2,4,6-trichlorophenol
35. 2,4,5-trichlorophenol
36. 2-fluorobiphenyl
37. 2-chloronaphthalene
38. 2-nitroaniline
39. dimethylphthalate
40. 2,6-dinitrotoluene
41. acenaphthylene
42. acenaphthene-d10
43. 3-nitroaniline
44. acenaphthene
45. 2,4-dinitrophenol
46. 4-nitrophenol
47. dibenzofuran
48. 2,4-dinitrotoluene
49. diethyl phthalate
50. 4-chlorophenyl phenyl ether
51. fluorene
52. 4-nitroaniline
53. 4,6-dinitro-2-methylphenol
54. diphenylamine
55. 2,4,6-tribromophenol
56. 4-bromophenyl-phenyl ether
57. hexachlorobenzene
58. pentachlorophenol
59. phenanthrene-d10
60. phenanthrene
61. anthracene
62. carbazole
63. di-*n*-butylphthalate
64. fluoranthene
65. pyrene
66. *p*-terphenyl-d14
67. butyl benzyl phthalate
68. benzo(a)anthracene
69. chrysene-d12
70. chrysene
71. bis(2-ethylhexyl)phthalate
72. di-*n*-octyl phthalate
73. benzo(b)fluoranthene
74. benzo(k)fluoranthene
75. benzo(a)pyrene
76. perylene-d12
77. indeno(1,2,3-cd)pyrene
78. dibenz(a,h)anthracene
79. benzo(ghi)perylene

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

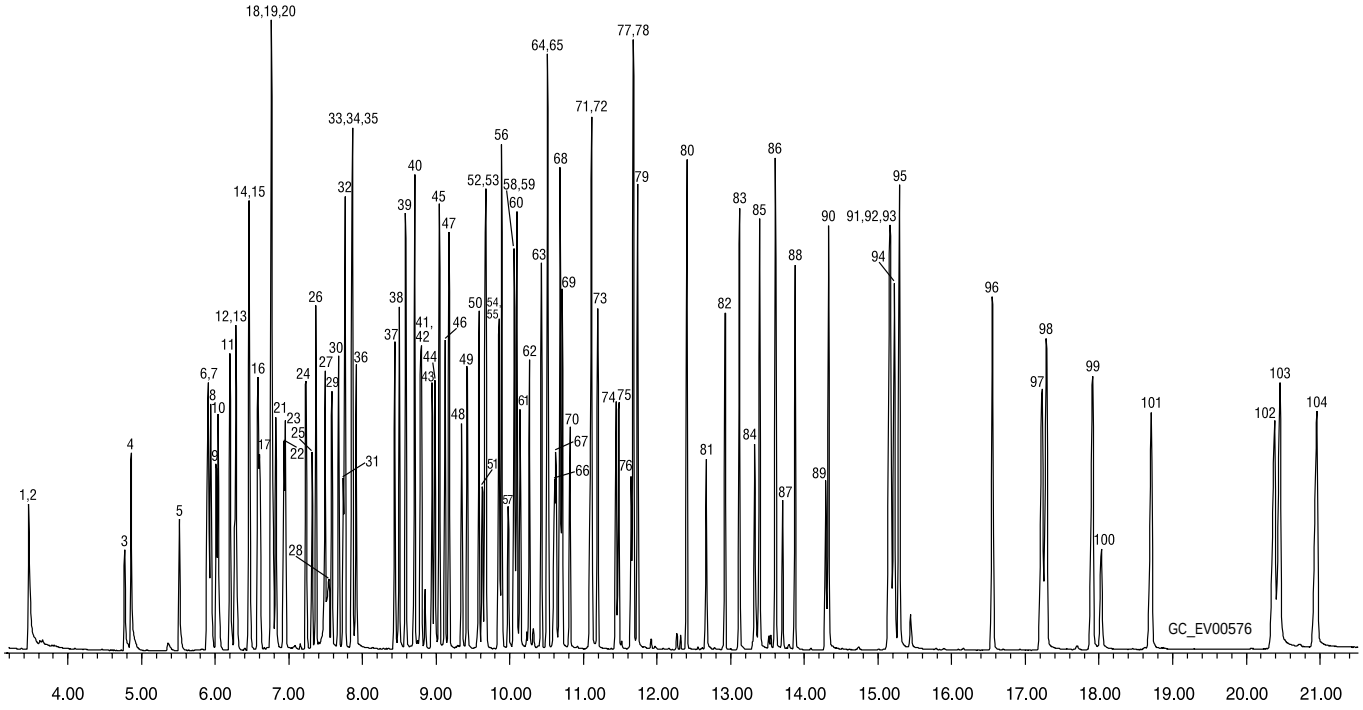
Questions?

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
2. pyridine
3. methyl methanesulfonate
4. 2-fluorophenol
5. ethyl methanesulfonate
6. phenol-d6
7. phenol
8. aniline
9. bis(2-chloroethyl)ether
10. 2-chlorophenol
11. 1,3-dichlorobenzene
12. 1,4-dichlorobenzene-d4
13. 1,4-dichlorobenzene
14. 1,2-dichlorobenzene
15. benzyl alcohol
16. 2-methylphenol
17. bis(2-chloroisopropyl)ether
18. acetophenone
19. 4-methylphenol/3-methylphenol
20. N-nitroso-di-*n*-propylamine
21. hexachloroethane
22. nitrobenzene-d5
23. nitrobenzene
24. isophorone
25. 2-nitrophenol
26. 2,4-dimethylphenol
27. bis(2-chloroethoxy)methane
28. benzoic acid
29. 2,4-dichlorophenol
30. 1,2,4-trichlorobenzene
31. naphthalene-d8
32. naphthalene
33. 2,6-dichlorophenol
34. 4-chloroaniline
35. hexachloropropene
36. hexachlorobutadiene
37. 4-chloro-3-methylphenol
38. isosafrole
39. 2-methylnaphthalene
40. 1-methylnaphthalene
41. hexachlorocyclopentadiene
42. 1,2,4,5-tetrachlorobenzene
43. 2,4,6-trichlorophenol
44. 2,4,5-trichlorophenol
45. 2-fluorobiphenyl
46. safrole
47. 2-chloronaphthalene
48. 2-nitroaniline
49. 1,4-naphthoquinone
50. dimethylphthalate
51. 1,3-dinitrobenzene
52. 2,6-dinitrotoluene
53. acenaphthylene
54. acenaphthene-d10
55. 3-nitroaniline
56. acenaphthene
57. 2,4-dinitrophenol
58. pentachlorobenzene
59. 4-nitrophenol
60. dibenzofuran
61. 2,4-dinitrotoluene
62. 2,3,4,6-tetrachlorophenol
63. diethyl phthalate
64. fluorene
65. 4-chlorophenyl phenyl ether
66. 4-nitroaniline
67. 4,6-dinitro-2-methylphenol
68. diphenylamine
69. azobenzene
70. 2,4,6-tribromophenol
71. phenacetin
72. 4-bromophenyl phenyl ether
73. hexachlorobenzene
74. pentachlorophenol
75. pentachloronitrobenzene
76. phenanthrene-d10
77. dinoseb
78. phenanthrene
79. anthracene
80. di-*n*-butylphthalate
81. 4-nitroquinoline-1-oxide
82. isodrin
83. fluoranthene
84. benzidine
85. pyrene
86. *p*-terphenyl-d14
87. aramite
88. chlorbenzilate
89. kepone
90. butyl benzyl phthalate
91. benzo(a)anthracene
92. 3,3'-dichlorobenzidine
93. chrysene-d12
94. chrysene
95. bis(2-ethylhexyl)phthalate
96. di-*n*-octyl phthalate
97. benzo(b)fluoranthene
98. benzo(k)fluoranthene
99. benzo(a)pyrene
100. perylene-d12
101. 3-methylcholanthrene
102. indeno(1,2,3-cd)pyrene
103. dibenzo(a,h)anthracene
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, 31206, 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° @ 6°C/min. (hold 1 min.)
Det. type:	MS
Transfer line temp.:	280°C
Scan range:	35 to 550amu
Ionization:	EI
Mode:	full scan

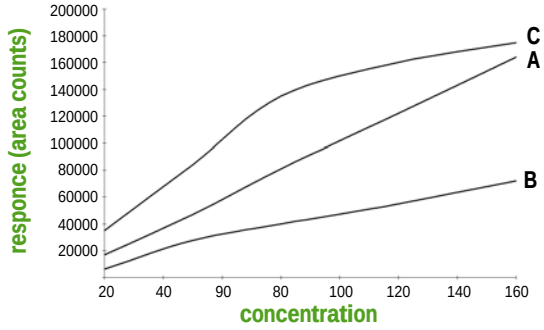
Quantitation

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 curve like Curve C is observed, select a column with higher capacity or reduce detector sensitivity by lowering the multiplier voltage.

Figure 9. Calibration curves showing linear response, adsorption, and high-end roll-off.



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 senstivity 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.

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

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.

Phases currently available as Integra-Guard™ columns:

Rtx®-1
Rtx®-1MS
Rtx®-5
Rtx®-5MS
Rtx®-5Sil MS
XTI®-5
Rtx®-1301
Rtx®-624
Rtx®-1701
Rtx®-Volatiles
Rtx®-20
Rtx®-35
Rtx®-35MS
Rtx®-BAC 1 & 2
Stabilwax®

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.

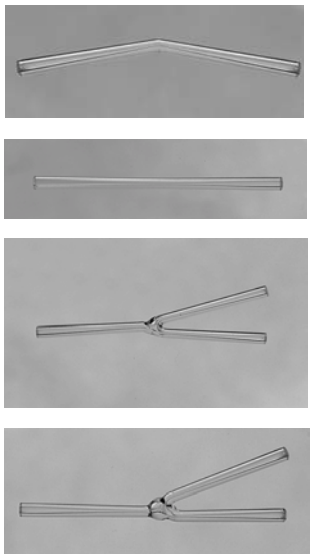
for*more*info

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)	cat.# ea.	cat.# 5-pk.	cat.# 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)	cat.# ea.	cat.# 5-pk.	cat.# 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)	cat.# ea.	cat.# 5-pk.	cat.# 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

*Nor

Inlet Liners

For Shimadzu GCs

Splitless Liners for Shimadzu GCs	Benefits/Uses:	ID**/OD & Length (mm)	cat.# ea.	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

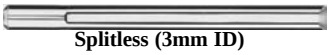
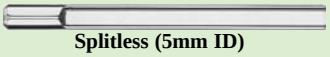
For PerkinElmer GCs

COLUMN INSTALLS THIS END

Splitless Liners for PerkinElmer GCs	Benefits/Uses:	ID**/OD & Length (mm)	cat.# ea.	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

COLUMN INSTALLS THIS END

Splitless Liners for 5000-6000 Series GCs	Benefits/Uses:	ID**/OD & Length (mm)	cat.# ea.	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.**	**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
**Nomi

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.

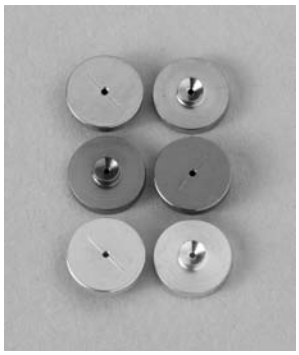
cat.# 20790, (10 grams)

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.

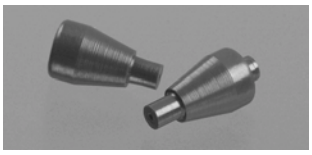
Septum Diameter	25-pk.	50-pk.	100-pk.
5mm (3/16")	20351	20352	20353
6mm (1/4")	20355	20356	20357
7mm	20381	20382	20383
8mm	20370	20371	—
9.5mm (3/8")	20359	20360	20361
10mm	20378	20379	20380
11mm (7/16")	20363	20364	20365
11.5mm	22385	22386	22387
12.5mm (1/2")	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Plug	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).

free guide

EZ-Vent™ 2000 for Agilent GCs

EZ-Vent™ 2000 for Varian GCs



EZ-Vent™ 2000 ferrules

EZ-Vent™ 2000

great idea!

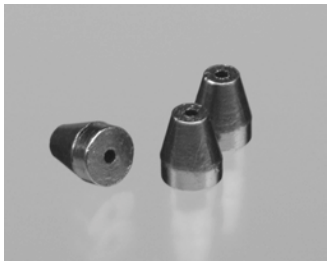
- Change GC/MS columns in minutes without venting.
- Silcosteel® treated for greater inertness.
- Deactivated transfer line minimizes bleed into the source.
- Stainless steel body and high-temperature polyimide ferrules minimize leaks at the problematic transfer line fitting.
- Less expensive than other "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	Vespel®/ 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	Vespel®/ 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*

Weldment for Agilent 5890 GCs

Shell weldment for Agilent 5890 GCs

Weldment and shell weldment for Agilent 6890/6850 GCs

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

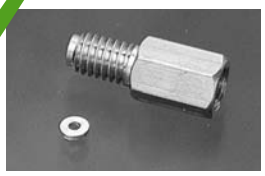
Description	cat.#, (ea.)
Replacement Weldment for Agilent 6890/6850 GCs with EPC	22674
Replacement Weldment for Agilent 6890/6850 GCs with manual flow	20265
Replacement Shell Weldment for Agilent 6890/6850 GCs	22673

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.

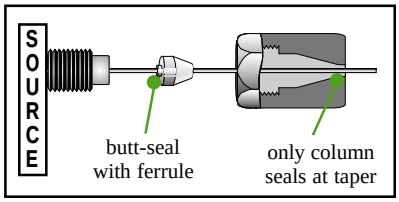
MSD Conversion Fitting: cat.# 21314, (ea.)
MSD Conversion Fitting Replacement Ring Seal: cat.# 21313, (2-pk.)

check it out

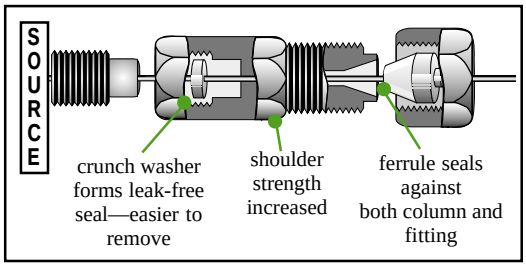


Agilent's MSD interface requires a butt-seal at the base of a Vespel® ferrule, which is prone to leakage. Restek's version uses a standard ferrule design that simultaneously seals the fitting and capillary tubing with compressive forces.

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.)

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.

US EPA Methods 8270C & 8270D Analytical Reference Materials

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

Internal Standard Mixtures

SV Internal Standard Mix

acenaphthene-d10
chrysene-d12
1,4-dichlorobenzene-d4
naphthalene-d8
perylene-d12
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
nitrobenzene-d5
p-terphenyl-d14

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
phenol-d6
2,4,6-tribromophenol

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
1,4-dichlorobenzene
2,4-dinitrotoluene
N-nitroso-di-*n*-propylamine
pyrene
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
2-chlorophenol
4-nitrophenol
pentachlorophenol
phenol

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
4,4'-DDT
DFTPP
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

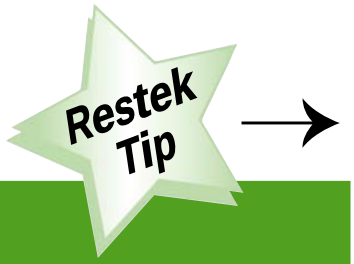
Revised GPC Calibration Mix

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



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	
bis(2-chloroethyl)ether	hexachloroethane
bis-(2-chloroisopropyl)ether	ideno(1,2,3-cd)pyrene
bis(2-ethylhexyl)phthalate	isophorone
4-bromophenyl phenyl ether	2-methylnaphthalene
butyl benzyl phthalate	naphthalene
caprolactam	nitrobenzene
carbazole	*n*-nitrosodi-*n*-propylamine
2-chloronaphthalene	*n*-nitrosodiphenylamine
4-chlorophenyl phenyl ether	phenanthrene
chrysene	pyrene
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)

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)

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™

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
4-chloroaniline
2-nitroaniline
3-nitroaniline
4-nitroaniline

2,000µg/mL each in CH_2Cl_2, 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-chloro-3-methylphenol
2-chlorophenol
2,4-dichlorophenol
2,4-dimethylphenol
2,4-dinitrophenol
2-methyl-4,6-dinitrophenol
2-methylphenol
4-methylphenol
2-nitrophenol
4-nitrophenol
pentachlorophenol
phenol
2,4,5-trichlorophenol
2,4,6-trichlorophenol

2,000µg/mL each in CH_2Cl_2, 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
bis(2-chloroethyl)ether
bis(2-chloroisopropyl)ether
bis(2-ethylhexyl)phthalate
4-bromophenyl phenyl ether
butyl benzyl phthalate
2-chloronaphthalene
4-chlorophenyl phenyl ether
dimethyl phthalate
di-*n*-butyl phthalate
di-*n*-octyl phthalate
N-nitrosodimethylamine
N-nitroso-di-*n*-propylamine
N-nitrosodiphenylamine

2,000µg/mL each in CH_2Cl_2, 1mL/ampul

	Each	5-pk.	10-pk.
	31009	31009-510	
w/data pack	31009-500	31009-520	31109

SV Calibration Mix #4

carbazole
hexachlorocyclopentadiene
dibenzofuran
diethyl phthalate
2,4-dinitrotoluene
2,6-dinitrotoluene
hexachlorobenzene
hexachlorobutadiene
hexachloroethane
isophorone
2-methylnaphthalene
nitrobenzene
1,2,4-trichlorobenzene

2,000µg/mL each in CH_2Cl_2, 1mL/ampul

	Each	5-pk.	10-pk.
	31010	31010-510	
w/data pack	31010-500	31010-520	31110

SV Calibration Mix #5

acenaphthene
acenaphthylene
anthracene
benzo(a)anthracene
benzo(a)pyrene
benzo(b)fluoranthene
benzo(k)fluoranthene
benzo(ghi)perylene
chrysene
dibenzo(a,h)anthracene
fluoranthene
fluorene
indeno(1,2,3-cd)pyrene
naphthalene
phenanthrene
pyrene

2,000µg/mL each in CH_2Cl_2, 1mL/ampul

	Each	5-pk.	10-pk.
	31011	31011-510	
w/data pack	31011-500	31011-520	31111

SV Calibration Mix #6

aldrin
α-BHC
β-BHC
δ-BHC
γ-BHC (lindane)
4,4'-DDD
4,4'-DDE
4,4'-DDT
dieldrin
endosulfan I
endosulfan II
endosulfan sulfate
endrin
endrin aldehyde
endrin ketone
heptachlor
heptachlor epoxide (B)
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 CH_2Cl_2, 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	31151

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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: www.restekcorp.com

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.

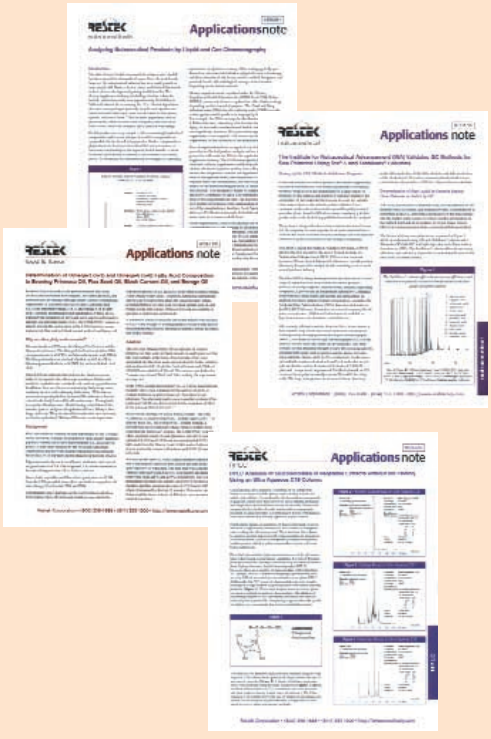
High-Resolution Analyses of Fatty Acid Methyl Esters (FAMEs) by Gas Chromatography (lit. cat.# 59489A)
Characterizing fats and oils, or determining fat content in food, calls for highly efficient separations of fatty acid methyl esters (FAMEs) on capillary GC columns. A properly chosen column can provide 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

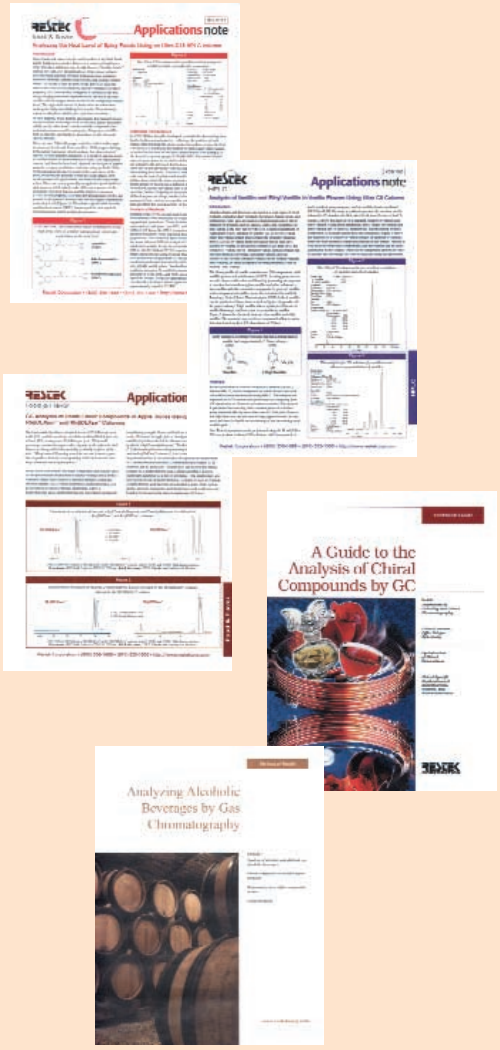


Analyzing Nutraceutical Products by Liquid and Gas Chromatography (lit. cat.# 59364)
HPLC and GC methodologies meet the varied challenges of herbal products analysis
HPLC analyses of allicin in garlic, hyperforin in St. John's wort, and phenolics in Echinacea, and GC analysis of fatty acids in saw palmetto, 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 o
mega-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-ßDEXsm™ and Rt-ßDEXse™ 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.

Chromatogram Search Wizard

Visit us online at www.restekcorp.com/cgrmsrch.htm to search Restek's entire on-line chromatogram archive of both GC and HPLC applications!

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.

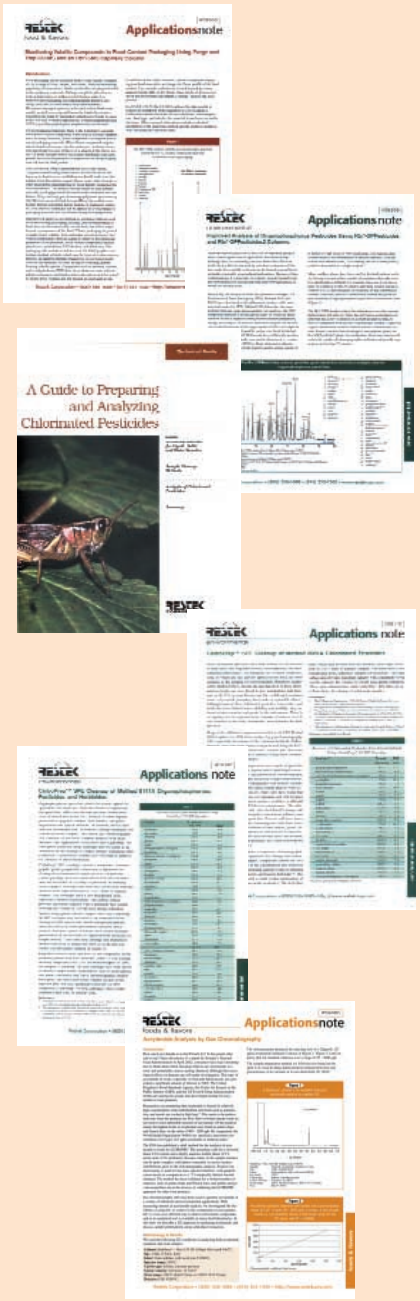
Acrylamide Analysis by Gas Chromatography
(lit. cat.# 59485)
GC is a simple, low-cost, efficient way of detecting acrylamide in prepared foods
A proposed LC/MS/MS method for analyses of acrylamide in foods requires reversed phase HPLC in a highly aqueous mobile phase, a positive ion electrospray MS interface, and quantification based on comparison to a ^{13}C-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.



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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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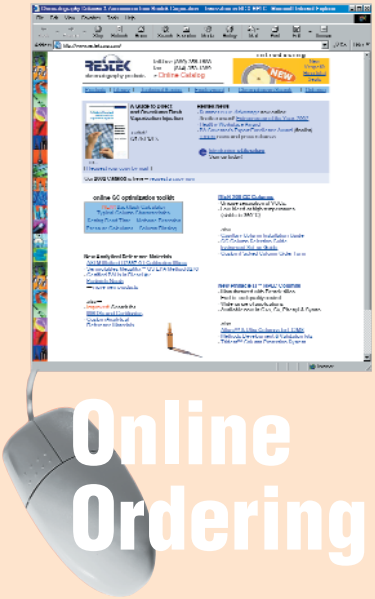

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RESTEK

Technical Guide

Analyzing Cleaning and Personal Care Products by Gas and Liquid Chromatography

Inside:

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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 heath. 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.

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 cleaners, 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 (carboxylate 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.

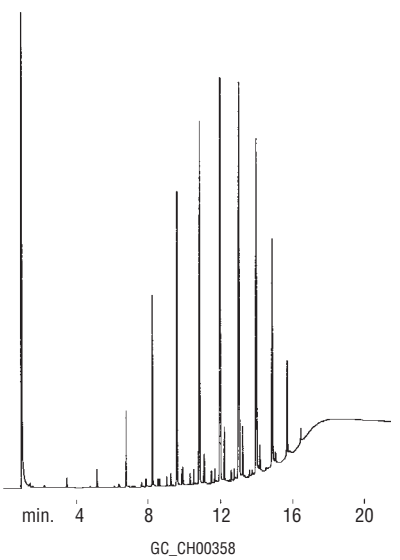
Fatty acids that make up a soap can be analyzed either in the free fatty acid form or after derivatization to the methyl esters (FAMEs). Figure 2 shows an analysis of free fatty acids by GC, using a Stabilwax®-DA capillary column. The acid-deactivated phase in the Stabilwax® DA column gives excellent 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

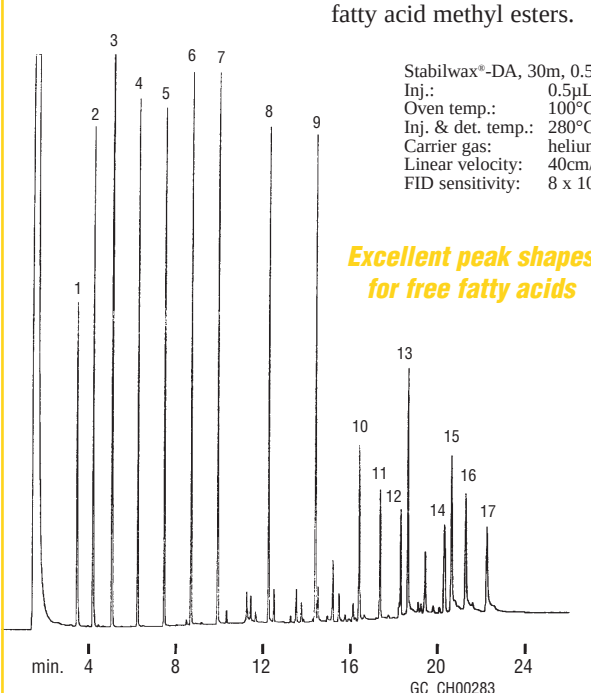
Triton® X-100 surfactant separated by number of ethylene oxide units on an MXT®-1 column.



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.



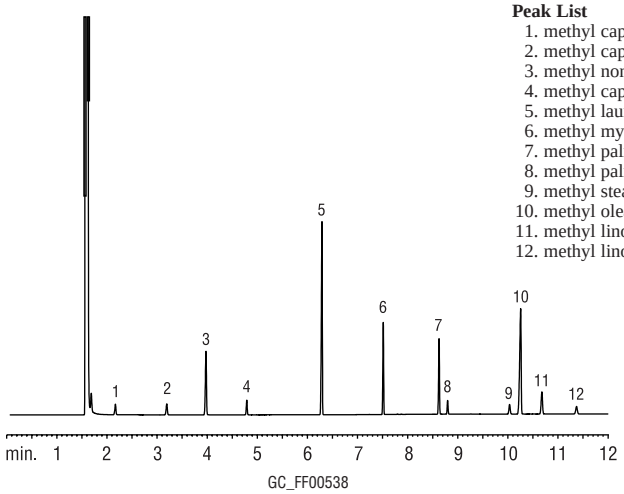
Stabilwax®-DA, 30m, 0.53mm ID, 0.25µm (cat.# 11025)
Inj.: 0.5µL direct injection of a 5mg/mL standard
Oven temp.: 100°C (hold 2 min.) to 250°C @ 8°C/min.
Inj. & det. temp.: 280°C
Carrier gas: helium
Linear velocity: 40cm/sec. (flow rate: 5.2cc/min.)
FID sensitivity: 8 x 10^{-11} AFS

Excellent peak shapes for free fatty acids

1. C2:0 - acetic acid
2. C3:0 - propionic acid
3. C4:0 - butyric acid
4. C5:0 - valeric acid
5. C6:0 - caproic acid
6. C7:0 - heptanoic acid
7. C8:0 - caprylic acid
8. C10:0 - capric acid
9. C12:0 - lauric acid
10. C14:0 - myristic acid
11. C15:0 - pentadecanoic acid
12. C16:0 - palmitic acid
13. C16:1 - palmitoleic acid
14. C18:0 - stearic acid
15. C18:1 - oleic acid
16. C18:2 - linoleic acid
17. C18:3 - linolenic acid

Figure 3

FAMEs analysis on an Rtx®-Wax column.

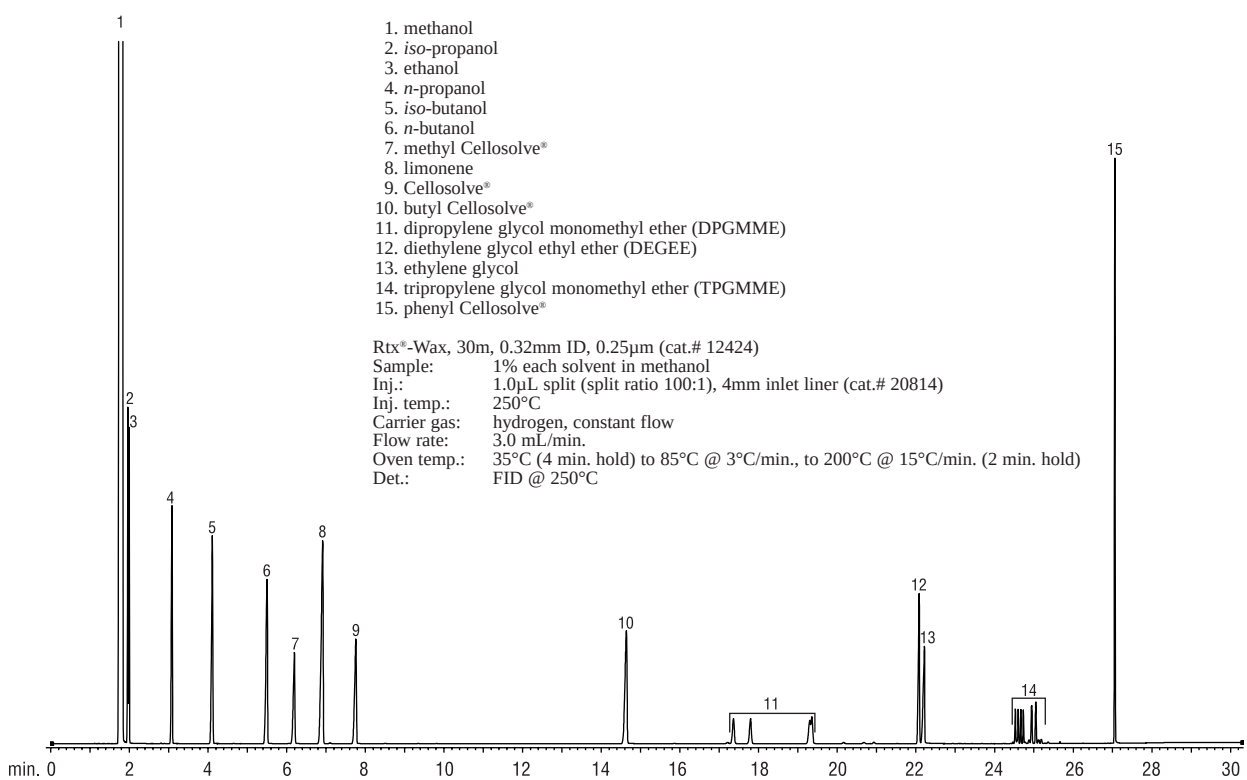


Peak List	Conc. (mg/mL)
1. methyl caproate (C6:0)	0.4
2. methyl caprylate (C8:0)	0.4
3. methyl nonanoate (C9:0)	2.0
4. methyl caprate (C10:0)	0.4
5. methyl laurate (C12:0)	5.0
6. methyl myristate (C14:0)	2.0
7. methyl palmitate (C16:0)	2.0
8. methyl palmitoleate (C16:1)	0.4
9. methyl stearate (C18:0)	0.4
10. methyl oleate (C18:1)	5.0
11. methyl linoleate (C18:2)	1.0
12. methyl linolenate (C18:3)	0.4

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.)

Figure 4

Alcohols, glycols, and other cleaning solvents can be quantified, using an Rtx®-Wax colum.



1. methanol
2. *iso*-propanol
3. ethanol
4. *n*-propanol
5. *iso*-butanol
6. *n*-butanol
7. methyl Cellosolve®
8. limonene
9. Cellosolve®
10. butyl Cellosolve®
11. dipropylene glycol monomethyl ether (DPGMME)
12. diethylene glycol ethyl ether (DEGEE)
13. ethylene glycol
14. tripropylene glycol monomethyl ether (TPGMME)
15. phenyl Cellosolve®

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.

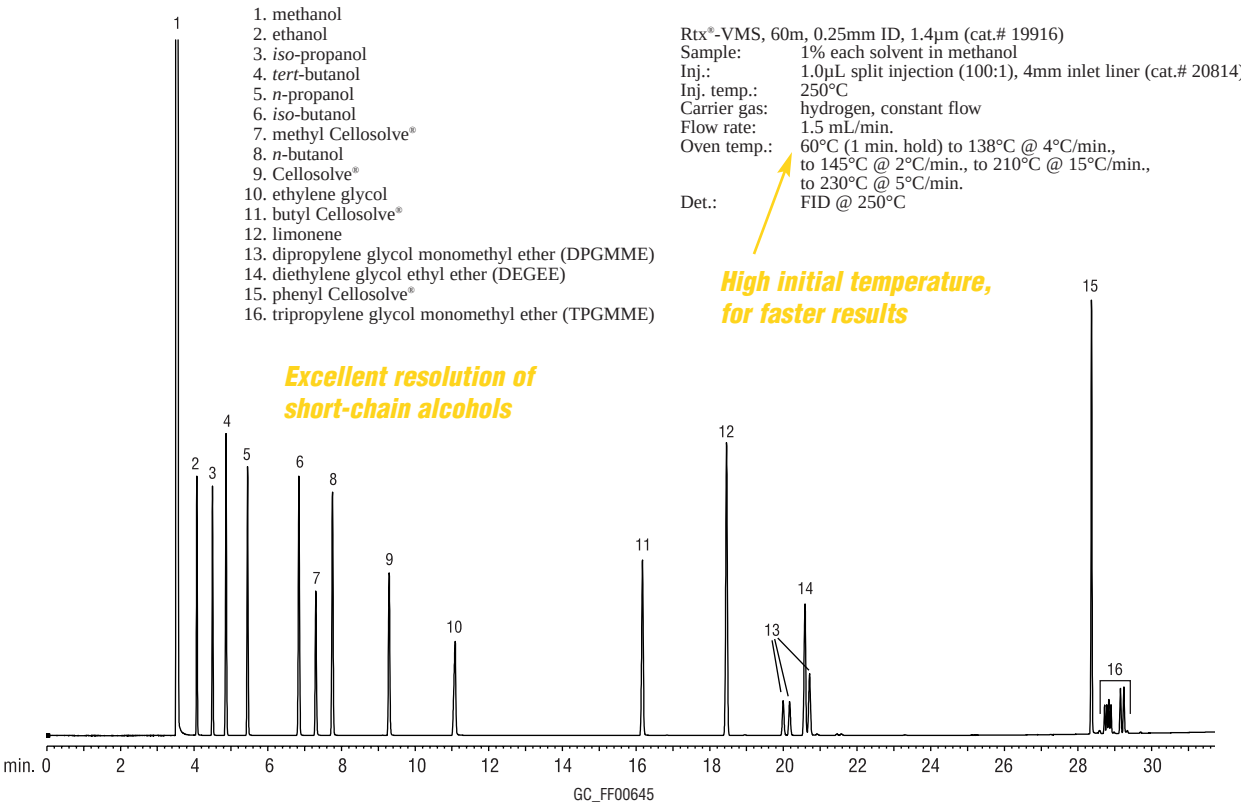
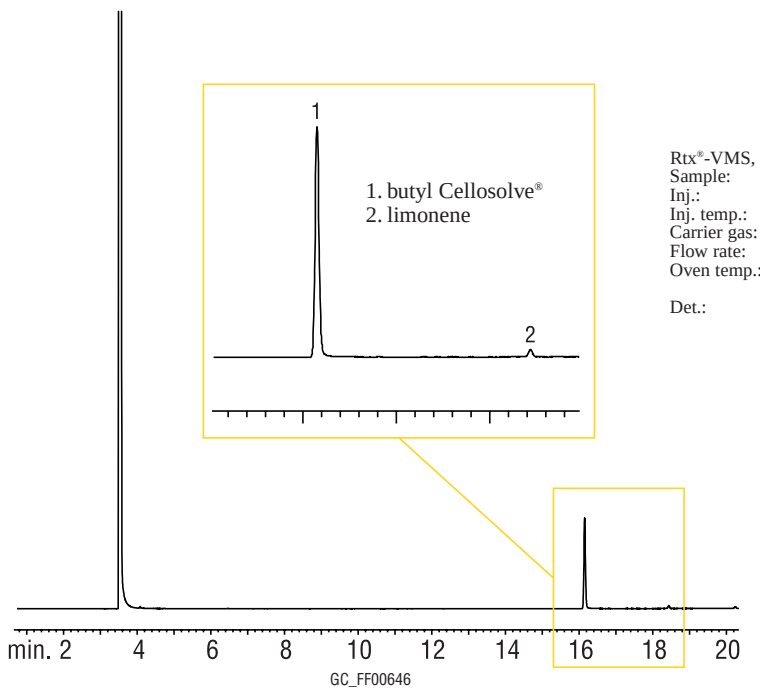


Figure 6

Quantify volatile ingredients in an all-purpose cleaner, using an Rtx®-VMS column.



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

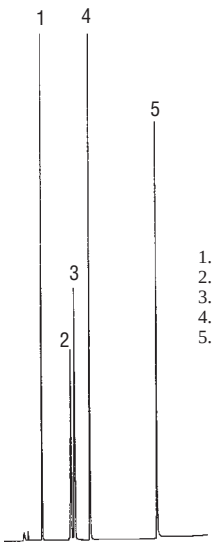
glycols are popular choices. Cleaning solvents can be resolved using an Rtx®-Wax column (Figure 4) or an Rtx®-VMS column (Figure 5). The latter column gives excellent selectivity and peak shape for a wide range of cleaning solvents. Analysis of an all-purpose cleaner is shown in Figure 6, and a glass 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 8

Glycols and alcohols on an ultra-low-bleed column.



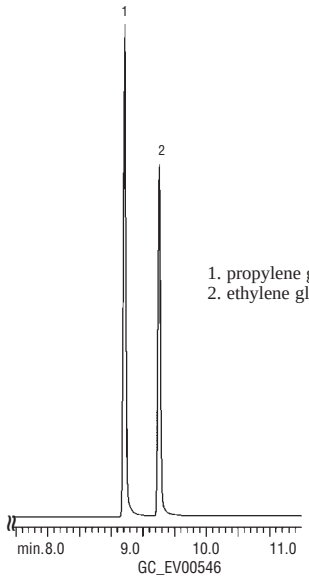
1. *n*-butyl alcohol
2. ethylene glycol
3. propylene glycol
4. butyl Cellosolve®
5. butyl carbitol

XTI®-5, 30m, 0.53mm ID, 1.0µm (cat.# 12255)

Inj.:	1.0µL direct injection of glycols and alcohols, 100ppm each
Oven temp.:	40°C to 185°C @ 15°C/min. (hold 5 min.)
Inj. / det. temp.:	150°C / 200°C
Carrier gas:	helium
Linear velocity:	40cm/sec. (flow rate: 5cc/min.)
FID sensitivity:	8 x 10^{-11} AFS

Figure 9

Glycols on a Stabilwax® column.



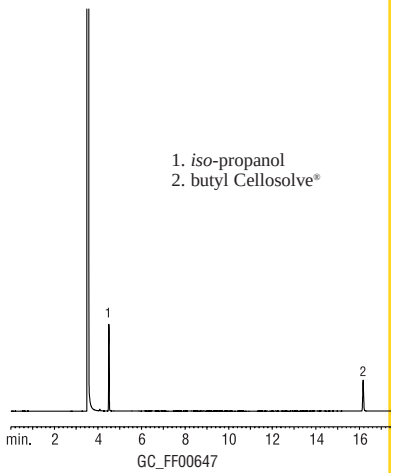
1. propylene glycol
2. ethylene glycol

Stabilwax®, 30m, 0.53mm ID, 1.0µm (cat.# 10655)

Inj.:	1.0µL direct injectionof 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 7

Glass cleaner on an Rtx®-VMS column.



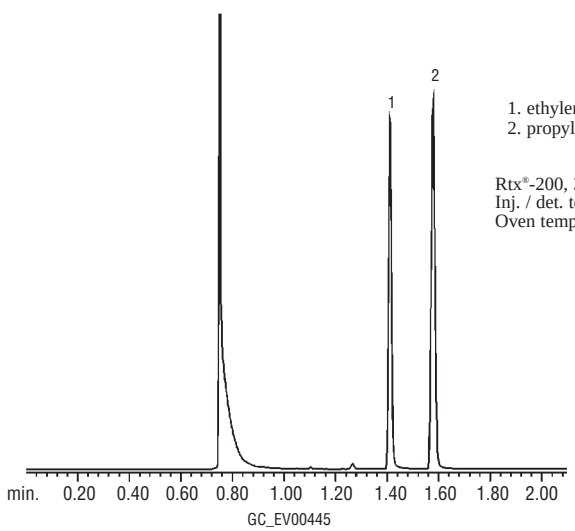
1. *iso*-propanol
2. butyl Cellosolve®

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 10

Glycols on a uniquely selective trifluoropropyl phase column.



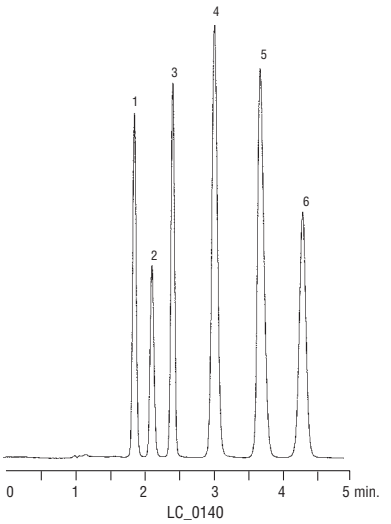
1. ethylene glycol
2. propylene glycol

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

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

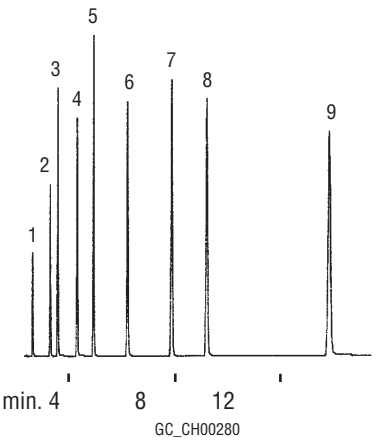
Sample:
Solvent: HPLC-grade water
Inj.: 10µL

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

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/µLeach 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

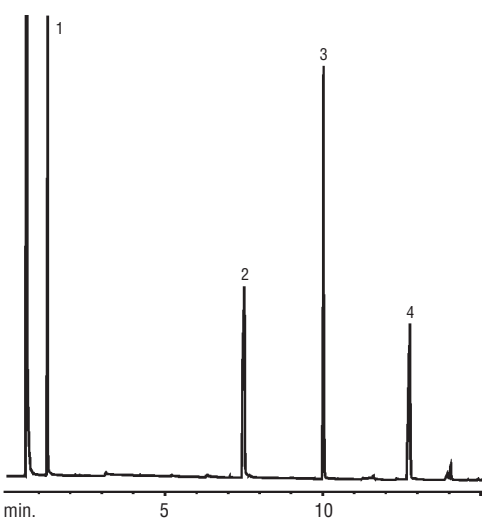
Products with higher pH are useful for dissolving fatty or oily soils. Alkalis, or bases, are used to neutralize acidic ingredients, or to raise the pH. Suitable alkalis include ethanolamines, ammonium hydroxide, and sodium silicate. The more basic compounds, such as ethanolamines, can be analyzed by 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

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.

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*-xylanol/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.

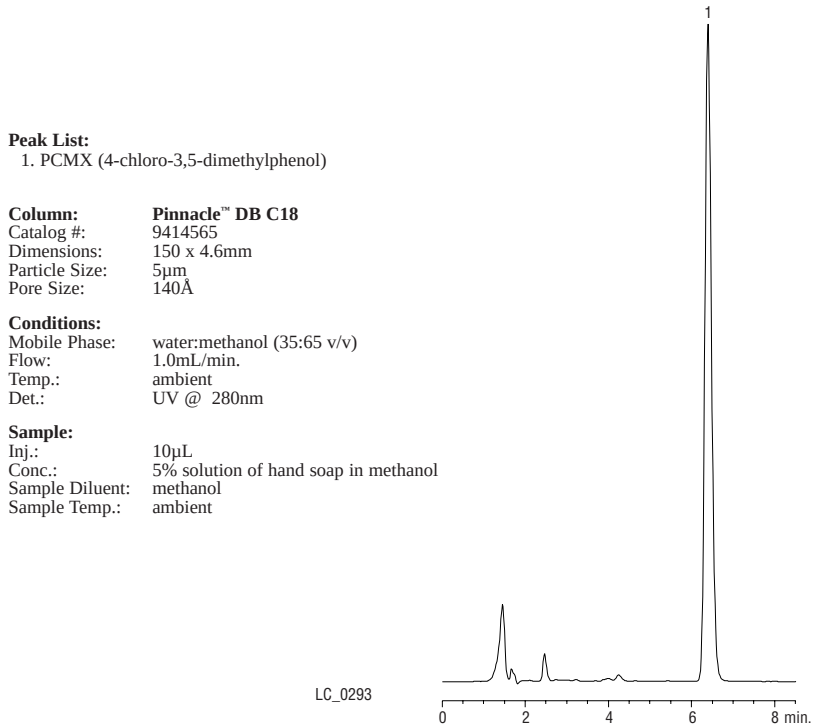


Figure 15

Resolution and symmetric peaks for sorbic and benzoic acids on an Ultra Phenyl HPLC column.

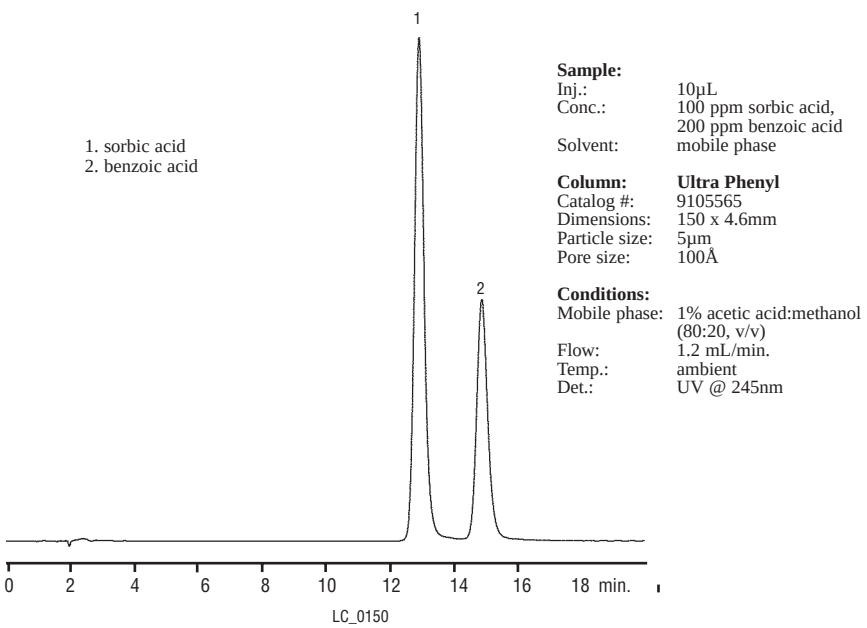
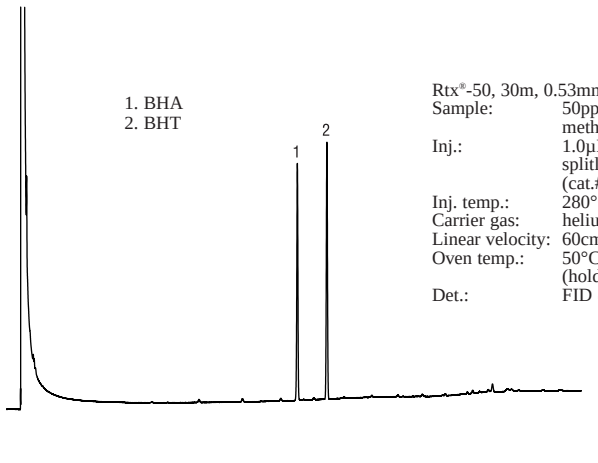


Figure 16

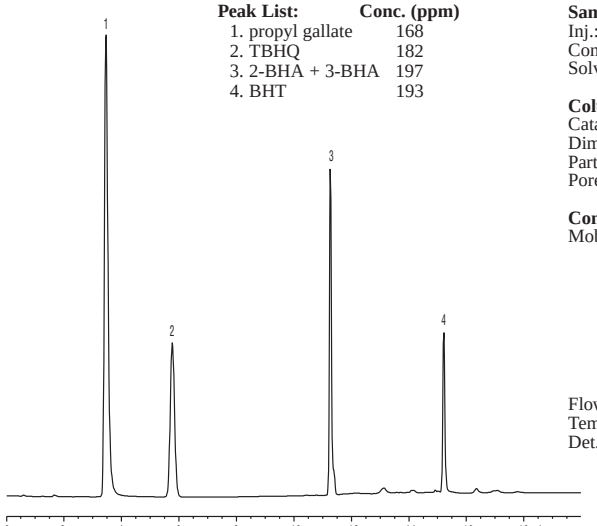
Preservatives BHA and BHT on an Rtx®-50 intermediate polarity column.



Rtx®-50, 30m, 0.53mm ID, 0.50µm (cat.# 10540)	
Sample:	50ppm each analyte in methanol
Inj.:	1.0µL direct, 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

Figure 17

Phenolic preservatives, including BHA and BHT, on a Pinnacle II™ C18 HPLC column.



Peak List:	Conc. (ppm)
1. propyl gallate	168
2. TBHQ	182
3. 2-BHA + 3-BHA	197
4. BHT	193

Sample:	
Inj.:	10µL
Conc.:	see peak list
Solvent:	methanol
Column:	**Pinnacle II™ C18**
Catalog #:	9214565
Dimensions:	150 x 4.6mm
Particle size:	5µm
Pore size:	110Å
Conditions:	
Mobile phase:	A = 1% acetic acid B = methanol

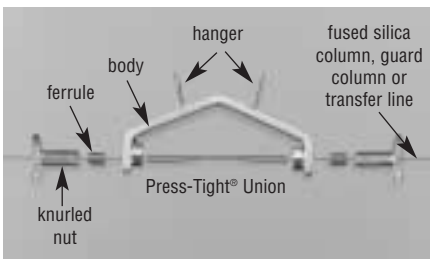
Time (min.)	B (%)
0	50
4	50
10	90
25	90
26	50

Flow:	1.0mL/min
Temp.:	30°C
Det.:	UV @ 280nm

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?

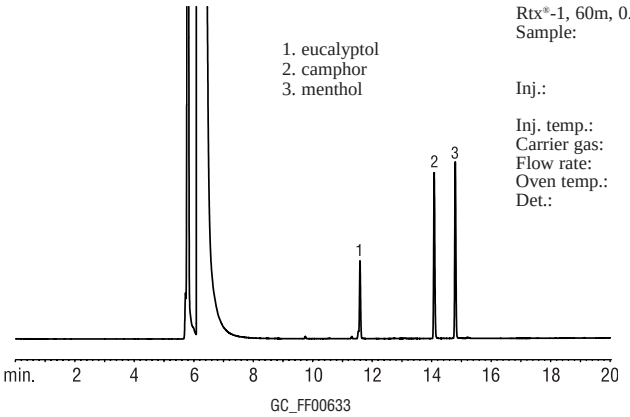
Contact Restek's Technical Service Team! We have answers to your toughest analytical questions. Call 800-356-1688 or 814-353-1300, ext. 4, email us at support@restekcorp.com or contact your local Restek representative.

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

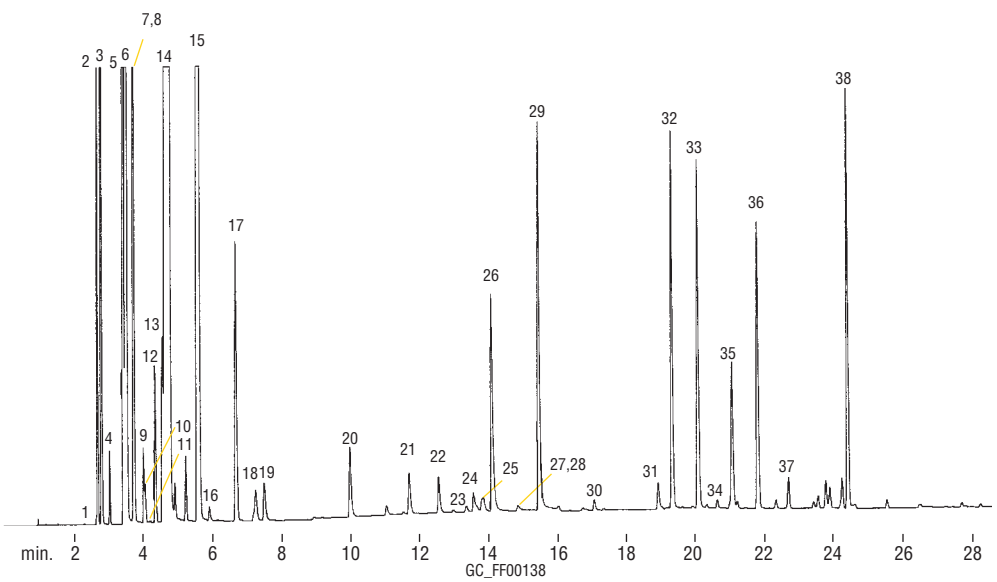
Personal care product fragrance compounds on an Rtx®-1 column.



Rtx®-1, 60m, 0.25mm ID, 0.25µm (cat.# 10126)
Sample: 130ppm eucalyptus oil
250ppm camphor
260ppm menthol in methanol
Inj.: 1.0µL split injection (20:1), 4mm inlet liner (cat.# 20814)
Inj. temp.: 275°C
Carrier gas: helium, constant flow
Flow rate: 0.6 mL/min.
Oven temp.: 80°C to 180°C @5°C/min.
Det.: FID @300°C

Figure 19

A complex fragrance, lemon oil, resolved on an Rtx®-5 column.

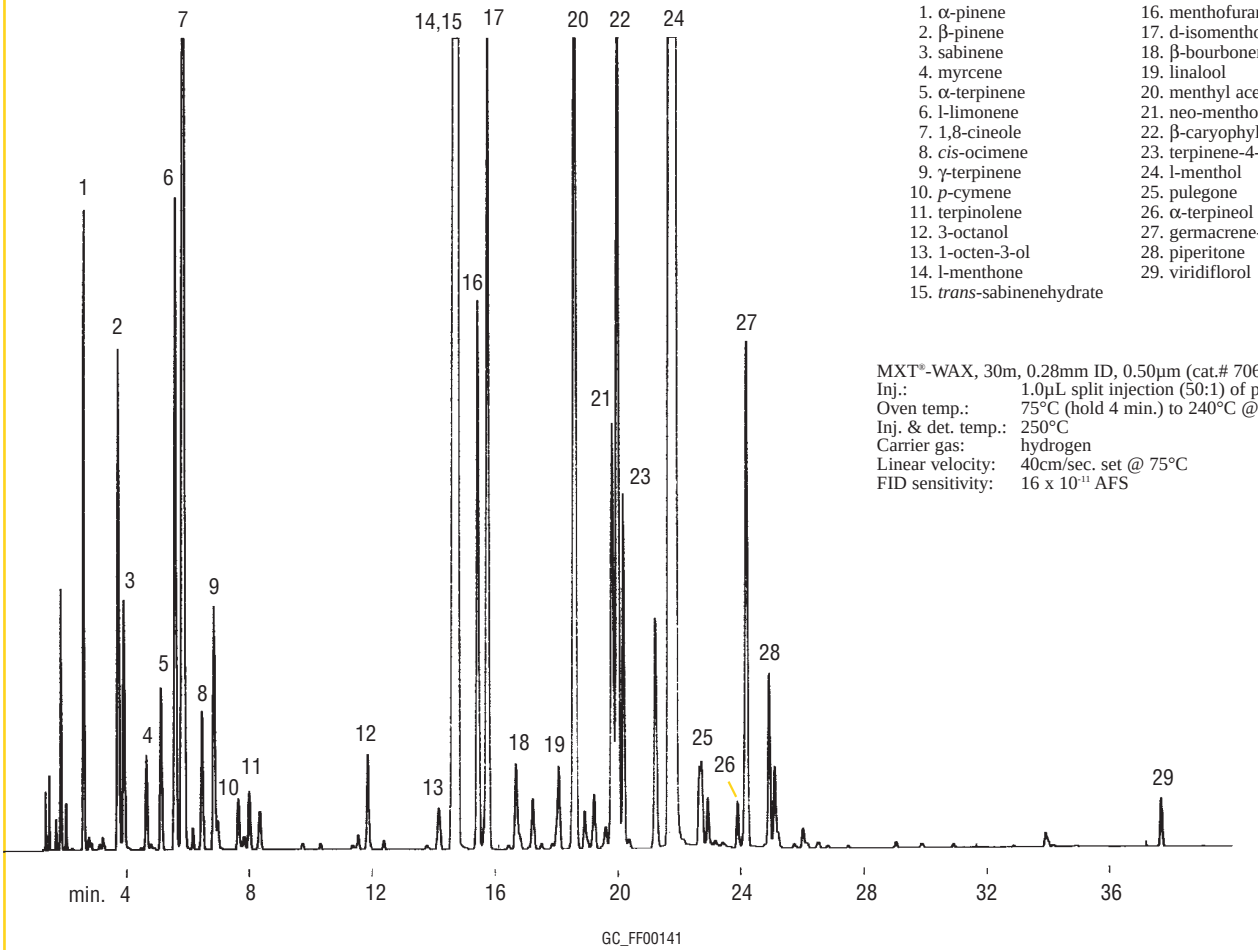


Rtx®-5, 30m, 0.32mm ID, 0.25µm (cat.# 10224)
Inj.: wet needle split injection (100:1) of neat lemon oil
Oven temp.: 75°C (hold 8 min.) to 250°C @ 4°C/min.
Inj. & det. temp.: 250°C
Carrier gas: hydrogen
Linear velocity: 40cm/sec. (flow rate: 3.2cc/min.)
FID sensitivity: 2 x 10^{-11} AFS

1. heptanol
2. α-thujene
3. α-pinene
4. camphene
5. sabinene
6. β-pinene
7. 6-methyl-5-hepten-2-one
8. myrcene
9. octanal
10. α-phellandrene
11. 3-carene
12. α-terpinene
13. *p*-cymene
14. limonene
15. γ-terpinene
16. octanol
17. terpinolene
18. linalool
19. nonanal
20. citronellal
21. terpinene-4-ol
22. α-terpineol
23. decanol
24. octyl acetate
25. nerol
26. neral
27. carvone
28. geraniol
29. geranial
30. nonyl acetate
31. citronellyl acetate
32. neryl acetate
33. geranyl acetate
34. dodecanal
35. β-caryophyllene
36. *trans*-α-bergamotene
37. α-humulene
38. β-bisabolene

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

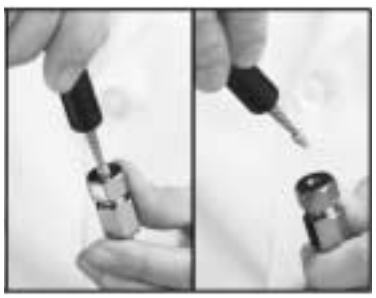
1. Branna, Tom *The I&I Market* in *Happi*, Nov. 2000.
2. The Soap and Detergent Association. www.sdahq.org
3. US Environmental Protection Agency. www.epa.gov/pesticides/citizens/antimic.htm

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

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

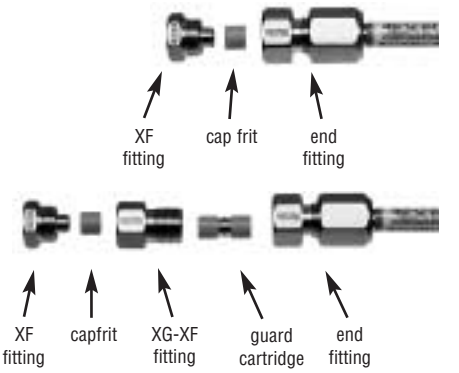
Trident™ HPLC In-Line Guard Cartridge Holders

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-styl*

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

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% dimethylpolysiloxane)
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

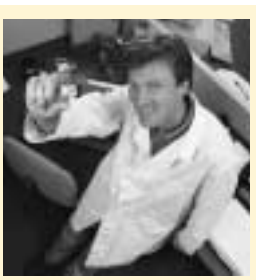
citric acid	2000µg/ml
fumaric acid	10
malic acid	2000
quinic acid	2000
tartaric acid	2000

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



Restek will create the right solution for you!

"The Company Chromatographers Trust"

- ✔ Quotations supplied quickly.
- ✔ 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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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-in-the-day order. Keep reaching for Restek products and service, and we will provide you with Plus 1™ quality and attention.

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Technical Guide

Optimizing the Analysis of Volatile Organic Compounds

Inside:

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Table of Contents

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.

We would like to thank the following people for their technical contributions to this guide:

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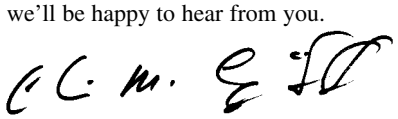
We hope you enjoy reading this guide and find it useful in your work. If you have any questions, or have input for future editions, please don't hesitate to contact Restek Corporation - we'll be happy to hear from you.



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.

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 refered 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.

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.*

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-C32
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, *m*-naphthalene, MTBE, etc.
Michigan (GRO)	BTEX, *m*-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

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.

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

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.

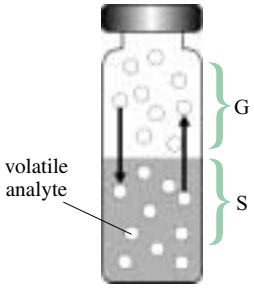
Purge and Trap Theory

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.

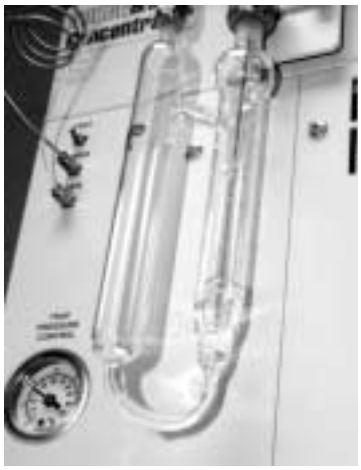
Volatile analyte in equilibrium between the gas and sample phases.



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.

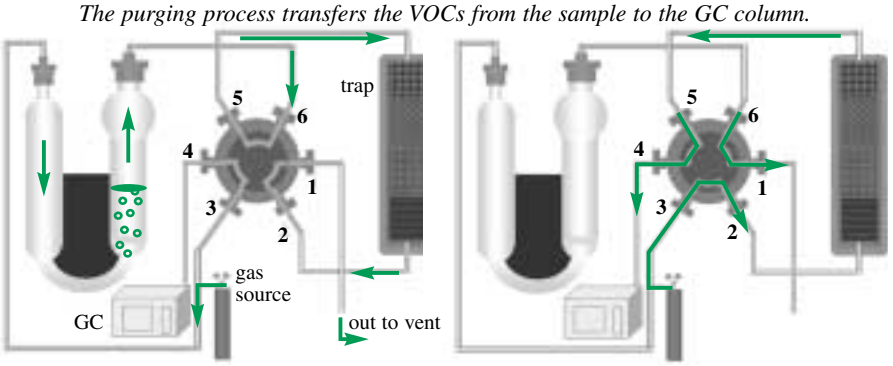


Sample purging in progress in a Tekmar 3100 concentrator.

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.

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)

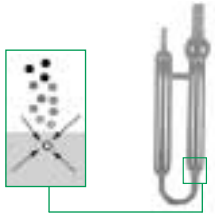
During the wet purge, a large amount of water is removed from the sample and collects on the trap. The dry purge removes the excess water that accumulated. During the dry purge, the purge gas bypasses the purge vessel and is directed to the trap. The dry purge gas removes water and carries it out the 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 5.
Desorb

- Analytes are backflushed into column.
- Typical time: 2-4 min.
- Typical temp.: 180°-250°C.
- Typical flow: 10-80mL/min.

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.

Step 6.
Trap Bake

- Trap is baked clean with flow.
- Typical time: 6-10 min.
- Typical temp.: 10-20°C higher than desorb temp.
- Avoid overheating adsorbents.

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, ½-inch mount	ea.	21150
25mL Fritted Sparger, ½-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).

Figure 6.

The 6-port automated switching valve (V) controls purge and desorb flow rates.

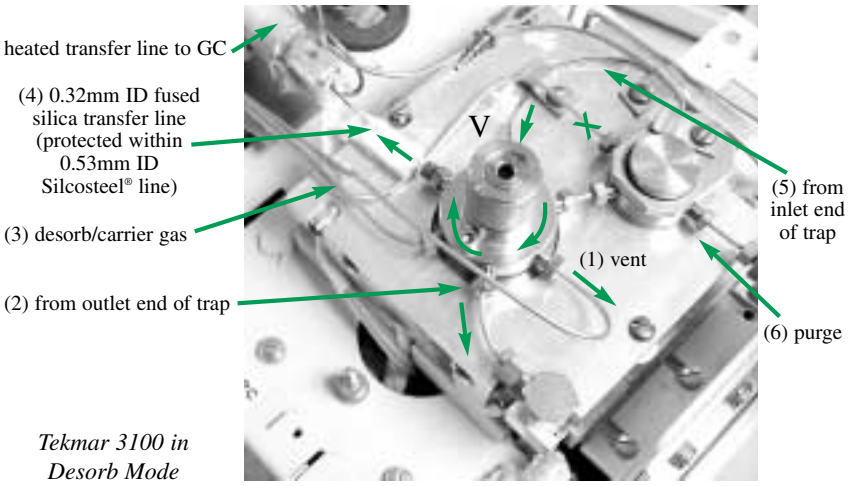
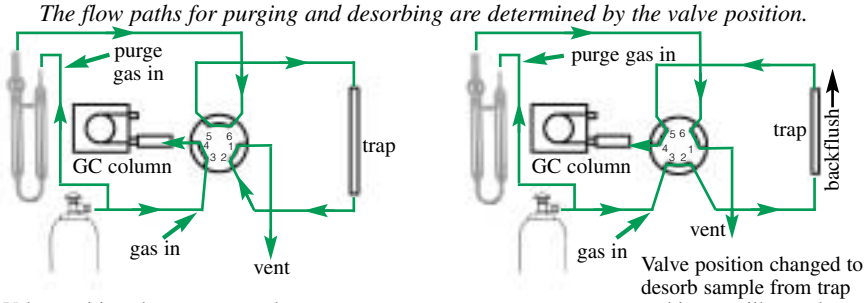


Figure 7.

The flow paths for purging and desorbing are determined by the valve position.



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-diphenylene-neoxide 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 (CO_2) purged from the sample, and it has been reported that charcoal is a source of CO_2, which can interfere with the quantitation of early-eluting compounds when using GC/MS systems.

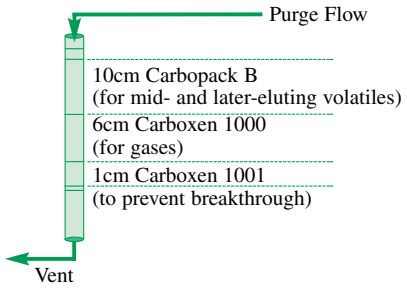
Graphitized Carbon Black or Carbopack® Adsorbent (surface area: 10-100m²/g): Graphitized carbon black (GCB) is an alternative to Tenax® adsorbent. GCB is available in many pore sizes and is effective in trapping volatile organics in the same range as Tenax® adsorbent. GCB is hydrophobic and has excellent 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 CO_2, 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.*

Figure 8.
Type "K" trap (Vocarb™ 3000 trap) showing the various layers.



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 breakdown, 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 Tenex®/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)
24cmTenax®	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 Carbopack® B/1.3cm Carbosieve® S-III	H	yes	245	250	260
8.5cm Carbopack® C/10cm Carbopack® B/ 6cm Carboxen® 1000/1cm Carboxen® 1001	I (Vocarb™ 4000)	yes	245	250	260
7.7cm Carbopack® C/1.2cm Carbopack® B	J (BTEXTRAP™)	yes	245	250	260
10cm Carbopack® 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.

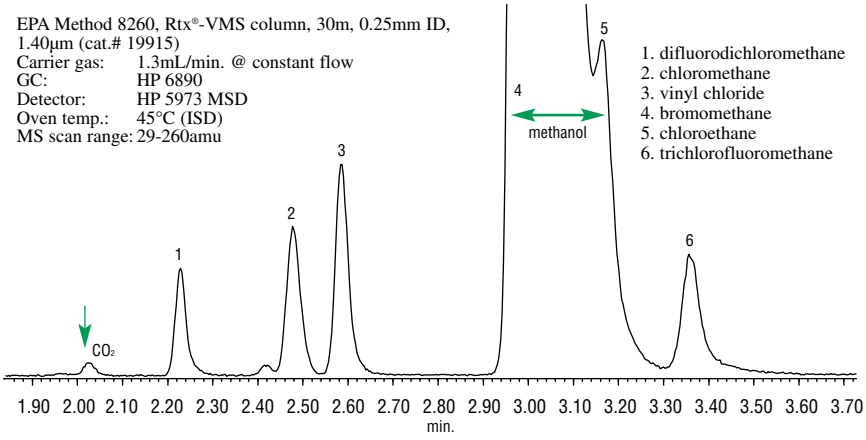
In similar fashion, methanol also causes interferences with target analytes. The PID gives a positive signal for methanol as a broad, flat-topped peak that usually interferes with 2-methylpentane, 3-methylpentane, and vinyl chloride. Adjusting the MS scan range to start above 35amu can minimize the effects 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.*

Figure 9.

MS scan rate adjusted to detect methanol and show the region where methanol elutes from an Rtx®-VMS column.

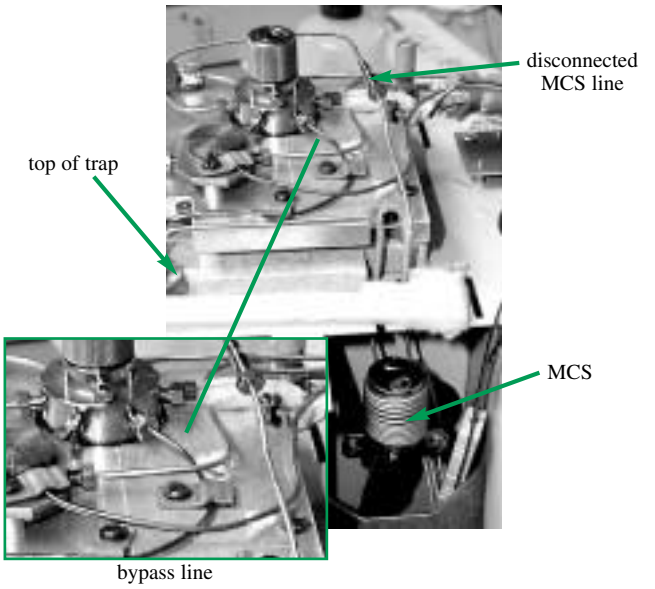


Transfer Line

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	qty.	cat.#
Moisture Control Bypass Line	ea.	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°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 replumbing 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.

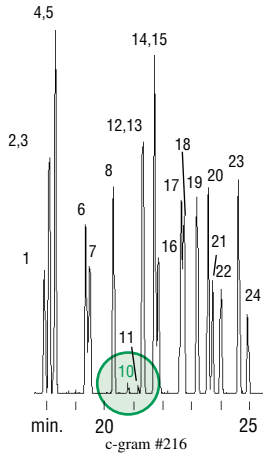
Figure 11.

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:	Vocarb™ 3000
Desorb temp.:	260°C
Desorb flow rate:	20mL/min.

1. chlorobenzene
2. ethylbenzene
3. 1,1,1,2-tetrachloroethane
4. *m*-xylene
5. *p*-xylene
6. *o*-xylene
7. styrene
8. isopropylbenzene
9. **bromoform (not detected)**
10. **1,1,2,2-tetrachloroethane**
11. 1,2,3-trichloropropane
12. propylbenzene
13. bromobenzene
14. 1,3,5-trimethylbenzene
15. 2-chlorotoluene
16. 4-chlorotoluene
17. *tert*-butylbenzene
18. 1,2,4-trimethylbenzene
19. *sec*-butylbenzene
20. *p*-isopropyltoluene
21. 1,3-dichlorobenzene
22. 1,4-dichlorobenzene
23. *n*-butylbenzene
24. 1,2-dichlorobenzene



c-gram #216

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.

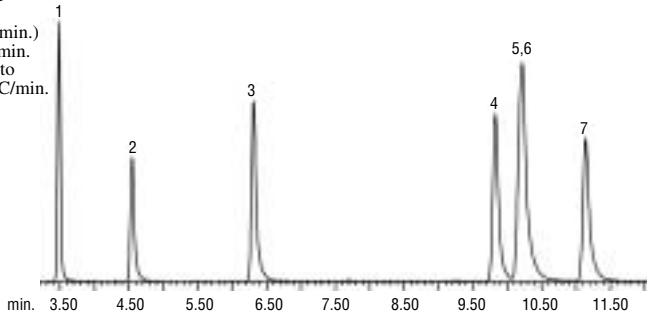
Wastewater samples commonly contain surfactants and other material that can contaminate the concentrator.

Figure 12.

A low desorb flow can produce tailing peaks, as in this example, desorbed at 9mL/min.

EPA Method 8020, Rtx®-5Sil 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.

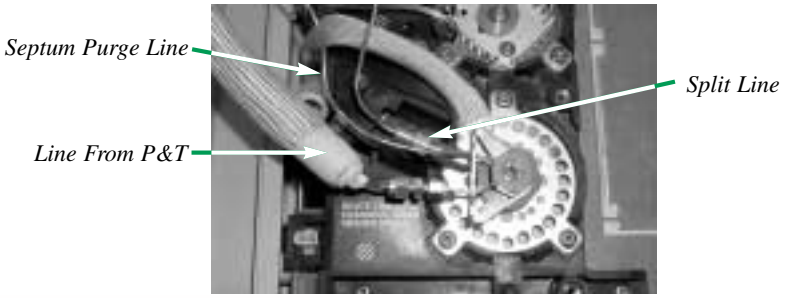


Figure 14.
Configuring your GC is simple! Prepare the lines by cutting them as shown. (Agilent 6880)

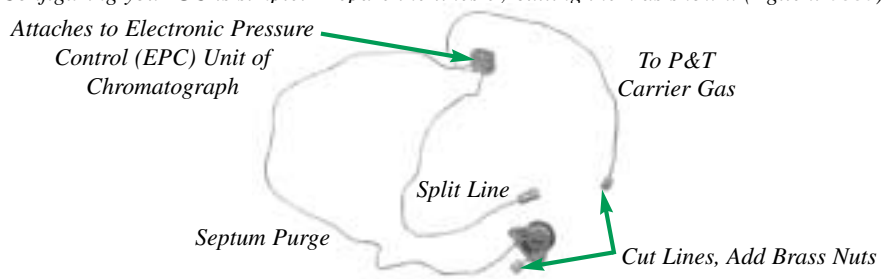


Figure 15.
Carrier gas flow is adjusted through the injection port regulator. Carrier gas sweeps analytes from the trap, through the transfer line, onto the column. (Tekmar 3100)

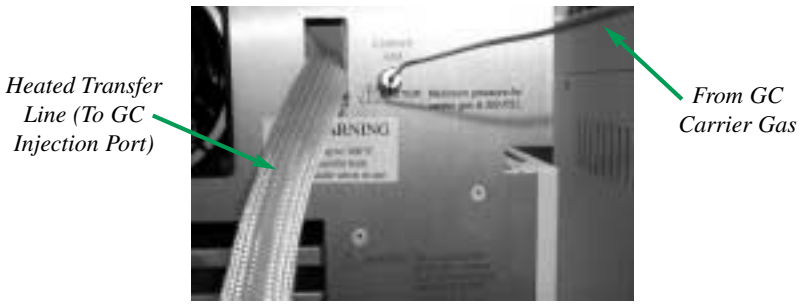
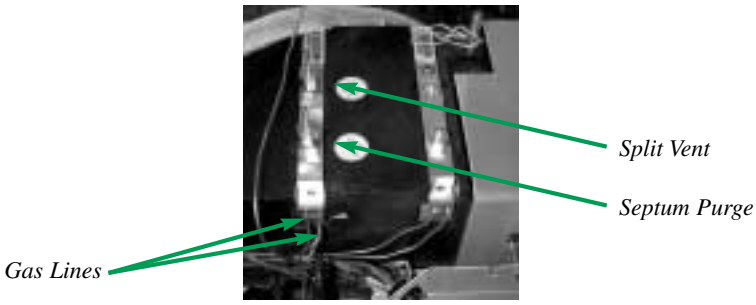


Figure 16.
Reconnect the weldment lines to the GC and check for leaks. Be sure to set up the lines correctly. (Agilent 6890)



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.

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.

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.

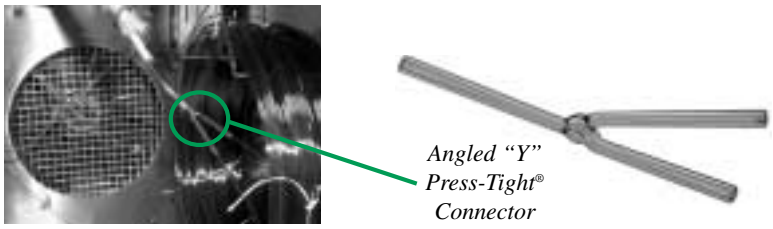
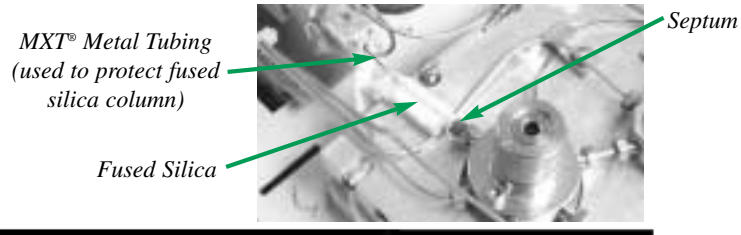


Figure 18.
Connect the fused silica line directly to the 6-port valve. Notice a small (5mm) septum helps determine how far the column is inserted into the valve, preventing breakage at the column end that could allow shards of fused silica to enter the valve.



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 quadrapole 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 quadrapole 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 coelutions 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.

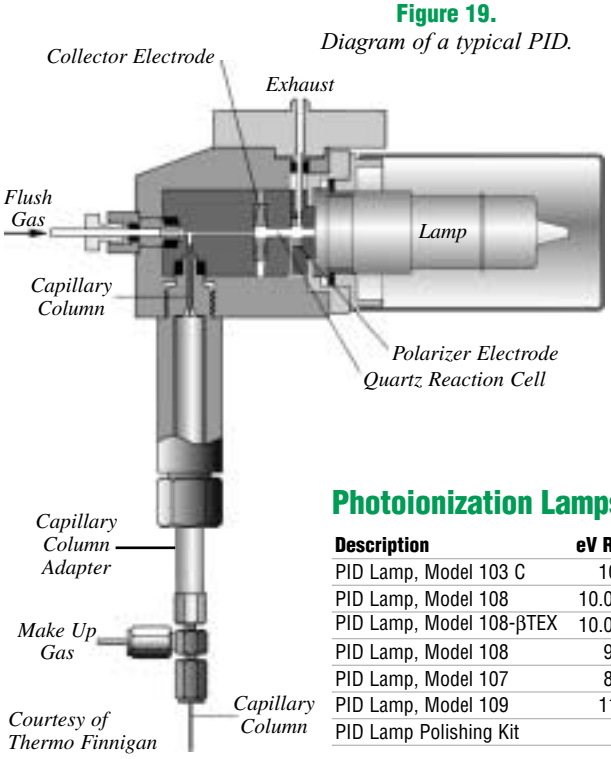
PID Operation

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.

Figure 19.
Diagram of a typical PID.



Photoionization Lamps

Description	eV Rating	Base	qty.	cat. #
PID Lamp, Model 103 C	10.2	1.375"	ea.	20676
PID Lamp, Model 108	10.0/10.6	0.781"	ea.	20675
PID Lamp, Model 108-βTEX	10.0/10.6	0.781"	ea.	23020
PID Lamp, Model 108	9.6	0.781"	ea.	23021
PID Lamp, Model 107	8.4	0.781"	ea.	23022
PID Lamp, Model 109	11.8	0.781"	ea.	23023
PID Lamp Polishing Kit			kit	20674

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

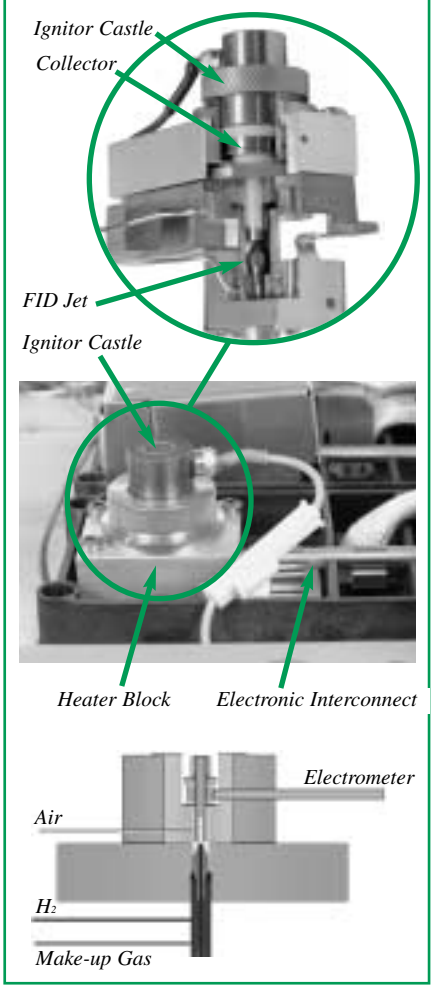
The flame ionization detector, FID (Figure 20), is a selective detector because it only responds to materials that ionize in an air/hydrogen flame. This condition covers a very broad range of compounds, however. An FID / PID combination often is used for petroleum or volatile analyte applications.

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/µL.

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.

Figure 20.
Components of a typical FID



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.)*	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.)*	qty.	cat.#	qty.	cat.#
Standard	ea.	21688	3-pk.	21689
High-Performance (Silcosteel®-Passivated)	ea.	21686	3-pk.	21687

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):

$$H_2 + CH_3(CH_2)_nCH_2R \text{ (heat and catalyst)} \rightarrow CH_3(CH_2)_nCH_3 + RH$$

where R=halogen (Br or Cl)

Example:

$$H_2 + CH_3Cl \rightarrow CH_4+HCl$$

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 21.
ELCD configured for detecting halogens.

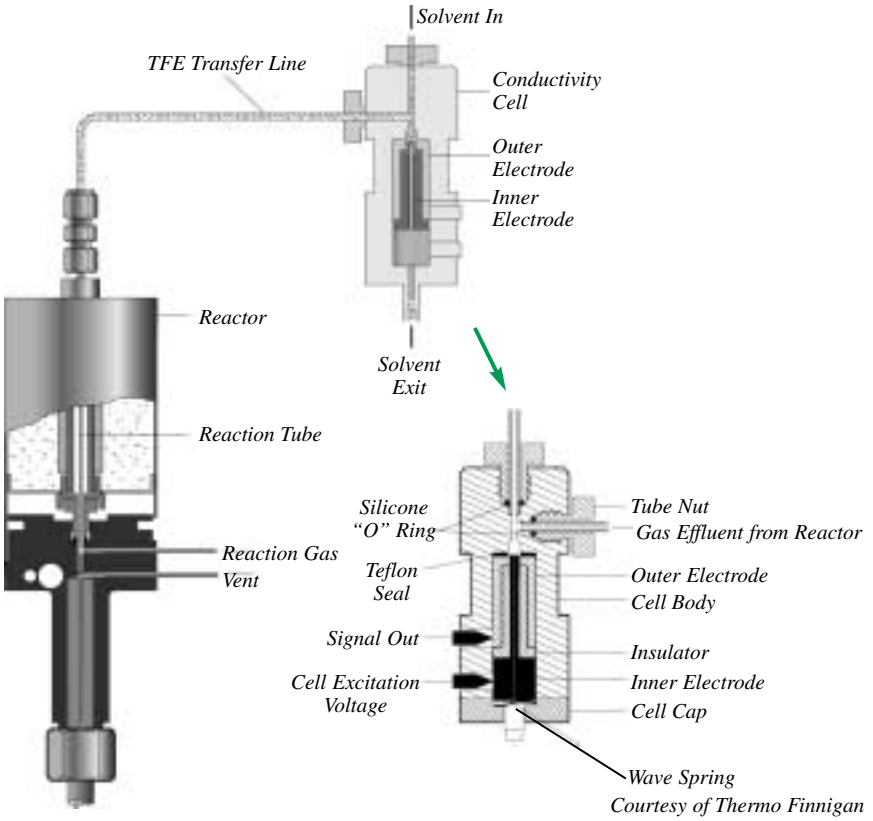
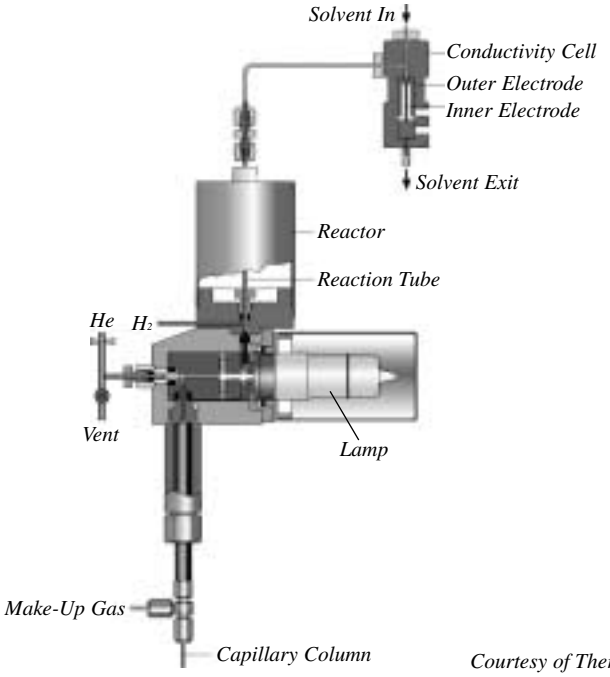


Figure 22.
Cross-section of the Thermo Finnigan PID/ELCD



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

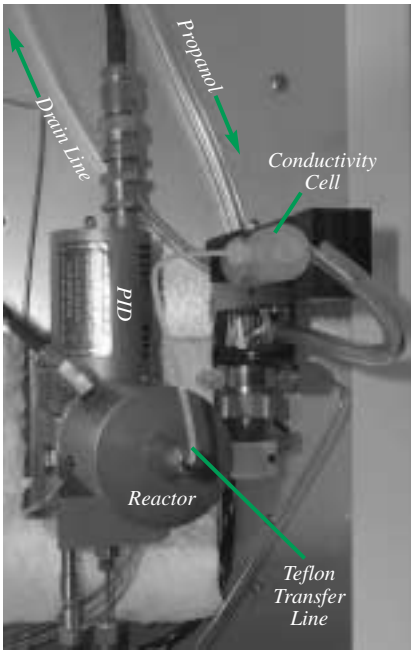
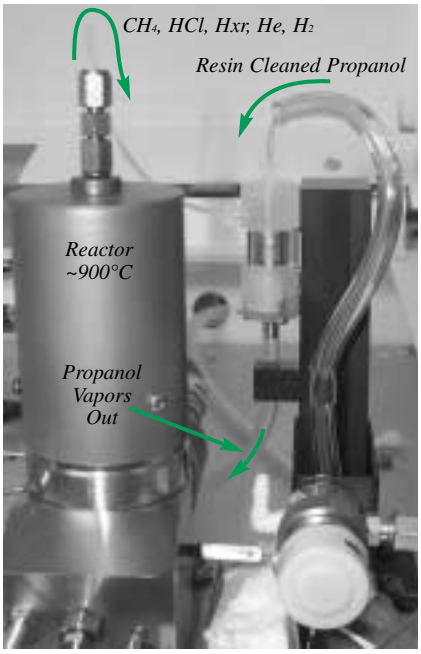


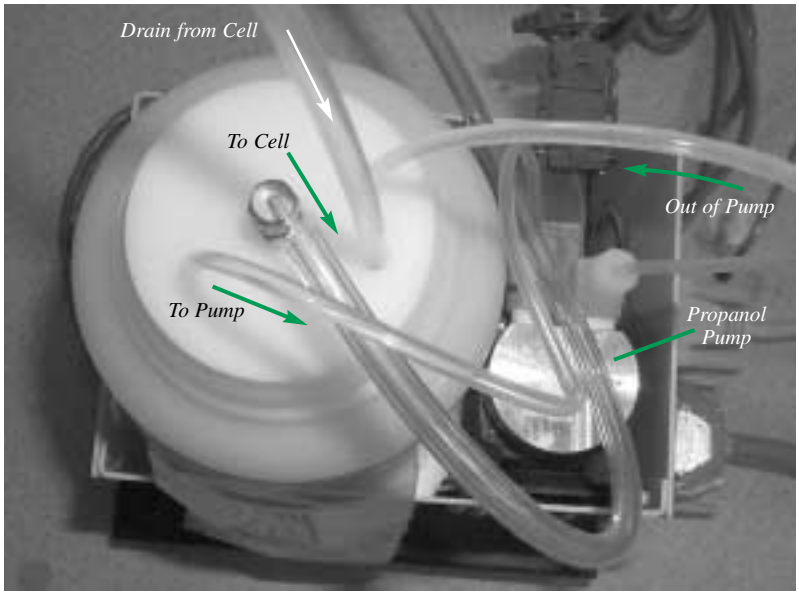
Figure 24.
Side view of the tandem detectors.



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

Figure 25.
Electrolytic conductivity detector: propanol tank with pump.



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 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.

Figure 27.
ELCD reactor removed from detector assembly, reaction tube exposed.

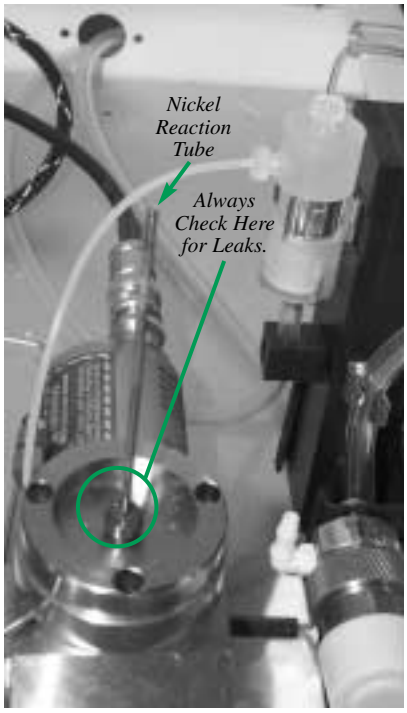


Table III.
Common causes of excessive peak tailing from an ELCD.

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	

GC/MS Operation

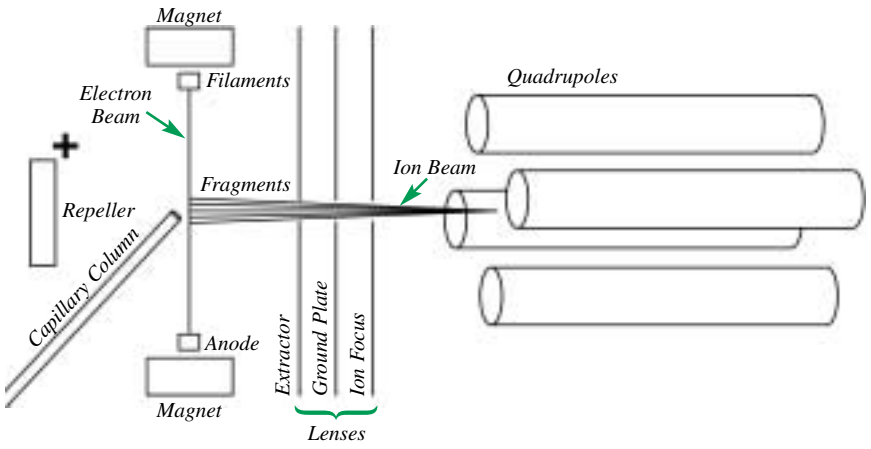
Mass spectrometry (MS) is the most common detection system used for VOC analysis. The MS provides unique spectral information for accurately identifying components eluting from the capillary column. As a compound exits the column it is bombarded with high-energy electrons and is broken into structurally 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

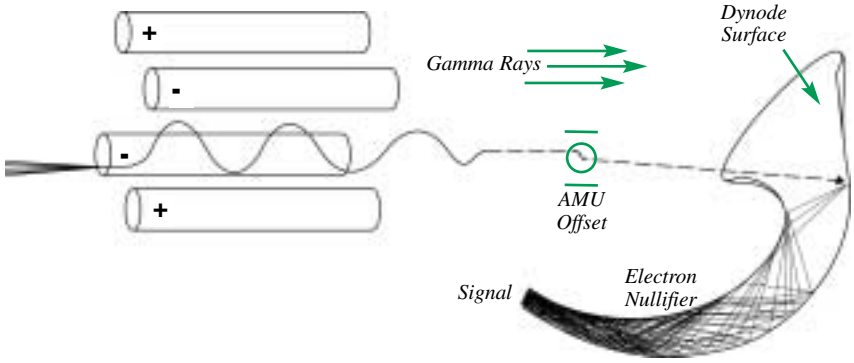
A narrow bore (≤0.25mm ID) capillary column can be inserted into the source of the quadrupole MS in electron impact mode (EI). The carrier gas flowing through the column, approximately 1mL/min., is quickly swept away under the high vacuum of the source while analytes exiting the column are bombarded 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]

Figure 28.
Electromagnets focus the ions produced from fragmented analyte molecules.



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.

Figure 30.
Ring and end caps form the core of an ion trap MS.

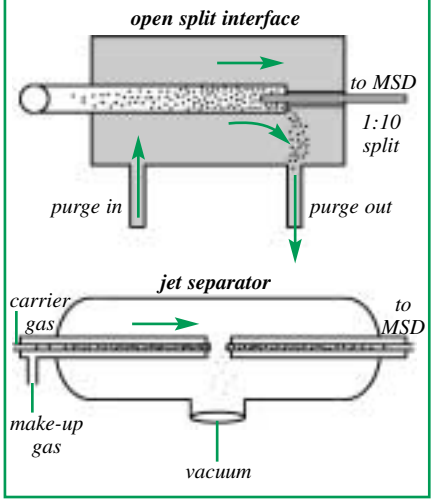
Interfacing the Capillary Column to the MS

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 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 peakwidths 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.

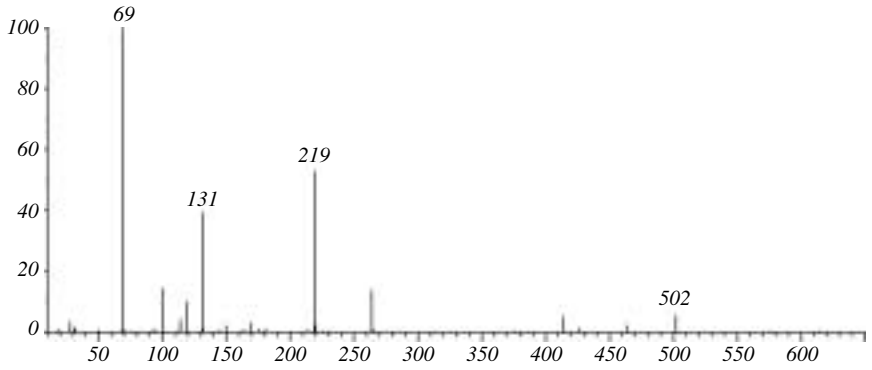
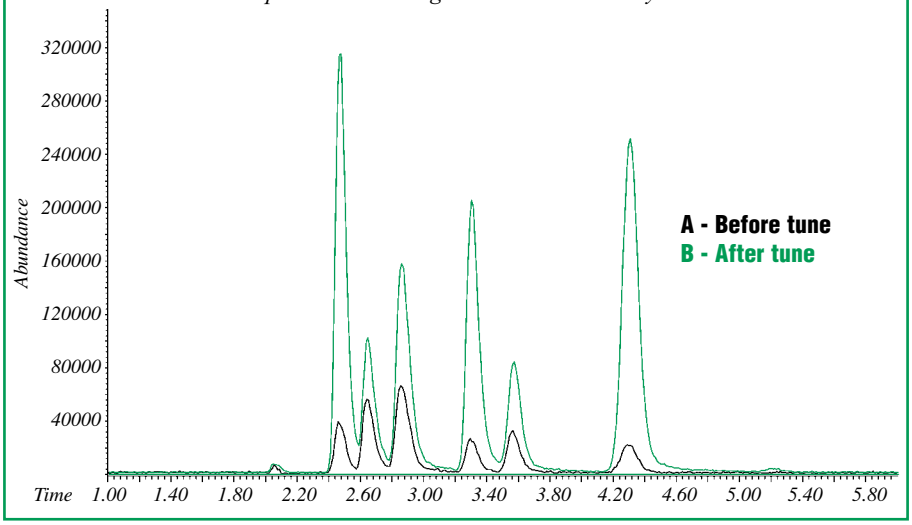


Table IV.
US EPA ion abundance criteria for 4-bromofluorobenzene (BFB).

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.

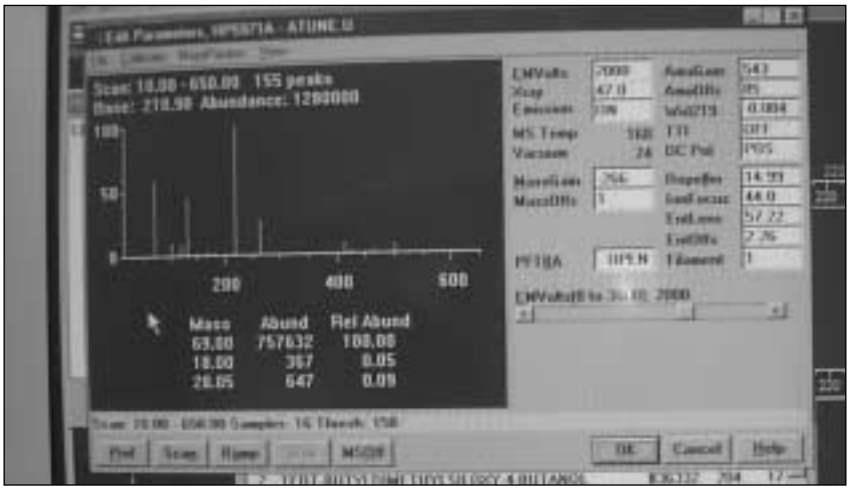


Figure 35.
The quantification ion for chlorobenzene-d5 can be changed from ion 117 to ion 82, to eliminate the need for chromatographic resolution from 1,1,1,2-tetrachloroethane.

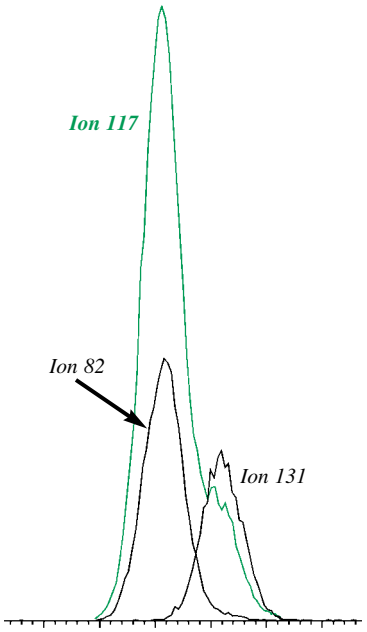
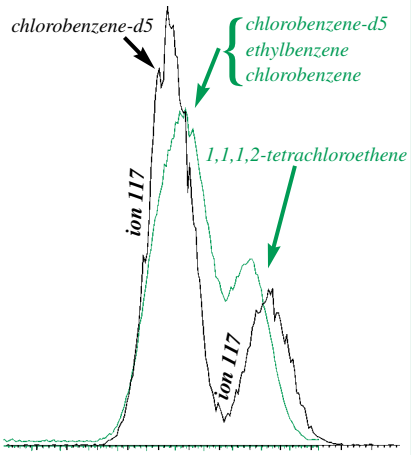


Figure 36.
A slower oven temperature program eliminates the need to change the internal standard or the quantification ion.



Extracted ion chromatogram (black) overlayed on total ion chromatogram (green)

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 1.0µm 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.

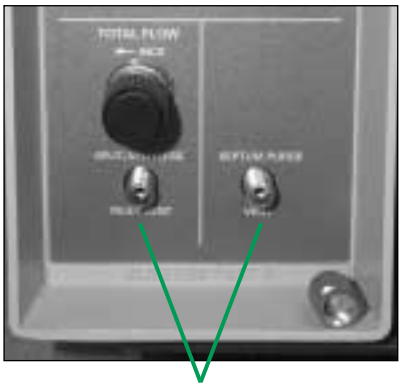
12. *F.W. McLafferty and F. Turecek. Interpretation of Mass Spectra, University Science Books, Mill Valley, 4th edition, 1993, pp. 283-291.*

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.

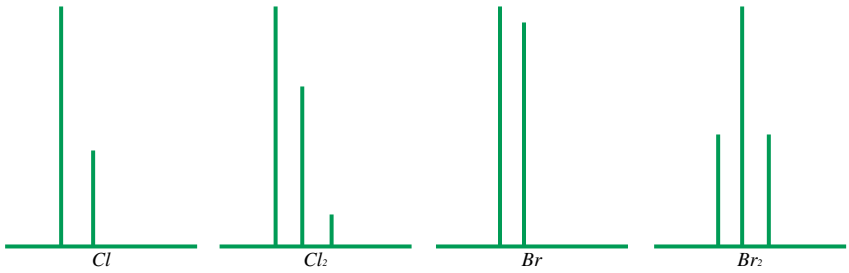
After you confirm the MS is leak-free, quickly install the inlet end of the column into the injection port. The MS will draw air into the source during this connection time; after another 2-hour equilibration the MS is ready for tuning/leak checking using PFTBA.

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).



Element	Mass	% Abundance	Mass	% Abundance
carbon	12	100	13	1.1
nitrogen	14	100	15	0.37
chlorine	35	100	37	32.5
bromine	79	100	81	98
oxygen	16	100	18	0.2

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

MSD Source Nut

(Similar to Agilent part # 05988-20066.)

Description	qty.	cat.#
(Detector) MSD Source Nut	2-pk.	20643

www.restekcorp.com

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 $1x10^{-4}$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 Vocarb® 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.

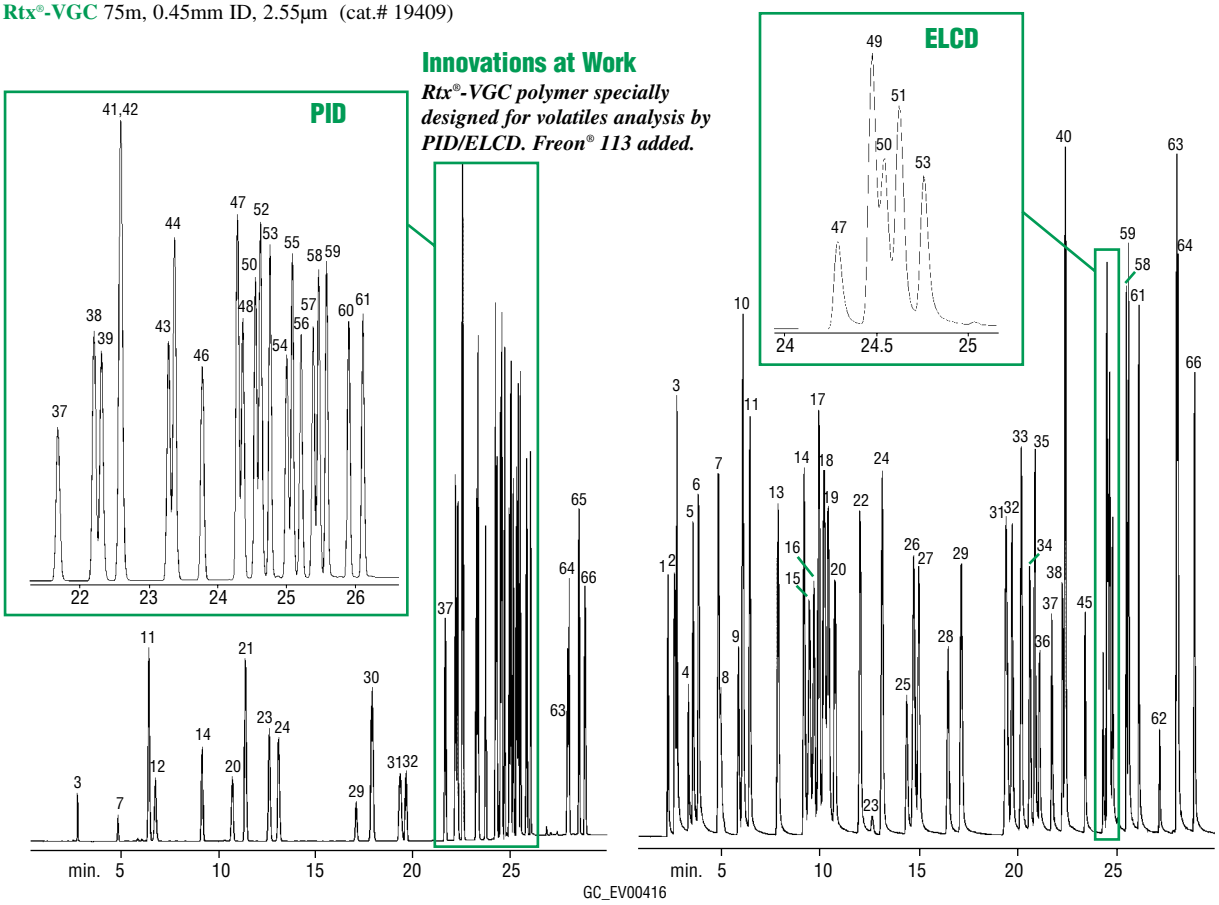
Figure 39A.

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)

Innovations at Work

Rtx®-VGC polymer specially designed for volatiles analysis by PID/ELCD. Freon® 113 added.



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.

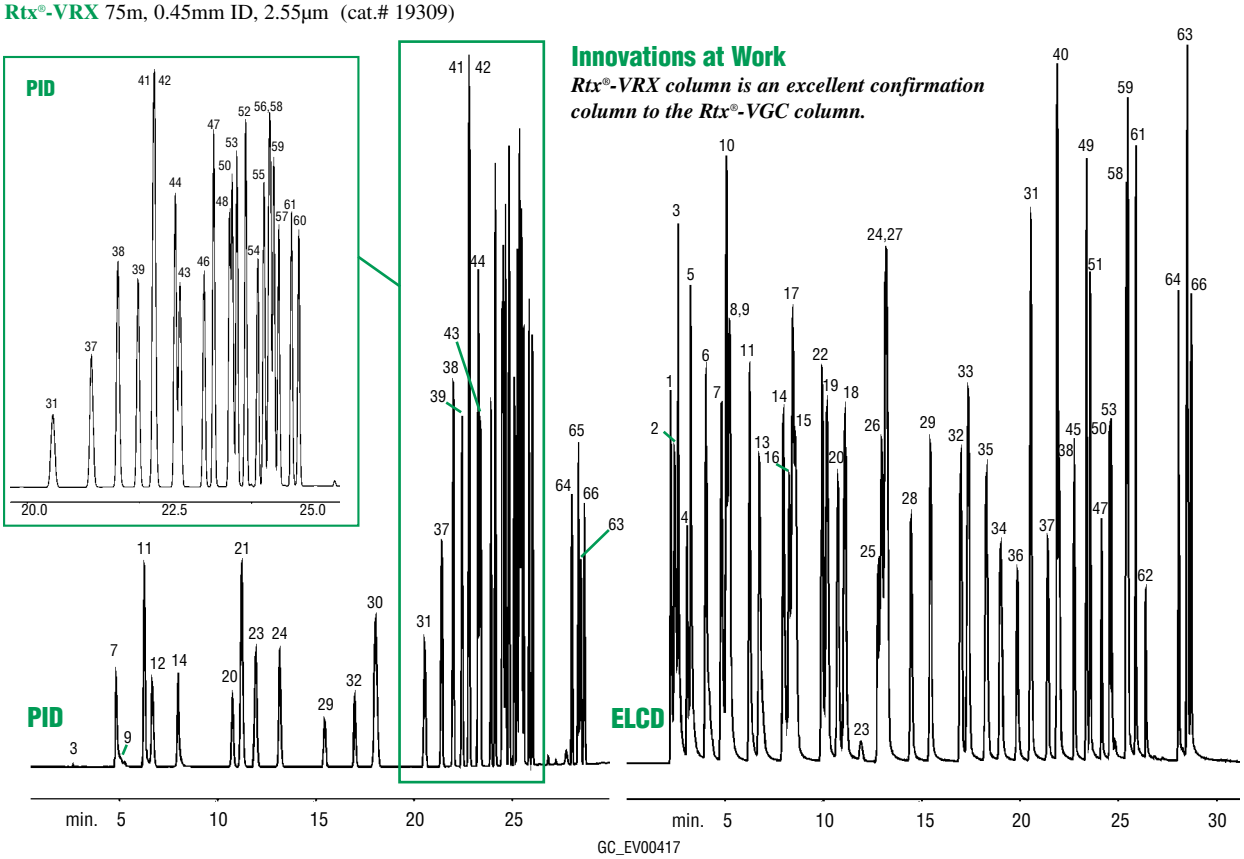
1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethene
8. Freon® 113
9. allyl chloride
10. methylene chloride
11. *trans*-1,2-dichloroethene
12. methyl *tert*-butyl ether
13. 1,1-dichloroethane
14. *cis*-1,2-dichloroethene
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

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)



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:	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:	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

Figure 40A.

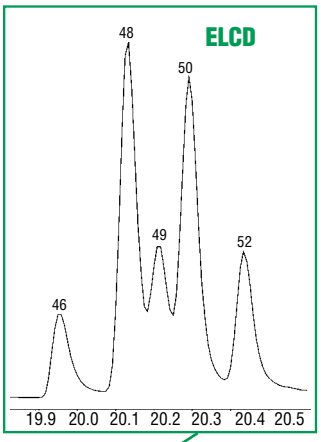
An Rtx®-VGC / Rtx®-502.2 column pair and a 50°C initial temperature reduce total cycle time to less than 30 minutes for EPA Method 8021A/502.2.

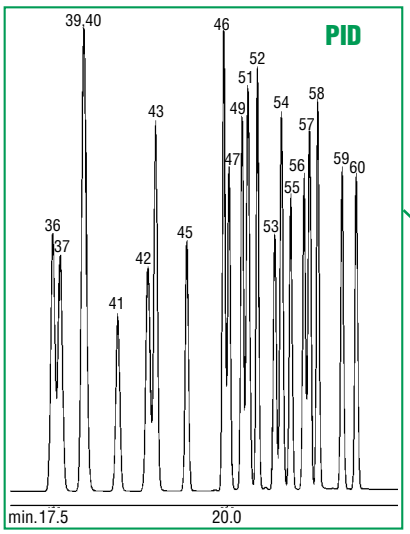
Innovations at Work

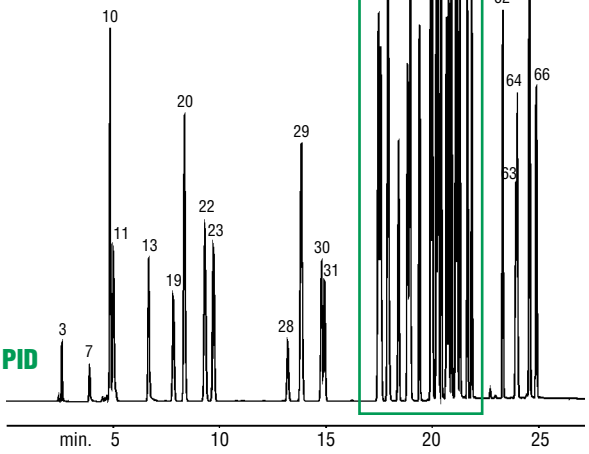
Fastest cycle time for 8021/502.2!
50°C starting temperature brings total cycle time to less than 30 min.

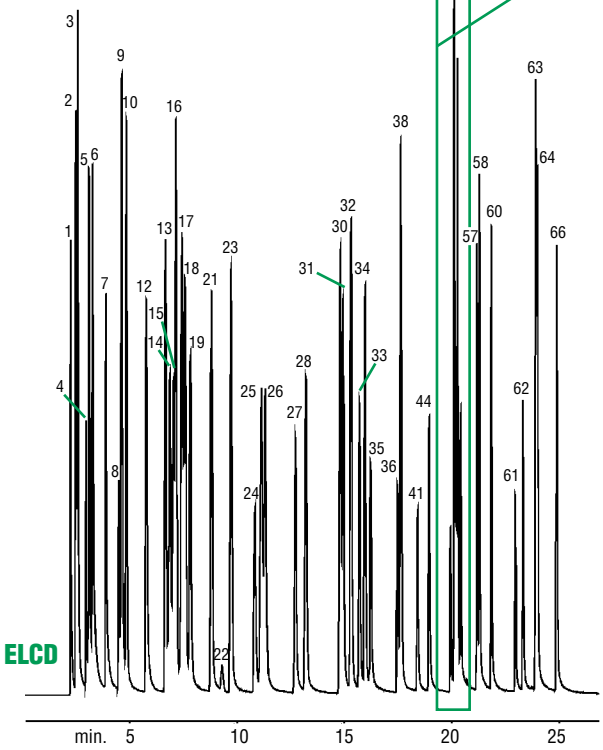
Rtx®-VGC 75m, 0.45mm ID, 2.55µm (cat.# 19409)

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:	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:	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.
Oven temp.:	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.)
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., Temp.940 C, propanol flow 470 L/min.









1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethene
8. allyl chloride
9. methylene chloride
10. *trans*-1,2-dichloroethene
11. methyl *tert*-butyl ether
12. 1,1-dichloroethane
13. *cis*-1,2-dichloroethene
14. 2,2-dichloropropane
15. bromochloromethane
16. chloroform
17. carbon tetrachloride
18. 1,1,1-trichloroethane
19. 1,1-dichloropropene
20. benzene
21. 1,2-dichloroethane
22. fluorobenzene (SS)
23. trichloroethene
24. dibromomethane
25. 1,2-dichloropropane
26. bromodichloromethane
27. 1-bromo-2-chloroethane (SS)
28. *cis*-1,3-dichloropropene
29. toluene
30. tetrachloroethene
31. *trans*-1,3-dichloropropene
32. 1,1,2-trichloroethane
33. dibromochloromethane
34. 1,3-dichloropropane
35. 1,2-dibromoethane
36. chlorobenzene
37. ethylbenzene
38. 1,1,1,2-tetrachloroethane
39. *m*-xylene
40. *p*-xylene
41. 1-chloro-2-fluorobenzene (SS)
42. *o*-xylene
43. styrene
44. bromoform
45. isopropylbenzene
46. bromobenzene
47. *n*-propylbenzene
48. 1,1,2,2-tetrachloroethane
49. 2-chlorotoluene
50. 1,2,3-trichloropropane
51. 1,3,5-trimethylbenzene
52. 4-chlorotoluene
53. *tert*-butylbenzene
54. 1,2,4-trimethylbenzene
55. *sec*-butylbenzene
56. *p*-isopropyltoluene
57. 1,3-dichlorobenzene
58. 1,4-dichlorobenzene
59. *n*-butylbenzene
60. 1,2-dichlorobenzene
61. 1,2-dibromo-3-chloropropane
62. 2-bromo-1-chlorobenzene (SS)
63. hexachlorobutadiene
64. 1,2,4-trichlorobenzene
65. naphthalene
66. 1,2,3-trichlorobenzene

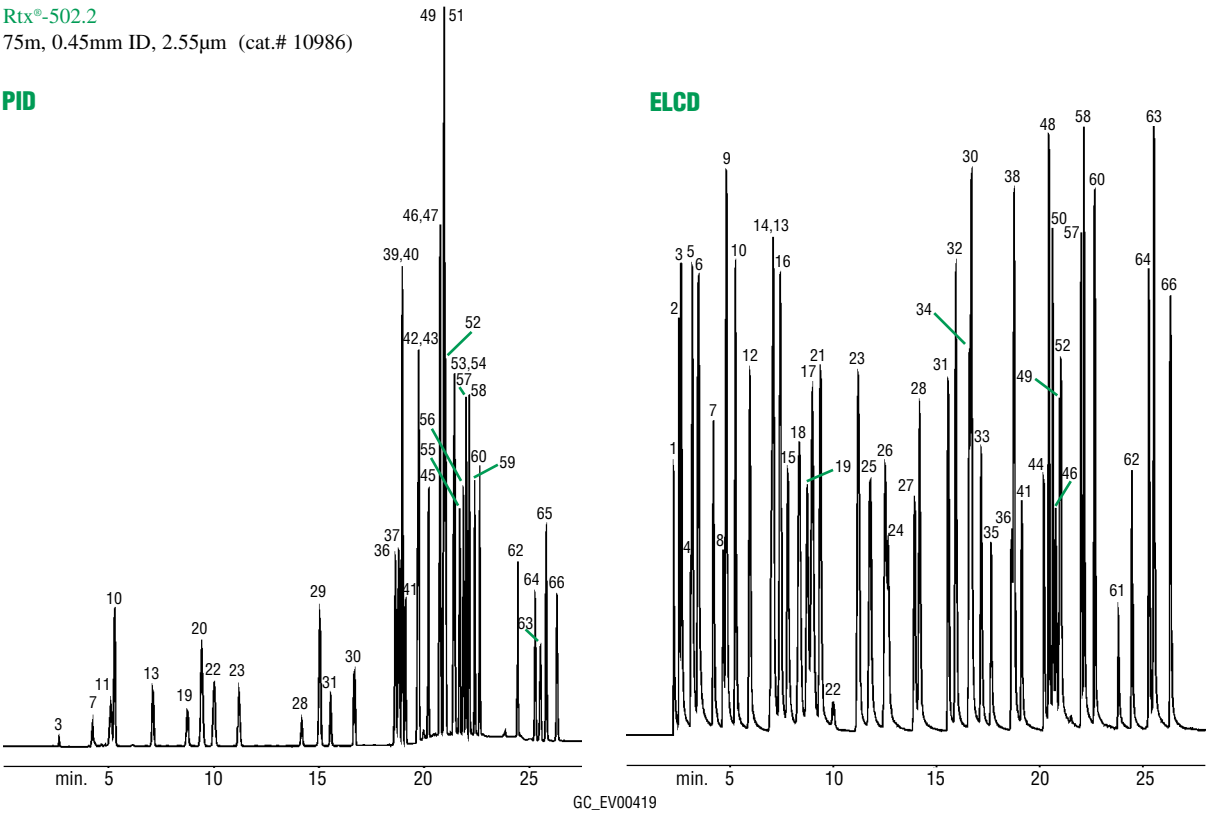
GC_EV00418

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)



1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethene
8. allyl chloride
9. methylene chloride
10. *trans*-1,2-dichloroethene
11. methyl *tert*-butyl ether
12. 1,1-dichloroethane
13. *cis*-1,2-dichloroethene
14. 2,2-dichloropropane
15. bromochloromethane
16. chloroform
17. carbon tetrachloride
18. 1,1,1-trichloroethane
19. 1,1-dichloropropene
20. benzene
21. 1,2-dichloroethane
22. fluorobenzene (SS)
23. trichloroethene
24. dibromomethane
25. 1,2-dichloropropane
26. bromodichloromethane
27. 1-bromo-2-chloroethane (SS)
28. *cis*-1,3-dichloropropene
29. toluene
30. tetrachloroethene
31. *trans*-1,3-dichloropropene
32. 1,1,2-trichloroethane
33. dibromochloromethane
34. 1,3-dichloropropane
35. 1,2-dibromoethane
36. chlorobenzene
37. ethylbenzene
38. 1,1,1,2-tetrachloroethane
39. *m*-xylene
40. *p*-xylene
41. 1-chloro-2-fluorobenzene (SS)
42. *o*-xylene
43. styrene
44. bromoform
45. isopropylbenzene
46. bromobenzene
47. *n*-propylbenzene
48. 1,1,2,2-tetrachloroethane
49. 2-chlorotoluene
50. 1,2,3-trichloropropane
51. 1,3,5-trimethylbenzene
52. 4-chlorotoluene
53. *tert*-butylbenzene
54. 1,2,4-trimethylbenzene
55. *sec*-butylbenzene
56. *p*-isopropyltoluene
57. 1,3-dichlorobenzene
58. 1,4-dichlorobenzene
59. *n*-butylbenzene
60. 1,2-dichlorobenzene
61. 1,2-dibromo-3-chloropropane
62. 2-bromo-1-chlorobenzene (SS)
63. hexachlorobutadiene
64. 1,2,4-trichlorobenzene
65. naphthalene
66. 1,2,3-trichlorobenzene

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:	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:	Siltek 0.32mm fused silica transfer line direct to columns w/ Press-Tight "Y" connector (cat. #20403)
Gas chromatograph:	Finnigan 9001
Carrier gas:	helium @ ~10 mL/min. constant pressure Adjust dichlorodifluoromethane to a retention time of 2.28 min. @ 50°C on the Rtx®-VGC column.
Oven temp.:	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.)
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.

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

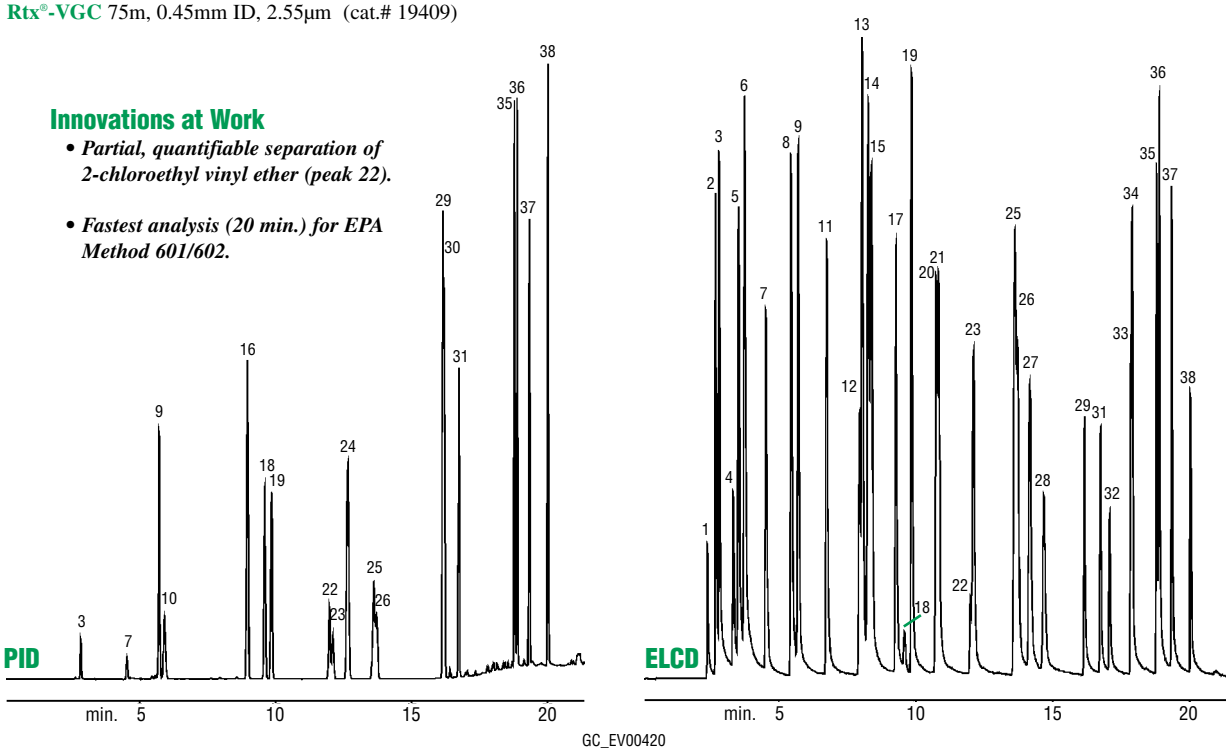
Figure 41A.

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
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethene
8. methylene chloride
9. *trans*-1,2-dichloroethene
10. methyl *tert*-butyl ether
11. 1,1-dichloroethane
12. bromochloromethane (SS)
13. chloroform
14. carbon tetrachloride
15. 1,1,1-trichloroethane
16. benzene
17. 1,2-dichloroethane
18. fluorobenzene (SS)
19. trichloroethene
20. 1,2-dichloropropane
21. bromodichloromethane
22. 2-chloroethyl vinyl ether
23. *cis*-1,3-dichloropropene
24. toluene
25. tetrachloroethene
26. *trans*-1,3-dichloropropene
27. 1,1,2-trichloroethane
28. dibromochloromethane
29. chlorobenzene
30. ethylbenzene
31. 1-chloro-2-fluorobenzene (SS)
32. bromoform
33. 1,4-dichlorobutane (SS)
34. 1,1,2,2-tetrachloroethane
35. 1,3-dichlorobenzene
36. 1,4-dichlorobenzene
37. 1,2-dichlorobenzene
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:	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.
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	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		

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.

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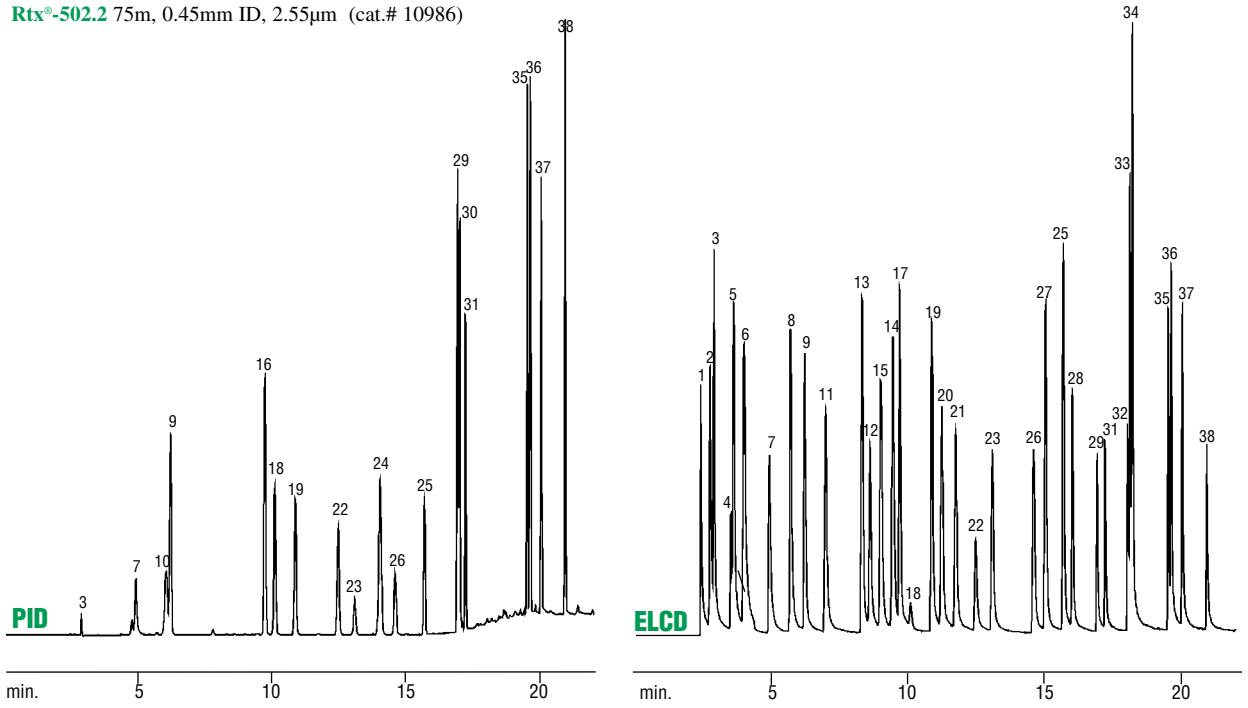
Figure 41B.

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)



1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethene
8. methylene chloride
9. *trans*-1,2-dichloroethene
10. methyl *tert*-butyl ether
11. 1,1-dichloroethane
12. bromochloromethane (SS)
13. chloroform
14. carbon tetrachloride
15. 1,1,1-trichloroethane
16. benzene
17. 1,2-dichloroethane
18. fluorobenzene (SS)
19. trichloroethene
20. 1,2-dichloropropane
21. bromodichloromethane
22. 2-chloroethyl vinyl ether
23. *cis*-1,3-dichloropropene
24. toluene
25. tetrachloroethene
26. *trans*-1,3-dichloropropene
27. 1,1,2-trichloroethane
28. dibromochloromethane
29. chlorobenzene
30. ethylbenzene
31. 1-chloro-2-fluorobenzene (SS)
32. bromoform
33. 1,4-dichlorobutane
34. 1,1,2,2-tetrachloroethane
35. 1,3-dichlorobenzene
36. 1,4-dichlorobenzene
37. 1,2-dichlorobenzene
38. 4-bromo-1-chlorobenzene (SS)

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		

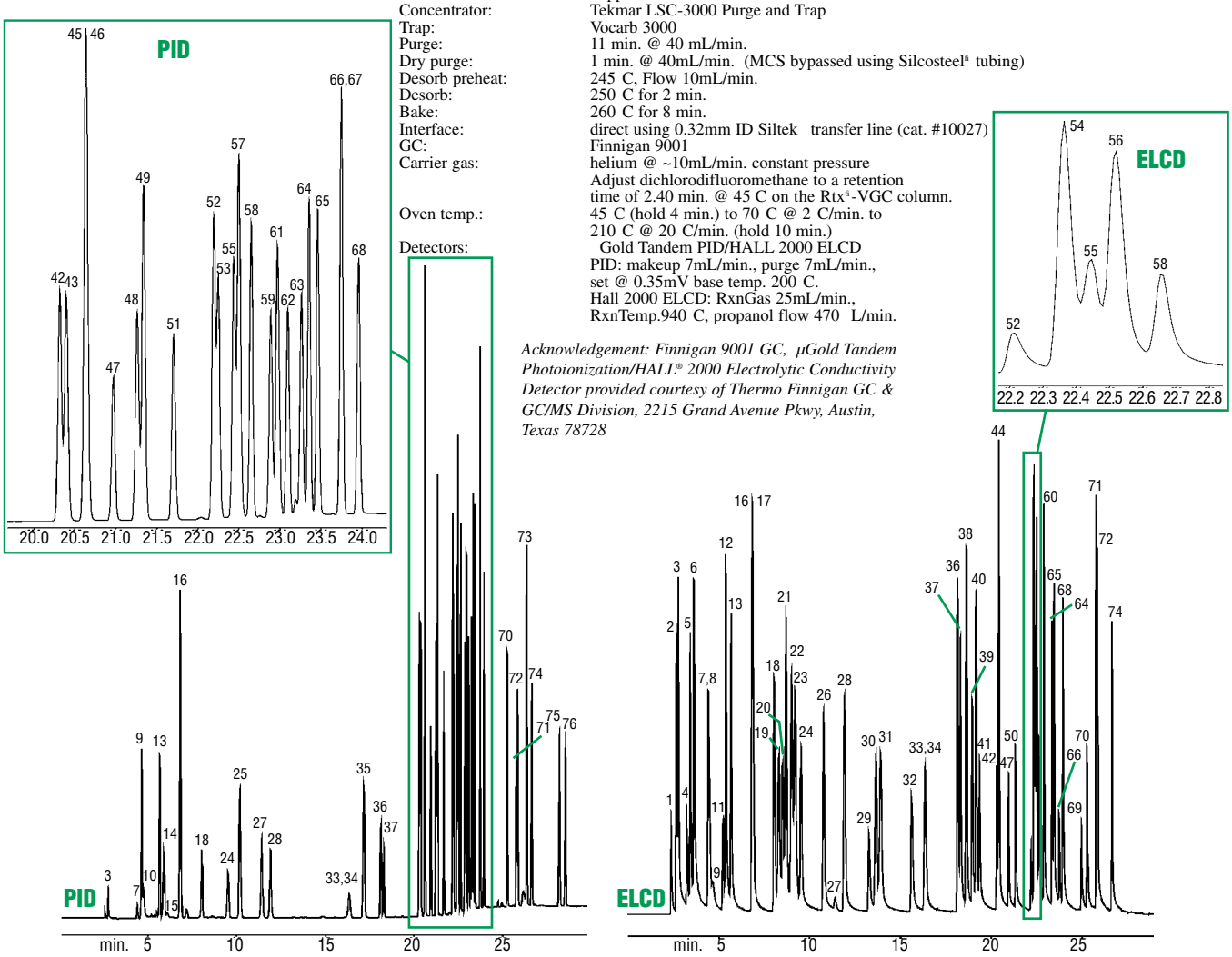
Figure 42A.

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 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 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
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethene
8. Freon® 113
9. chloromethyl methyl ether
10. iodomethane (40ppb)
11. allyl chloride
12. methylene chloride
13. *trans*-1,2-dichloroethene
14. methyl *tert*-butyl ether
15. *tert*-butyl alcohol (40ppb)
16. chloroprene
17. 1,1-dichloroethane
18. *cis*-1,2-dichloroethene
19. 2,2-dichloropropane
20. bromochloromethane
21. chloroform
22. carbon tetrachloride
23. 1,1,1-trichloroethane
24. 1,1-dichloropropene
25. benzene
26. 1,2-dichloroethane
27. fluorobenzene (SS)
28. tetrachloroethene
29. dibromomethane
30. 1,2-dichloropropane
31. bromodichloromethane
32. 1-bromo-2-chloroethane (SS)
33. 2-chloroethyl vinyl ether
34. *cis*-1,3-dichloropropene
35. toluene
36. tetrachloroethene
37. *trans*-1,3-dichloropropene
38. 1,1,2-trichloroethane
39. dibromochloromethane
40. 1,3-dichloropropane
41. 1,2-dibromoethane
42. chlorobenzene
43. ethylbenzene
44. 1,1,1,2-tetrachloroethane
45. *m*-xylene
46. *p*-xylene
47. 1-chloro-2-fluorobenzene (SS)
48. *o*-xylene
49. styrene
50. bromoform
51. isopropylbenzene
52. bromobenzene
53. *n*-propylbenzene
54. 1,1,2,2-tetrachloroethane
55. 2-chlorotoluene
56. 1,2,3-trichloropropane
57. 1,3,5-trimethylbenzene
58. 4-chlorotoluene
59. *tert*-butylbenzene
60. pentachloroethane
61. 1,2,4-trimethylbenzene
62. *sec*-butylbenzene
63. *p*-isopropyltoluene
64. 1,3-dichlorobenzene
65. 1,4-dichlorobenzene
66. benzyl chloride
67. *n*-butylbenzene
68. 1,2-dichlorobenzene
69. 1,2-dibromo-3-chloropropane
70. 2-bromo-1-chlorobenzene (SS)
71. hexachlorobutadiene
72. 1,2,4-trichlorobenzene
73. naphthalene
74. 1,2,3-trichlorobenzene
75. 2-methylnaphthalene (40ppb)
76. 1-methylnaphthalene (40ppb)

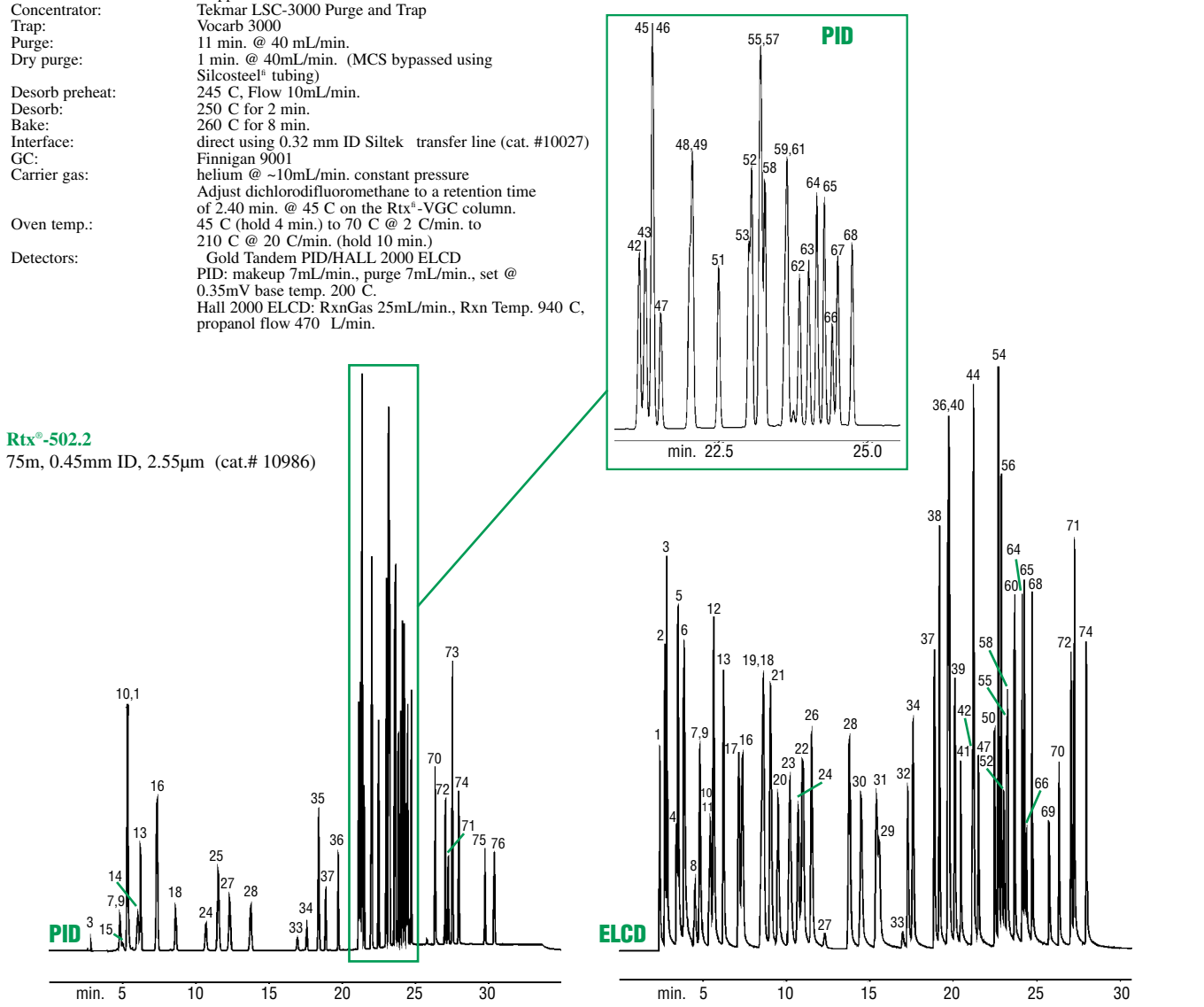
GC_EV00421

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.

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:	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 0.32 mm 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., Rxn Temp. 940 C, propanol flow 470 L/min.



1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethene
8. Freon® 113
9. chloromethyl methyl ether
10. iodomethane (40ppb)
11. allyl chloride
12. methylene chloride
13. *trans*-1,2-dichloroethene
14. methyl *tert*-butyl ether
15. *tert*-butyl alcohol (40ppb)
16. chloroprene
17. 1,1-dichloroethane
18. *cis*-1,2-dichloroethene
19. 2,2-dichloropropane
20. bromochloromethane
21. chloroform
22. carbon tetrachloride
23. 1,1,1-trichloroethane
24. 1,1-dichloropropene
25. benzene
26. 1,2-dichloroethane
27. fluorobenzene (SS)
28. trichloroethene
29. dibromomethane
30. 1,2-dichloropropane
31. bromodichloromethane
32. 1-bromo-2-chloroethane (SS)
33. 2-chloroethyl vinyl ether
34. *cis*-1,3-dichloropropene
35. toluene
36. tetrachloroethene
37. *trans*-1,3-dichloropropene
38. 1,1,2-trichloroethane
39. dibromochloromethane
40. 1,3-dichloropropane
41. 1,2-dibromoethane
42. chlorobenzene
43. ethylbenzene
44. 1,1,1,2-tetrachloroethane
45. *m*-xylene
46. *p*-xylene
47. 1-chloro-2-fluorobenzene (SS)
48. *o*-xylene
49. styrene
50. bromoform
51. isopropylbenzene
52. bromobenzene
53. *n*-propylbenzene
54. 1,1,2,2-tetrachloroethane
55. 2-chlorotoluene
56. 1,2,3-trichloropropane
57. 1,3,5-trimethylbenzene
58. 4-chlorotoluene
59. *tert*-butylbenzene
60. pentachloroethane
61. 1,2,4-trimethylbenzene
62. *sec*-butylbenzene
63. *p*-isopropyltoluene
64. 1,3-dichlorobenzene
65. 1,4-dichlorobenzene
66. benzyl chloride
67. *n*-butylbenzene
68. 1,2-dichlorobenzene
69. 1,2-dibromo-3-chloropropane
70. 2-bromo-1-chlorobenzene (SS)
71. hexachlorobutadiene
72. 1,2,4-trichlorobenzene
73. naphthalene
74. 1,2,3-trichlorobenzene
75. 2-methylnaphthalene (40ppb)
76. 1-methylnaphthalene (40ppb)

GC_EV00422

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

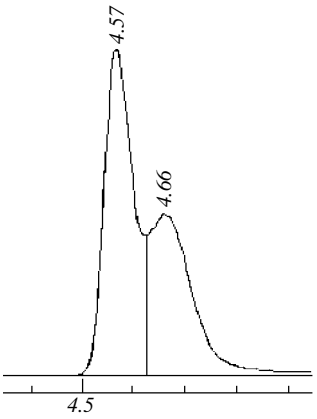
Importance of Resolution in GC Analysis

Figure 43 shows the effects of resolution on quantification for methylene chloride and Freon® 113 (1,1,2-trichloro-1,2,2-trifluororoethane) 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 methlyene 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.

Figure 43.
Peak shape affects quantification:
separation of methylene chloride and Freon® 113 on a "VRX" column.



Quantification Values

Methylene Chloride & Freon® 113		
Replicate 1	19.85	18.48
Replicate 2	19.29	18.48
Replicate 3	19.36	18.52
Methylene Chloride		
Replicate 1	21.48	
Replicate 2	20.79	
Replicate 3	20.95	
Freon® 113		
Replicate 1		16.3
Replicate 2		16.46
Replicate 3		16.25

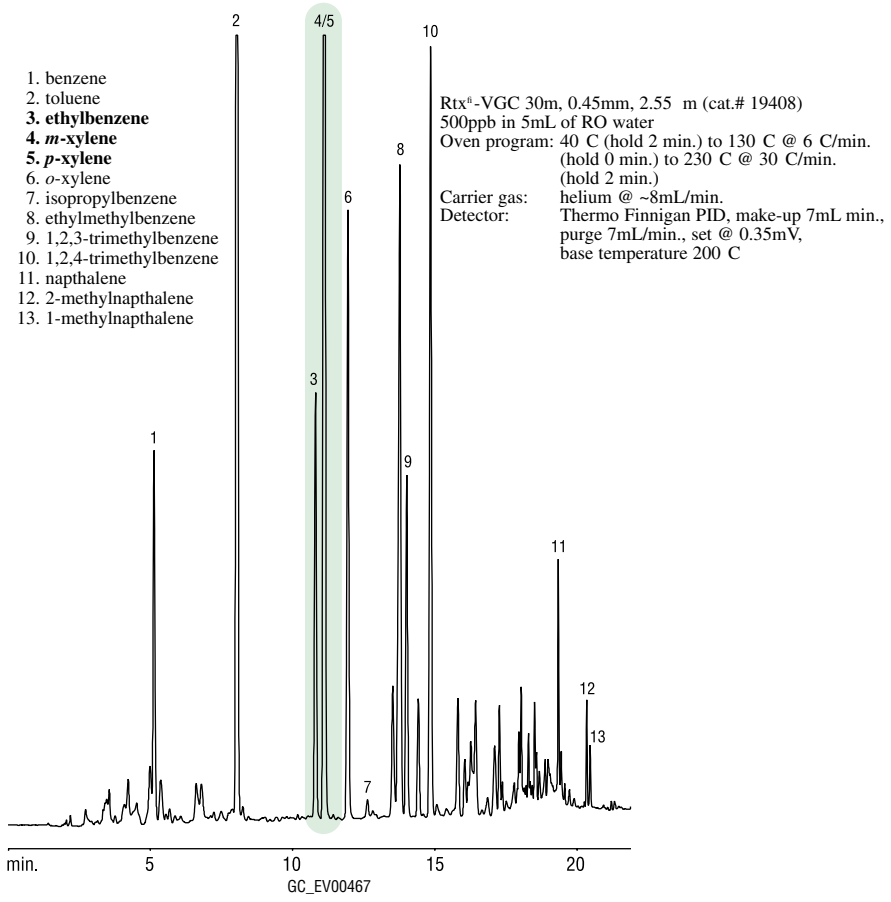
Results of resolution tests @ 20ppb. 20ppb each component in 5mL RO water, analyzed according to US EPA Method 5030A/502.2.

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).

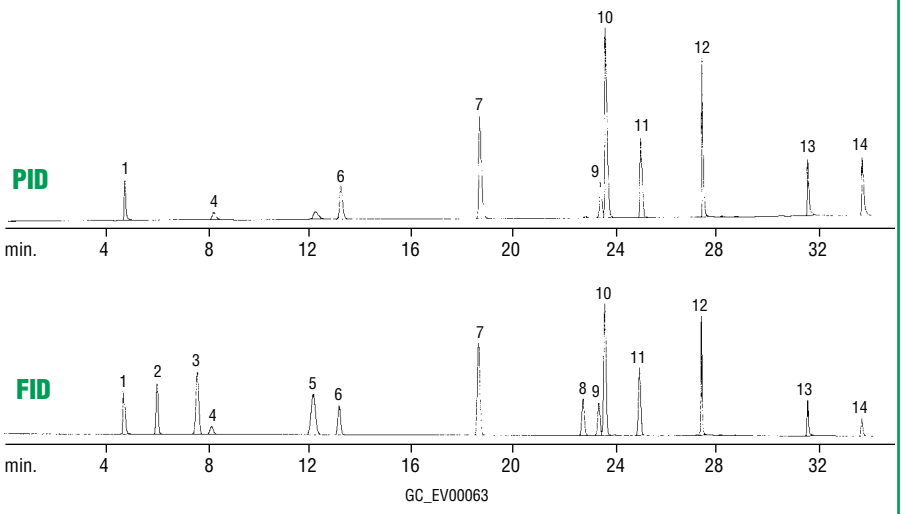
Figure 44.

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.

Figure 45.
For VPH analysis on an Rtx®-502.2 column, use PID for aromatic compounds and FID for aliphatic compounds



105m, 0.53mm ID, 3.0 m Rtx®-502.2 (cat.# 10910).
Concentration: on-column at levels listed

Oven temp:	45 C to 90 C @ 3 C/min., to 140 @ 5 C/min., to 230 C @ 45 C/min. (hold 8 min.)
Carrier gas:	helium @ 15mL/min. Tekmar® Model LSC 2000
Trap:	BTEX
Purge:	helium @ 40mL/min. for 11 min.
Dry purge:	2 min.
Desorb preheat:	245 C
Desorb:	2 min. @ 250 C
Bake:	6 min. @ 260 C

Chromatograms courtesy of Severn Trent Laboratories, Burlington, VT.

1. methanol
2. *n*-pentane (40ng)
3. 2-methylpentane (60ng)
4. methyl *tert*-butyl ether (60ng)
5. 2,2,4-trimethylpentane (60ng)
6. benzene (20ng)
7. toluene
8. *n*-nonane
9. ethylbenzene (20ng)
10. *p*+*m*-xylene (40ng ea.)
11. *o*-xylene (40ng)
12. 1,2,4-trimethylbenzene (40ng)
13. naphthalene (40ng)
14. 2,5-dibromotoluene (SS)

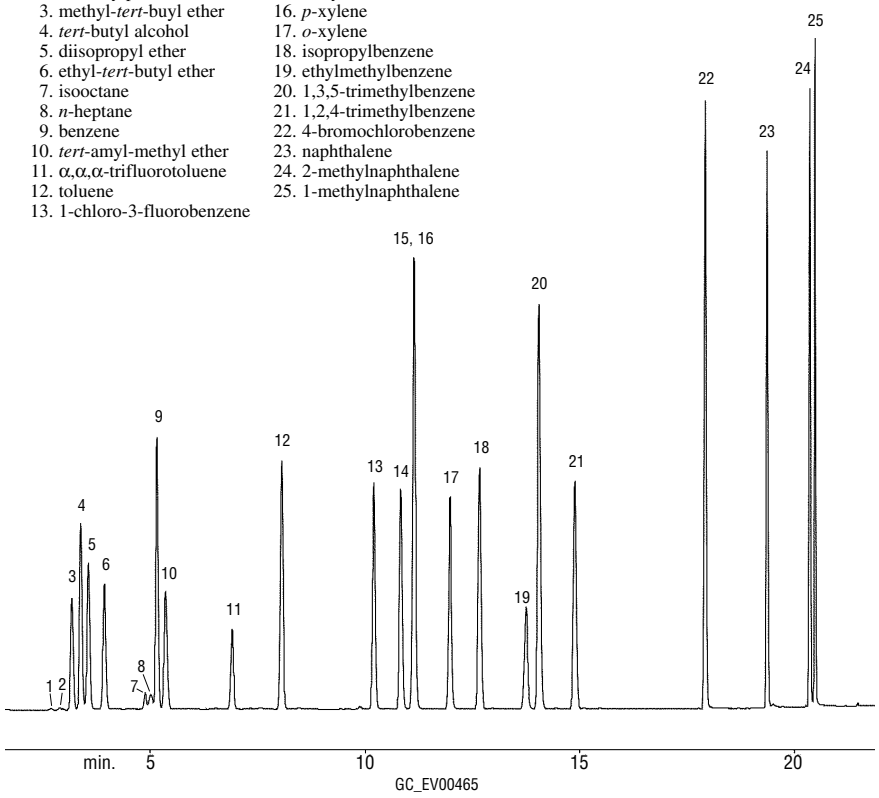
Gasoline Analysis with Oxygenates and Alcohols

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.

1. 2-methylpentane
2. 3-methylpentane
3. methyl-*tert*-buyl ether
4. *tert*-butyl alcohol
5. diisopropyl ether
6. ethyl-*tert*-butyl ether
7. isooctane
8. *n*-heptane
9. benzene
10. *tert*-amyl-methyl ether
11. α,α,α-trifluorotoluene
12. toluene
13. 1-chloro-3-fluorobenzene
14. ethylbenzene
15. *m*-xylene
16. *p*-xylene
17. *o*-xylene
18. isopropylbenzene
19. ethylmethylbenzene
20. 1,3,5-trimethylbenzene
21. 1,2,4-trimethylbenzene
22. 4-bromochlorobenzene
23. naphthalene
24. 2-methylnaphthalene
25. 1-methylnaphthalene



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-methynapthalene 150ppb; ethylmethylbenzene 50ppb.

Concentrator:	Tekmar LSC-3100 Purge and Trap
Trap:	Vocarb 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.

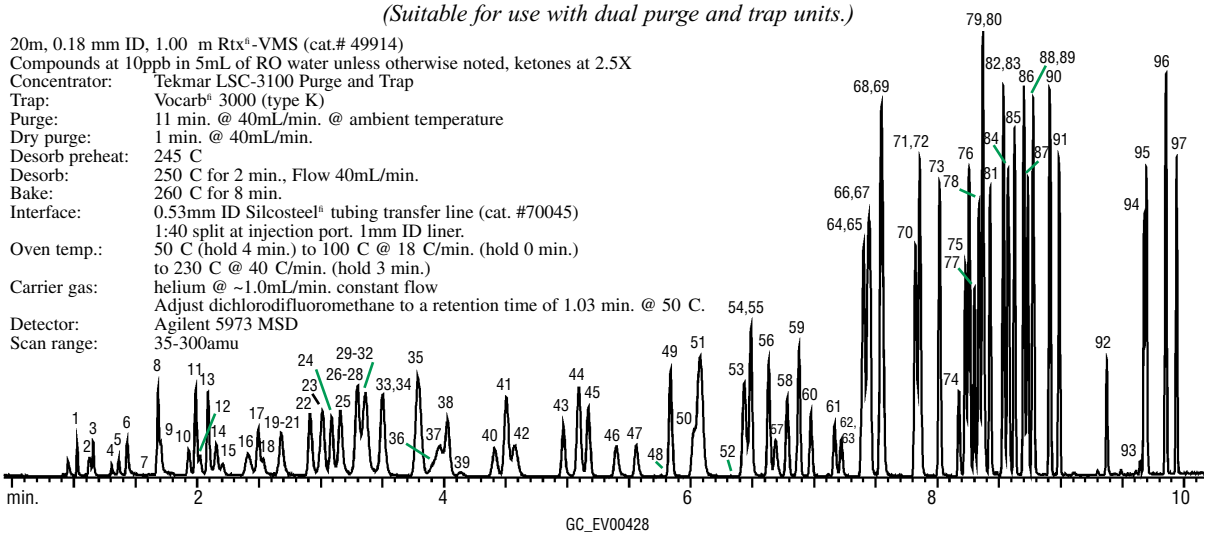
Retention Time (min.)	Rtx®-VMS	Rtx®-624	Rtx®-502.2	Rtx®-VRX	Rtx®-1
2-Chlorotoluene	8.35	8.63	8.80	8.49	8.38
4-Chlorotoluene	8.44	8.69	8.84	8.53	8.41
RT diff.	0.09	0.06	0.04	0.04	0.03

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.)

20m, 0.18 mm ID, 1.00 m Rtx®-VMS (cat.# 49914)
Compounds at 10ppb in 5mL of RO water unless otherwise noted, ketones at 2.5X

Concentrator:	Tekmar LSC-3100 Purge and Trap
Trap:	Vocarb® 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 40mL/min.
Bake:	260 C for 8 min.
Interface:	0.53mm ID Silcosteel® tubing transfer line (cat. #70045) 1:40 split at injection port. 1mm ID liner.
Oven temp.:	50 C (hold 4 min.) to 100 C @ 18 C/min. (hold 0 min.) to 230 C @ 40 C/min. (hold 3 min.)
Carrier gas:	helium @ ~1.0mL/min. constant flow Adjust dichlorodifluoromethane to a retention time of 1.03 min. @ 50 C.
Detector:	Agilent 5973 MSD
Scan range:	35-300amu



1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. ethanol (2500ppb)
8. 1,1-dichloroethene
9. carbon disulfide (40ppb)
10. allyl chloride
11. methylene chloride
12. acetone
13. *trans*-1,2-dichloroethene
14. methyl *tert*-butyl ether
15. *tert*-butyl alcohol (100ppb)
16. diisopropyl ether
17. 1,1-dichloroethane
18. acrylonitrile
19. vinyl acetate
20. allyl alcohol (250ppb)
21. ethyl-*tert*-buyl ether
22. *cis*-1,2-dichloroethene
23. 2,2-dichloropropane
24. bromochloromethane
25. chloroform
26. ethyl acetate
27. carbon tetrachloride
28. methyl acrylate
29. propargyl alcohol (500ppb)
30. dibromofluoromethane (SMC)
31. tetrahydrofuran
32. 1,1,1-trichloroethane
33. 2-butanone
34. 1,1-dichloropropene
35. benzene
36. pentafluorobenzene (IS)
37. *tert*-amyl-methyl ether
38. 1,2-dichloroethane
39. isobutyl alcohol (500ppb)
40. isopropyl acetate
41. trichloroethene
42. 1,4-difluorobenzene (SMC)
43. dibromomethane
44. 1,2-dichloropropane
45. bromodichloromethane
46. methyl methacrylate
47. *n*-propyl acetate
48. 2-chloroethanol (2500ppb)
49. *cis*-1,3-dichloropropene
50. toluene-d8(SMC)
51. toluene
52. pyridine (250ppb)
53. tetrachloroethene
54. 4-methyl-2-pentanone
55. *trans*-1,3-dichloropropene
56. 1,1,2-trichloroethane
57. ethyl methacrylate
58. dibromochloromethane
59. 1,3-dichloropropane
60. 1,2-dibromoethane
61. *n*-butyl acetate
62. 2-hexanone
63. 2-picoline (250ppb)
64. chlorobenzene-d5 (IS)
65. chlorobenzene
66. ethylbenzene
67. 1,1,1,2-tetrachloroethane
68. *m*-xylene
69. *p*-xylene
70. *o*-xylene
71. stryrene
72. bromoform
73. isopropylbenzene
74. 4-bromo-1-fluorobenzene (SMC)
75. bromobenzene
76. *n*-propylbenzene
77. 1,1,2,2-tetrachloroethane
78. 2-chlorotoluene
79. 1,3,5-trimethylbenzene
80. 1,2,3-trichloropropane
81. 4-chlorotoluene
82. *tert*-butylbenzene
83. pentachloroethane
84. 1,2,4-trimethylbenzene
85. *sec*-butylbenzene
86. *p*-isopropyltoluene
87. 1,3-dichlorobenzene
88. 1,4-dichlorobenzene-d4 (IS)
89. 1,4-dichlorobenzene
90. *n*-butylbenzene
91. 1,2-dichlorobenzene
92. 1,2-dibromo-3-chloropropane
93. nitrobenzene (250ppb)
94. hexachlorobutadiene
95. 1,2,4-trichlorobenzene
96. naphthalene
97. 1,2,3-trichlorobenzene

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.

Figure 48.

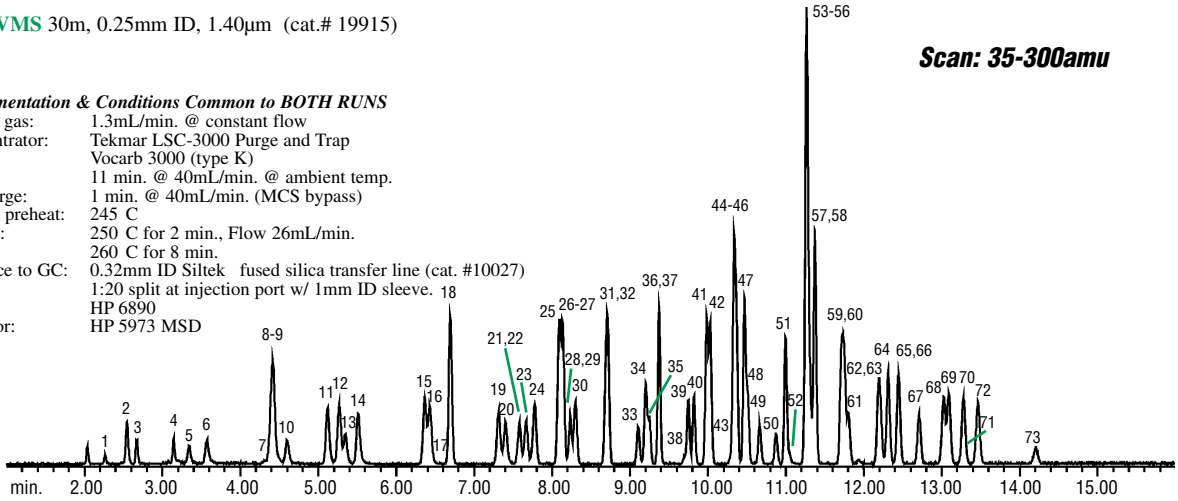
EPA Method 8240 analysis using an Rtx®-VMS column. Alcohols require scans below 35amu.

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:	Vocarb 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.
GC:	HP 6890
Detector:	HP 5973 MSD



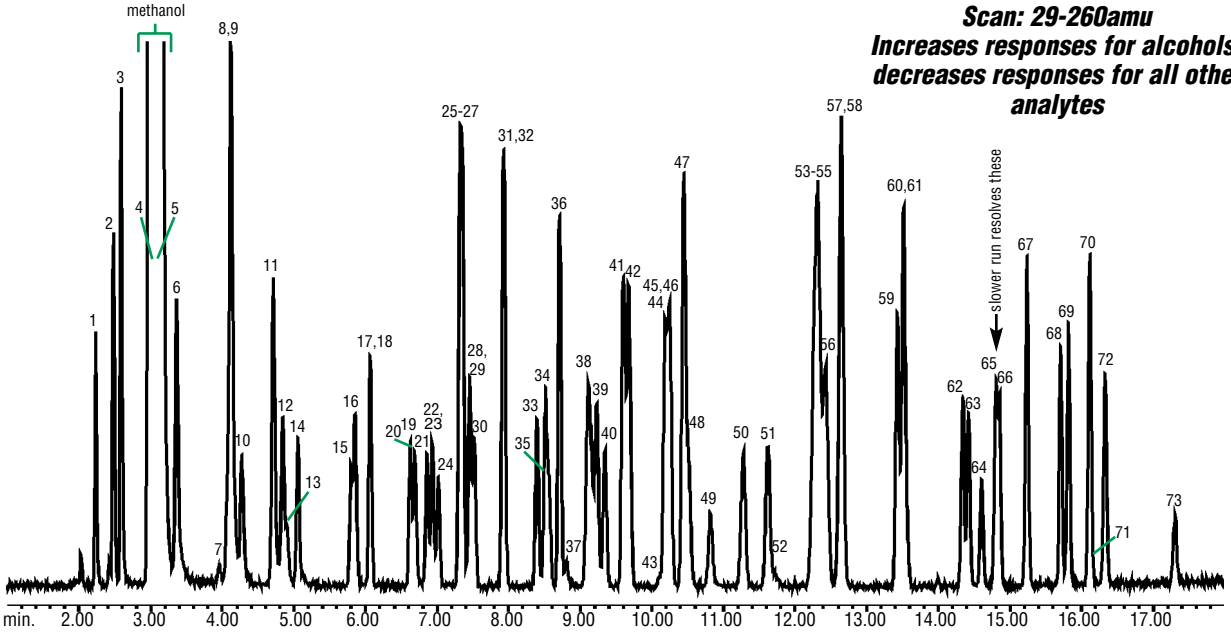
Top chromatogram:

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



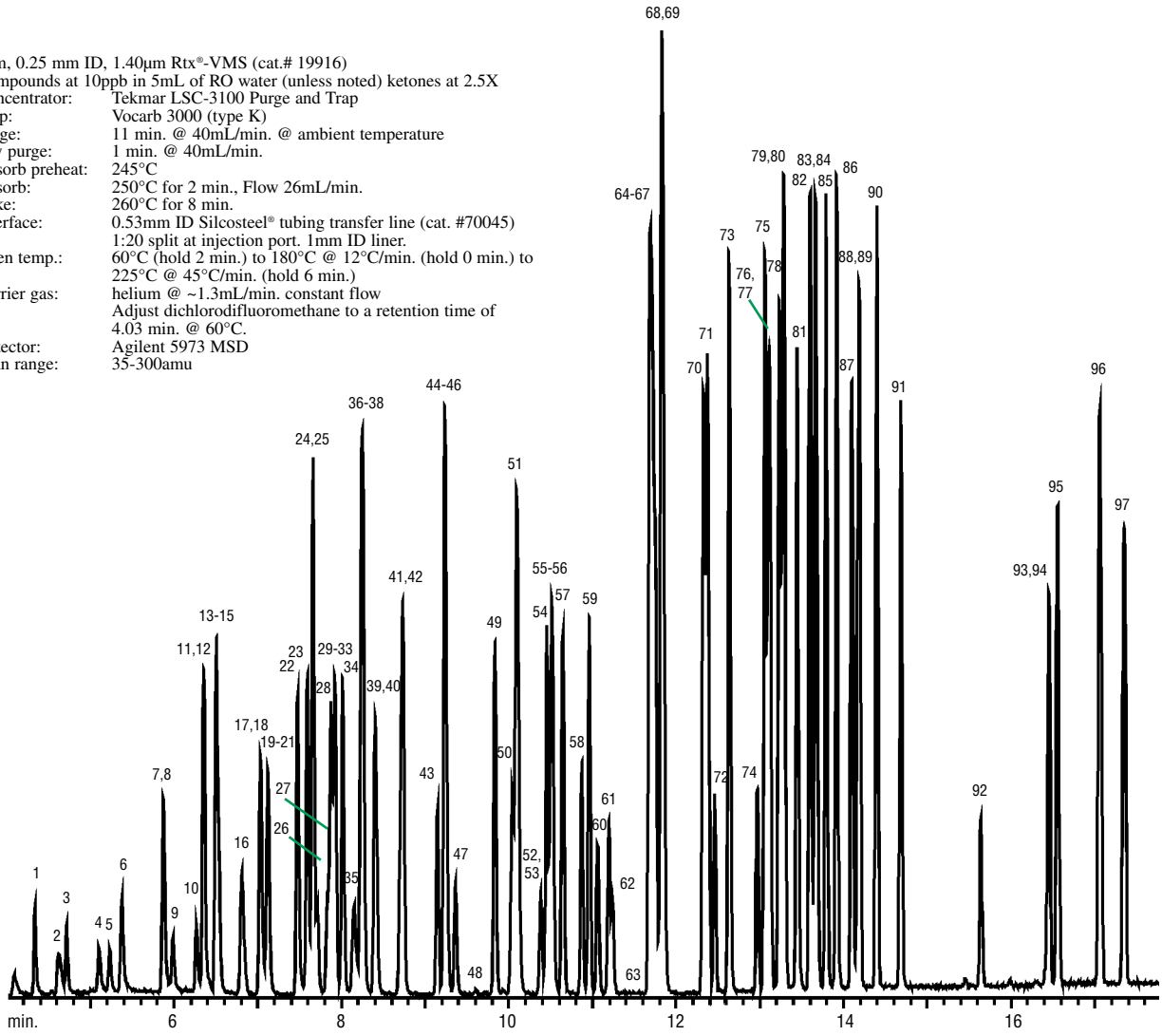
1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. ethanol
8. 1,1-dichloroethene
9. carbon disulfide
10. iodomethane
11. allyl chloride
12. methylene chloride
13. acetone
14. *trans*-1,2-dichloroethene
15. 1,1-dichloroethane
16. acrylonitrile
17. allyl alcohol
18. vinyl acetate
19. bromochloromethane
20. chloroform
21. carbon tetrachloride
22. propargyl alcohol
23. 1,1,1-trichloroethane
24. 2-butanone
25. benzene
26. propionitrile
27. methacrylonitrile
28. 1,2-dichloroethane-d4
29. isobutyl alcohol
30. 1,2-dichloroethane
31. trichloroethene
32. 1,4-difluorobenzene
33. dibromomethane
34. 1,2-dichloropropane
35. bromodichloromethane
36. methyl methacrylate
37. 1,4-dioxane
38. 2-chloroethanol
39. 2-chloroethyl vinyl ether
40. *cis*-1,3-dichloropropene
41. toluene-d8
42. toluene
43. pyridine
44. 4-methyl-2-pentanone
45. tetrachloroethene
46. *trans*-1,3-dichloropropene
47. ethyl methacrylate
48. 1,1,2-trichloroethane
49. dibromochloromethane
50. 1,2-dibromoethane
51. 2-hexanone
52. 2-picoline
53. chlorobenzene-D5
54. ethylbenzene
55. chlorobenzene
56. 1,1,1,2-tetrachloroethane
57. *m*-xylene
58. *p*-xylene
59. *o*-xylene
60. styrene
61. bromoform
62. 4-bromo-1-fluorobenzene
63. *cis*-1,4-dichloro-2-butene
64. 1,1,2,2-tetrachloroethane
65. 1,2,3-trichloropropane
66. *trans*-1,4-dichloro-2-butene
67. pentachloroethane
68. 1,3-dichlorobenzene
69. 1,4-dichlorobenzene
70. benzyl chloride
71. malononitrile
72. 1,2-dichlorobenzene
73. 1,2-dibromo-3-chloropropane

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: Vocarb 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
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. ethanol (2500ppb)
8. 1,1-dichloroethene
9. carbon disulfide (40ppb)
10. allyl chloride
11. methylene chloride
12. acetone
13. *trans*-1,2-dichloroethene
14. *tert*-butyl alcohol (100ppb)
15. methyl *tert*-butyl ether
16. diisopropyl ether
17. 1,1-dichloroethane
18. acrylonitrile
19. vinyl acetate*
20. allyl alcohol (250ppb)
21. ethyl-*tert*-butyl ether*
22. *cis*-1,2-dichloroethene
23. 2,2-dichloropropane
24. bromochloromethane
25. chloroform
26. ethyl acetate
27. methyl acrylate
28. propargyl alcohol (500ppb)
29. dibromofluoromethane (SMC)
30. tetrahydrofuran
31. carbon tetrachloride
32. 2-butanone
33. 1,1,1-trichloroethane
34. 1,1-dichloropropene
35. pentafluorobenzene (IS)
36. *tert*-amyl methyl ether
37. benzene
38. isobutyl alcohol (500ppb)
39. 1,2-dichloroethane
40. isopropyl acetate
41. 1,4-difluorobenzene (SMC)
42. trichloroethene
43. dibromomethane
44. bromodichloromethane
45. 1,2-dichloropropane
46. methyl methacrylate
47. *n*-propyl acetate
48. 2-chloroethanol (2500ppb)
49. *cis*-1,3-dichloropropene
50. toluene-d8 (SMC)
51. toluene
52. 4-methyl-2-pentanone
53. pyridine (250ppb)
54. *trans*-1,3-dichloropropene
55. ethyl methacrylate
56. tetrachloroethene
57. 1,1,2-trichloroethane
58. dibromochloromethane
59. 1,3-dichloropropane
60. *n*-butyl acetate
61. 1,2-dibromoethane
62. 2-hexanone
63. 2-picoline (250ppb)
64. ethylbenzene
65. chlorobenzene-D5 (IS)
66. chlorobenzene
67. 1,1,1,2-tetrachloroethane
68. *m*-xylene
69. *p*-xylene
70. *o*-xylene
71. styrene
72. bromoform
73. isopropylbenzene
74. 4-bromo-1-fluorobenzene (SMC)
75. *n*-propylbenzene
76. 1,1,2,2-tetrachloroethane
77. bromobenzene
78. 1,3,5-trimethylbenzene
79. 2-chlorotoluene
80. 1,2,3-trichloropropane
81. 4-chlorotoluene
82. *tert*-butylbenzene
83. 1,2,4-trimethylbenzene
84. pentachloroethene
85. *sec*-butylbenzene
86. *p*-isopropyltoluene
87. 1,3-dichlorobenzene
88. 1,4-dichlorobenzene-d4 (IS)
89. 1,4-dichlorobenzene
90. *n*-butylbenzene
91. 1,2-dichlorobenzene
92. 1,2-dibromo-3-chloropropane
93. nitrobenzene (250ppb)
94. hexachlorobutadiene
95. 1,2,4-trichlorobenzene
96. naphthalene
97. 1,2,3-trichlorobenzene

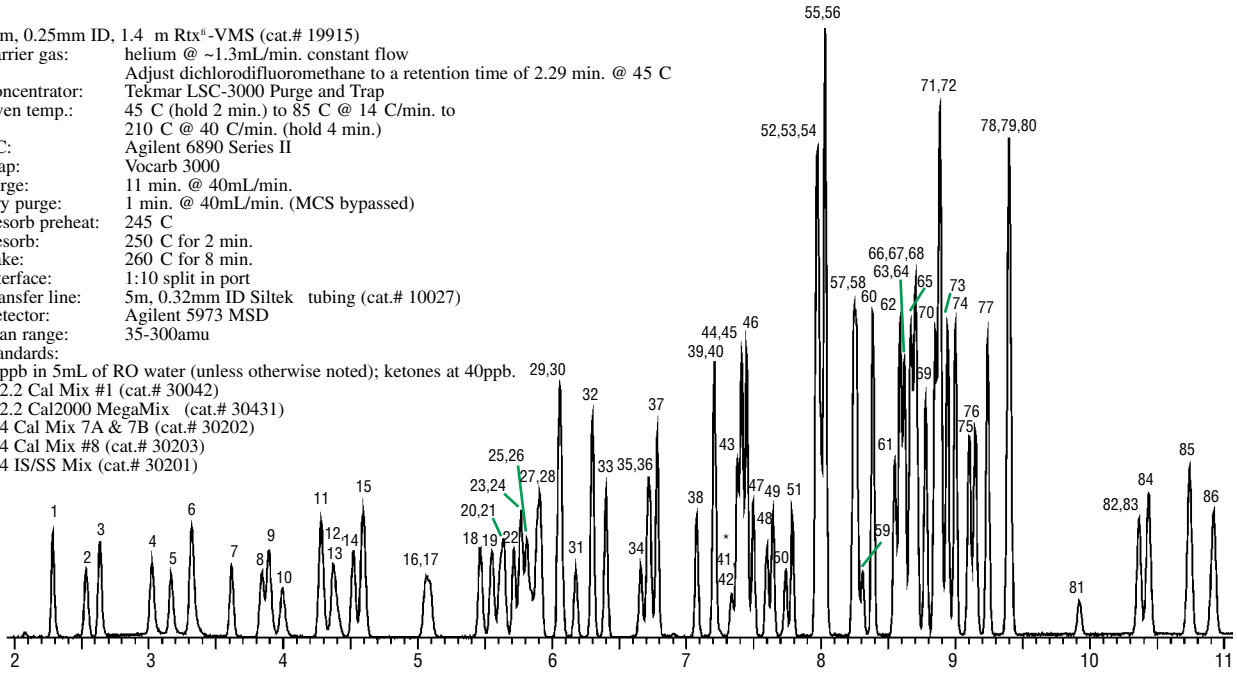
GC_EV00427

*These compounds share ions, and coelute on "624"-type columns.

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: Vocarb 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)



1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. diethyl ether
8. 1,1-dichloroethene
9. carbon disulfide (40ppb)
10. iodomethane (40ppb)
11. allyl chloride
12. methylene chloride
13. acetone
14. *trans*-1,2-dichloroethene
15. methyl *tert*-butyl ether
16. 1,1-dichloroethane
17. acrylonitrile
18. *cis*-1,2-dichloroethene
19. 2,2-dichloropropane
20. bromochloromethane
21. chloroform
22. methyl acrylate
23. carbon tetrachloride
24. tetrahydrofuran (40ppb)
25. 1,1,1-trichloroethane
26. 2-butanone
27. 1,1-dichloropropene
28. 1-chlorobutane
29. benzene
30. propionitrile
31. 1,2-dichloroethane
32. fluorobenzene
33. trichloroethene
34. dibromomethane
35. 1,2-dichloropropane
36. bromodichloromethane
37. methyl methacrylate
38. *cis*-1,3-dichloropropene
39. toluene
40. chloroacetonitrile
41. 2-nitropropane*
42. 1,1-dichloropropanone*
43. 4-methyl-2-pentanone
44. tetrachloroethene
45. *trans*-1,3-dichloropropene
46. ethyl methacrylate
47. 1,1,2-trichloroethane
48. dibromochloromethane
49. 1,3-dichloropropane
50. 1,2-dibromoethane
51. 2-hexanone
52. ethylbenzene
53. chlorobenzene
54. 1,1,1,2-tetrachloroethane
55. *m*-xylene
56. *p*-xylene
57. *o*-xylene
58. styrene
59. bromoform
60. isopropylbenzene
61. 4-bromofluorobenzene
62. *n*-propylbenzene
63. bromobenzene
64. 1,1,2,2-tetrachloroethane
65. 1,3,5-trimethylbenzene
66. 2-chlorotoluene
67. 1,2,3-trichloropropane
68. *trans*-1,4-dichloro-2-butene
69. 4-chlorotoluene
70. *tert*-butylbenzene
71. 1,2,4-trimethylbenzene
72. pentachloroethane
73. *sec*-butylbenzene
74. *p*-isopropyltoluene
75. 1,3-dichlorobenzene
76. 1,4-dichlorobenzene
77. *n*-butylbenzene
78. hexachloroethane
79. 1,2-dichlorobenzene-d4
80. 1,2-dichlorobenzene
81. 1,2-dibromo-3-chloropropane
82. nitrobenzene
83. hexachlorobutadiene
84. 1,2,4-trichlorobenzene
85. naphthalene
86. 1,2,3-trichlorobenzene

GC_EV00430

**These compounds share a quantitation ion (43)*

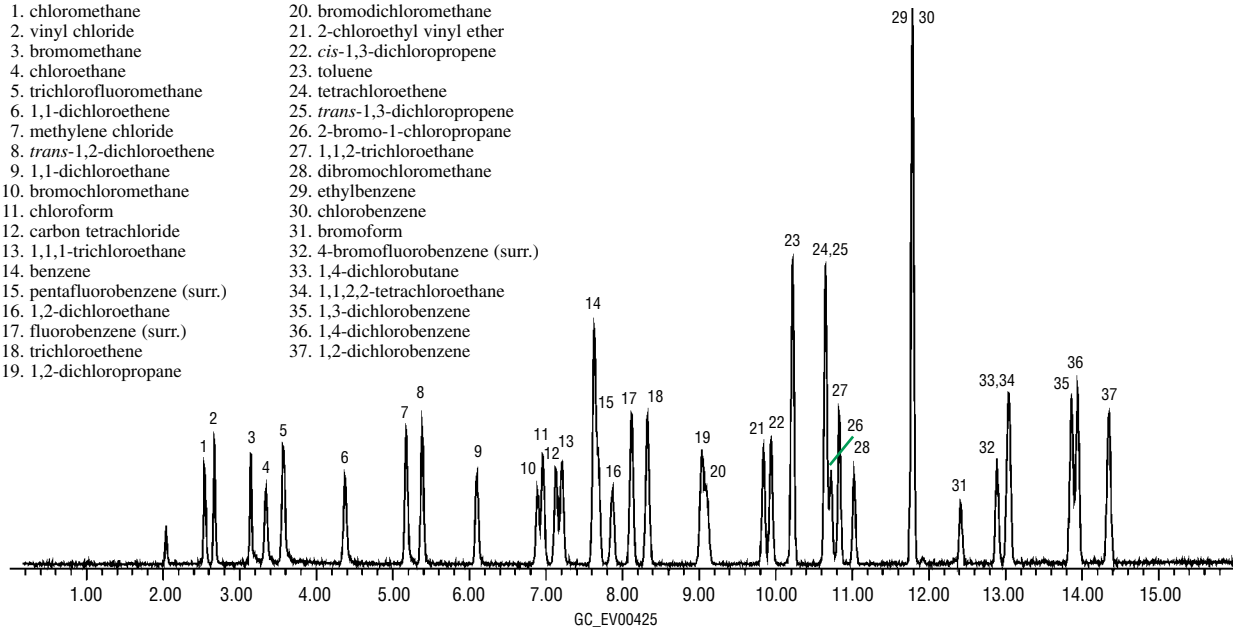
Method 524.2: In EPA method updates, such as Method 524.2, rev. IV, minor ions from newly listed target compounds interfere with the quantification ions from other target compounds.[17] An example of this problem occurs when a 75m, 0.53mm ID "624/1301" column is used to resolve methyl acrylate and propionitrile. 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, Cinncinnati, OH. 1992.

Figure 51.

Gaseous analytes resolved to baseline on an Rtx®-VMS column.

1. chloromethane
2. vinyl chloride
3. bromomethane
4. chloroethane
5. trichlorofluoromethane
6. 1,1-dichloroethene
7. methylene chloride
8. *trans*-1,2-dichloroethene
9. 1,1-dichloroethane
10. bromochloromethane
11. chloroform
12. carbon tetrachloride
13. 1,1,1-trichloroethane
14. benzene
15. pentafluorobenzene (surr.)
16. 1,2-dichloroethane
17. fluorobenzene (surr.)
18. trichloroethene
19. 1,2-dichloropropane
20. bromodichloromethane
21. 2-chloroethyl vinyl ether
22. *cis*-1,3-dichloropropene
23. toluene
24. tetrachloroethene
25. *trans*-1,3-dichloropropene
26. 2-bromo-1-chloropropane
27. 1,1,2-trichloroethane
28. dibromochloromethane
29. ethylbenzene
30. chlorobenzene
31. bromoform
32. 4-bromofluorobenzene (surr.)
33. 1,4-dichlorobutane
34. 1,1,2,2-tetrachloroethane
35. 1,3-dichlorobenzene
36. 1,4-dichlorobenzene
37. 1,2-dichlorobenzene



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:	Vocarb 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		
Component Name	**Rtx-502.2**	**Rtx-1**	**Rtx-624**
dichlorodifluoromethane	4.173	4.24	4.255
C3	4.181	4.241	4.211
chloromethane	4.838	4.513	4.857
C4	5.136	5.319	6.321
vinyl chloride	5.159	4.872	5.243
ethylene oxide	6.289	5.467	6.389
bromomethane	6.527	5.604	6.412
chloroethane	6.812	5.938	6.798
ethanol	7.109	6.261	9.056
C5	7.692	8.133	8.069
trichlorofluoromethane	7.715	7.426	7.672
diethyl ether	8.857	8.232	8.977
1,1,2-trichloro-1,2,2-trifluoroethane	9.142	9.745	10.044
acrolein	9.285	6.831	9.817
acetone	9.558	7.042	10.6
1,1-dichloroethene	10.117	8.74	9.953
tert-butyl alcohol	10.516	8.864	13.546
acetonitrile	10.661	6.571	12.909
iodomethane	11.311	8.567	10.634
methyl iodide	11.311	8.567	10.634
allyl chloride	11.717	9.323	11.9
n-propylamine	11.815	9.36	11.69
carbon disulfide	12.069	9.658	10.747
dichloromethane	12.169	9.001	13.034
methylene chloride	12.169	9.001	13.034
allyl alcohol	12.659	10.215	16.115
methyl *tert*-butyl ether	12.785	12.074	13.734
acrylonitrile	13.429	7.947	14.158
trans-1,2-dichloroethene	13.438	11.346	13.725
C6	13.828	14.557	14.668
propargyl alcohol	14.458	11.32	18.532
di-isopropyl ether	14.874	14.602	15.745
1,1-dichloroethane	15.151	11.849	15.616
vinyl acetate	15.256	12.283	15.883
2-butanone	16.82	12.834	17.766
methyl ethyl ketone	16.82	12.834	17.766
propionitrile	17.053	10.733	18.167
2,2-dichloropropane	17.337	14.659	17.506
cis-1,2-dichloroethene	17.478	13.924	17.628
methacrylonitrile	17.837	12.732	18.505
methylacrylate	17.947	14.54	18.351
isobutyl alcohol	18.015	15.723	20.048
chloroform	18.104	14.659	18.625
pentafluorobenzene	18.628	16.371	19.273
bromochloromethane	18.667	14.289	18.355
tetrahydrofuran	18.777	15.554	18.341
dibromofluoromethane	18.893	14.966	19.086
1,1,1-trichloroethane	19.575	17.042	18.972
cyclohexane	19.619	18.779	16.854
1-chlorobutane	19.724	17.221	19.314

	Retention Time		
Component Name	**Rtx-502.2**	**Rtx-1**	**Rtx-624**
1,1-dichloropropene	20.163	17.808	19.448
carbon tetrachloride	20.502	18.493	19.333
1,2-dichloroethane-d4	20.726	16.133	20.04
benzene-d6	20.91	17.998	19.888
C7	20.935	21.532	20.582
1,2-dichloroethane	21.047	16.411	20.263
benzene	21.097	18.124	19.999
fluorobenzene	21.897	18.917	20.802
2-chloroethanol	21.951	17.171	23.115
trichloroacetonitrile	22.125	33.558	30.181
1,4-difluorobenzene	22.209	19.061	21.179
trichloroethene	23.326	20.674	21.713
trifluorotoluene	23.607	21.311	22.527
1,2-dichloropropane	23.924	20.036	22.378
methyl methacrylate	24.233	21.258	22.632
chloroacetonitrile	24.239	17.035	23.993
bromodichloromethane	24.743	20.545	23.045
1,4-dioxane	24.808	20.645	22.644
dibromomethane	24.881	19.975	22.668
2-nitropropane	24.999	19.356	23.762
2-chloroethyl vinyl ether	25.888	22.108	23.796
dichloroacetonitrile	25.894	19.998	25.478
4-methyl-2-pentanone (MIBK)	26.001	22.887	24.458
epichlorohydrin	26.109	20.907	23.971
1,1-dichloropropanone	26.444	22.124	25.469
cis-1,3-dichloropropene	26.674	22.744	24.104
C8	27.186	27.642	24.868
toluene-d8	27.502	24.835	24.612
pyridine	27.525	22.8	24.598
toluene	27.78	25.081	24.768
trans-1,3-dichloropropene	28.428	24.014	25.417
ethyl methacrylate	28.455	25.852	25.489
1,1,2-trichloroethane	28.925	24.384	25.829
2-bromo-1-chloropropane	28.965	25.018	25.569
2-hexanone	29.03	25.778	26.326
1,3-dichloropropane	29.826	25.124	26.205
tetrachloroethene	30.036	27.8	25.928
dibromochloromethane	30.685	26.024	26.639
2-picoline	31.242	27.462	26.977
1,2-dibromoethane (EDB)	31.375	26.613	26.924
1-chlorohexane	32.066	29.717	27.831
bromochloroacetonitrile	32.079	25.757	28.837
C9	32.546	32.893	28.243
1,1,1-trichloro-2-propanane	32.633	28.967	28.104
chlorobenzene-d5	32.749	29.372	27.887
ethylbenzene-d10	32.756	30.191	27.934
chlorobenzene	32.864	29.473	27.949
ethylbenzene-d5	32.976	30.378	28.089
3-chloropropionitrile	33.001	24.824	29.176
1,1,1,2-tetrachloroethane	33.03	29.429	28.133
ethylbenzene	33.087	30.476	28.118

	Retention Time		
Component Name	**Rtx-502.2**	**Rtx-1**	**Rtx-624**
m-xylene	33.351	30.898	28.37
p-xylene	33.354	30.961	28.358
1-chloro-2-fluorobenzene	33.68	30.232	28.605
malononitrile	34.623	26.078	31.796
o-xylene	34.877	32.149	29.238
styrene	34.975	31.843	29.294
1,3-dichloro-2-propanol	36.032	31.018	31.41
bromoform	36.123	30.983	29.746
isopropylbenzene	36.139	33.718	30.014
cis-1,4-dichloro-2-butene	36.312	31.594	30.302
1,4-dichlorobutane	36.471	31.864	30.377
1,1,2,2-tetrachloroethane	36.735	32.053	30.779
4-bromofluorobenzene	37.004	33.318	30.426
1,2,3-trichloropropane	37.233	32.402	30.902
C10	37.305	37.539	31.095
trans-1,4-dichloro-2-butene	37.516	32.626	30.929
n-propylbenzene	37.566	35.151	30.895
bromobenzene	37.652	33.875	30.772
dibromoacetonitrile	37.812	30.956	31.86
1,3,5-trimethylbenzene	38.133	35.817	31.268
2-chlorotoluene	38.166	34.986	31.145
4-chlorotoluene	38.33	35.214	31.4
tert-butylbenzene	39.393	37.008	31.959
1,2,4-trimethylbenzene	39.521	37.033	32.089
pentachloroethane	39.576	35.98	32.088
thiophenol	39.606	35.186	31.919
sec-butylbenzene	40.151	37.886	32.428
p-isopropyltoluene	40.657	38.413	32.731
1,3-dichlorobenzene	40.938	37.404	32.765
1,4-dichlorobenzene-d4	41.228	37.544	32.92
1,4-dichlorobenzene	41.317	37.624	32.972
C11	41.585	41.76	33.638
benzyl chloride	41.739	37.378	33.279
n-butylbenzene	42.12	39.764	33.612
bis(2-chloroisopropyl)ether	42.498	39.009	34.229
1,2-dichlorobenzene-d4	42.536	38.589	33.74
1,2-dichlorobenzene	42.62	38.685	33.8
hexachloroethane	43.898	40.901	34.244
3-bromochlorobenzene	45.335	41.342	35.231
1,2-dibromo-3-chloropropane	45.501	40.095	35.485
4-bromochlorobenzene	45.567	41.476	35.377
C12	45.586	45.659	35.96
nitrobenzene	45.742	40.282	35.992
2-bromochlorobenzene	46.885	42.47	36.162
bis(2-chloroethoxy)methane	47.514	42.935	36.98
1,2,4-trichlorobenzene	48.643	44.704	37.165
hexachlorobutadiene	49.219	46.558	37.418
C13	49.276	49.295	38.078
naphthalene	49.544	45.073	37.729
1,2,3-trichlorobenzene	50.481	46.076	38.23
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	*trans*-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	*tert*-butylbenzene	21.81
methanol	7.93	2-butanone	14.18	*trans*-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
n-pentane	8.61	1-chlorobutane	14.51	dibromochloromethane	18.40	*sec*-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	*p*-isopropyltoluene	22.22
carbon disulfide	9.65	benzene	14.60	1,2-dibromoethane	18.78	1,3-dichlorobenzene	22.53
Freon® 113	9.70	*n*-heptane (C7)	14.62	*n*-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	*n*-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	*tert*-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	*bis* (2-chloroisopropyl) ether	24.06
acetone	11.24	formic acid	15.37	*m*-xylene	19.53	4-bromo-1-chlorobenzene	24.09
trans-1,2-dichloroethene	11.24	trichloroethene	15.39	*p*-xylene	19.54	benzyl alcohol	24.23
methyl *tert*-butyl ether	11.42	1,4-difluorobenzene	15.58	chlorobenzene-d5	19.55	heptanoic acid	24.29
2-propanol	11.52	*n*-butanol	15.60	1-chloro-2-fluorobenzene	19.67	n-dodecane	24.54
tert-butyl alcohol	11.56	methyl cyclohexane	15.78	*o*-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	*n*-tridecane	26.51
2,4-dimethylpentane	12.68	*n*-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-*tert*-buyl ether	13.08	2-chloroethanol	16.93	*cis*-1,4-dichloro-2-butene	20.97	*n*-tetradecane	28.83
1-propanol	13.18	*cis*-1,3-dichloropropene	17.04	*n*-decane	21.04	2-methylnaphthalene	30.36
cis-1,2-dichloroethene	13.32	1-bromo-2-chloroethane	17.05	*n*-propylbenzene	21.07	1-methyl-naphthalene	30.96
allyl alcohol	13.35	*n*-octane	17.17	1,1,2,2-tetrachloroethane	21.10	*n*-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 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)	Recomended 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
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethene
8. Freon ®113
9. methylene chloride
10. *trans*-1,2-dichloroethene
11. methyl *tert*-butyl ether
12. *tert*-butyl alcohol
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
24. trichloroethene
25. dibromomethane
26. 1,2-dichloropropane
27. bromodichloromethane
28. 2-chloroethyl vinyl ether
29. *cis*-1,3-dichloropropene
30. toluene
31. tetrachloroethene
32. *trans*-1,3-dichloropropene
33. 2-bromo-1-chloropropane
34. 1,1,2-trichloroethane
35. dibromochloromethane
36. 1,3-dichloropropane
37. 1,2-dibromoethane
38. chlorobenzene
39. ethyl benzene
40. 1,1,1,2-tetrachloroethane
41. *m*-xylene
42. *p*-xylene
43. 1-chloro-2-fluorobenzene
44. *o*-xylene
45. styrene
46. bromoform
47. isopropyl benzene
48. bromobenzene
49. *n*-propylbenzene
50. 1,4-dichlorobutane
51. 1,1,2,2-tetrachloroethane
52. 2-chlorotoluene
53. 1,3,5-trimethylbenzene
54. 1,2,3-trichloropropane
55. 4-chlorotoluene
56. *tert*-butylbenzene
57. 1,2,4-trimethylbenzene
58. *sec*-butylbenzene
59. *p*-isopropyl toluene
60. 1,3-dichlorobenzene
61. 1,4-dichlorobenzene
62. *n*-butylbenzene
63. 1,2-dichlorobenzene
64. 4-bromo-1-chlorobenzene
65. 1,2-bromo-3-chloropropane
66. hexachlorobutadiene
67. 1,2,4-trichlorobenzene
68. naphthalene
69. 1,2,3-trichlorobenzene

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

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		

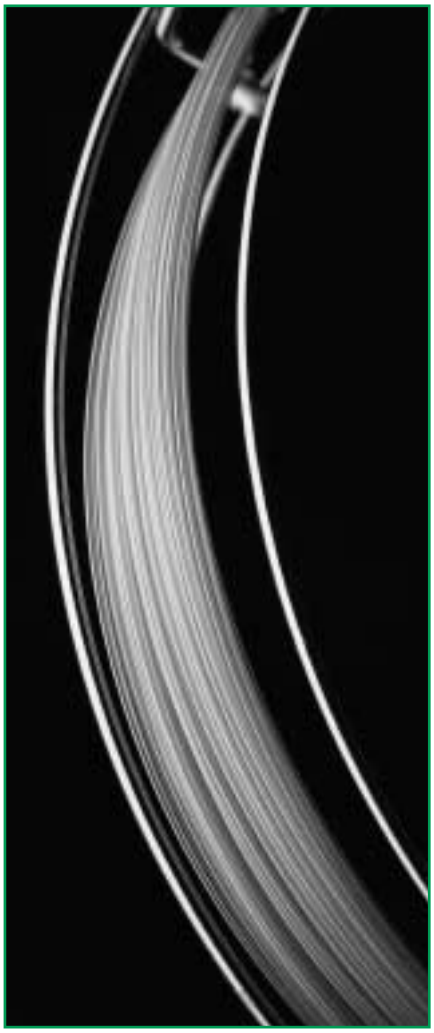
Rtx®-624

- 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	



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

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

			Valcon Polyimide	
Tubing OD	Tubing ID	Valco® #	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 $1x10^{-4}$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.
1mm Split	1.0 ID 6.3 OD x 78.5	20972	20973

for Varian GCs	ID**/OD & Length (mm)	cat.# ea.	cat.# 5-pk.
1mm Split	1.0 ID 6.3 OD x 72	20970	20971

for Shimadzu GCs	ID**/OD & Length (mm)	cat.# ea.	cat.# 5-pk.	cat.# 25-pk.
17A 1mm Split	1.0 ID 5.0 OD x 95	20976	20977	20978

PSS Liners for PerkinElmer GCs	ID*/OD & Length (mm)	Similar to PE part #	cat.# ea.	cat.# 5-pk.	cat.# 25-pk.
PSS Split/Splitless (1mm ID)	1.0 ID 4.0 OD x 86.2	N612-1006	20738	20741	—

for Thermo Finnigan 8000 & TRACE Series GCs	ID**/OD & Length (mm)	Similar to TF Part #	cat.# ea.	cat.# 5-pk.
1mm Split	1.0 ID 8.0 OD x 105	453 20075	20916	20917

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

www.restekcorp.com

Method 8260B

8260A/B Internal Standard Mix

chlorobenzene-d5
1,4-dichlorobenzene-d4
fluorobenzene

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
dibromofluoromethane
1,2-dichloroethane-d4
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
chlorobenzene
1,1-dichloroethene
toluene
trichloroethylene

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-butanone
2-hexanone
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
acrylonitrile
allyl chloride
benzene
bromobenzene
bromochloromethane
bromodichloromethane
bromoform
n-butylbenzene
sec-butylbenzene
tert-butylbenzene
carbon disulfide
carbon tetrachloride
chlorobenzene
2-chloroethanol
chloroform
chloroprene
2-chlorotoluene
4-chlorotoluene
dibromochloromethane
1,2-dibromo-3-chloro-propane
1,2-dibromoethane
dibromomethane
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
cis-1,4-dichloro-2-butene
trans-1,4-dichloro-2-butene
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethene
cis-1,2-dichloroethene
trans-1,2-dichloroethene
1,2-dichloropropane
1,3-dichloropropane
2,2-dichloropropane
1,1-dichloropropene
cis-1,3-dichloropropene
trans-1,3-dichloropropene
diethyl ether
1,4-dioxane
ethylbenzene
ethyl methacrylate
hexachlorobutadiene
iodomethane
isobutyl alcohol
isopropylbenzene
p-isopropyltoluene
methacrylonitrile
methyl acrylate
methyl methacrylate
methylene chloride
naphthalene
nitrobenzene
2-nitropropane
pentachloroethane
propionitrile
n-propylbenzene
styrene
1,1,1,2-tetrachloroethane
1,1,2,2-tetrachloroethane
tetrachloroethene
tetrahydrofuran
toluene
1,2,3-trichlorobenzene
1,2,4-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethene
1,2,3-trichloropropane
1,1,2-trichlorotrifluorethane (Freon® 113)
1,2,4-trimethylbenzene
1,3,5-trimethylbenzene
m-xylene
o-xylene
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
ethyl acetate
isopropyl acetate
n-propyl acetate
n-butyl 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
butyl acetate
ethyl acetate
isopropyl acetate
methyl acetate
propyl acetate
vinyl 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
30042: 502.2 Calib. Mix #1
30043: 502.2 Calib. Mix #2
30044: 502.2 Calib. Mix #3
30045: 502.2 Calib. Mix #4
30046: 502.2 Calib. Mix #5
30047: 502.2 Calib. Mix #6
30067: 4-bromofluorobenzene
30240: 8260A Surrogate Mix
30241: 8260A Internal Standard Mix
30075: 8240/8260 System Performance Check Mix

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30242	30242-500

Method 8260

8260 Internal Standard Mix

chlorobenzene-d5
1,4-dichlorobenzene-d4
1,4-difluorobenzene
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
dibromofluoromethane
toluene-d8

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
chlorobenzene
chloromethane
1,1-dichloroethane
1,1,2,2-tetrachloroethane

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
1,1-dichloroethene
1,2-dichloropropane
ethylbenzene
toluene
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
30042: 502.2 Calib. Mix #1
30043: 502.2 Calib. Mix #2
30044: 502.2 Calib. Mix #3
30045: 502.2 Calib. Mix #4
30046: 502.2 Calib. Mix #5
30047: 502.2 Calib. Mix #6
30067: 4-bromofluorobenzene
30073: 8260 Surrogate Mix
30074: 8260 Internal Standard Mix
30075: 8240/8260 System Performance Check Mix

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30076	30076-500

PVOC, GRO, & BTEX

PVOC Mix (California)

benzene
ethylbenzene
methyl *tert*-butyl ether
toluene
m-xylene
o-xylene
p-xylene

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
ethylbenzene
methyl *tert*-butyl ether
naphthalene
toluene
1,2,4-trimethylbenzene
1,3,5-trimethylbenzene
m-xylene
o-xylene
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
ethylbenzene
3-methylpentane
naphthalene
1,2,4-trimethylbenzene
2,2,4-trimethylpentane (isooctane)
toluene
m-xylene
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	*m*-xylene	1000
2-methylpentane	1500	*o*-xylene	1000
toluene	1500	*p*-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	*m*-xylene	1,000
2-methylpentane	1,500	*o*-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
ethylbenzene
toluene
m-xylene
o-xylene
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
ethylbenzene
isopropyl benzene
methyl *tert*-butyl ether
naphthalene
toluene
m-xylene
o-xylene
p-xylene

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
ethylbenzene
toluene
m-xylene
o-xylene
p-xylene

In nitrogen, 104 liters @ 1800psig

1ppm	100ppb
34414 (ea.)	34428 (ea.)

Method 624

624 Internal Standard Mix

bromochloromethane
2-bromo-1-chloropropane
1,4-dichlorobutane

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
fluorobenzene
pentafluorobenzene

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
chloroethane
chloromethane
trichlorofluoromethane
vinyl chloride

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
carbon tetrachloride
chlorobenzene
2-chloroethyl vinyl ether
dibromochloromethane
1,1-dichloroethane
1,1-dichloroethene
1,2-dichloropropane
methylene chloride
tetrachloroethene
1,1,2-trichloroethane
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
bromoform
chloroform
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
1,2-dichloroethane
trans-1,2-dichloroethene
cis-1,3-dichloropropene
trans-1,3-dichloropropene
ethylbenzene
1,1,2,2-tetrachloroethane
toluene
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
30021: 624 Calib. Mix #2
30022: 624 Calib. Mix #3
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
30021: 624 Calib. Mix #2
30022: 624 Calib. Mix #3
30023: 624 Int. Standard Mix
30243: 624 Surrogate 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
30021: 624 Calib. Mix #2
30022: 624 Calib. Mix #3

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30061	30061-500

602 Purgeable Aromatics Calibration Mix

benzene
chlorobenzene
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
ethylbenzene
toluene

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
bromobenzene
bromochloromethane
bromodichloromethane
bromoform
n-butylbenzene
sec-butylbenzene
tert-butylbenzene
carbon tetrachloride
chlorobenzene
chloroform
2-chlorotoluene
4-chlorotoluene
dibromochloromethane
1,2-dibromo-3-chloropropane
1,2-dibromoethane
dibromomethane
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
1,3-dichloropropane
2,2-dichloropropane
1,1-dichloropropene
cis-1,3-dichloropropene
trans-1,3-dichloropropene
ethylbenzene
hexachlorobutadiene
isopropylbenzene
p-isopropyltoluene
methylene chloride
naphthalene
n-propylbenzene
styrene
1,1,1,2-tetrachloroethane
1,1,2,2-tetrachloroethene
tetrachloroethene
toluene
1,2,3-trichlorobenzene
1,2,4-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethene
1,2,3-trichloropropane
1,2,4-trimethylbenzene
1,3,5-trimethylbenzene
m-xylene
o-xylene
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

04.2, 04.1, & 3/90 SOW

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)

30042: 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

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.

3/90 SOW

CLP VOA CAL2000 MegaMix™ Mixture (29 components)

Note: This product is provided as a two-ampul set:

benzene
bromodichloromethane
bromoform
carbon disulfide
carbon tetrachloride
chlorobenzene
chloroform
dibromochloromethane
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
ethylbenzene
methylene chloride
styrene
1,1,2,2-tetrachloroethane
tetrachloroethene
toluene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethene
m-xylene
o-xylene
p-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
chloroethane
chloromethane
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
carbon disulfide
ethylbenzene
toluene
vinyl acetate
o-xylene
p-xylene

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
chlorobenzene
chloroform
1,1-dichloroethane
1,1-dichloroethene
1,2-dichloropropane
methylene chloride
1,1,2-trichloroethane
trichloroethene
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
bromoform
dibromochloromethane
1,2-dichloroethane
cis-1,2-dichloroethene
trans-1,2-dichloroethene
cis-1,3-dichloropropene
trans-1,3-dichloropropene
styrene
1,1,2,2-tetrachloroethene
tetrachloroethene
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
30002: VOA Screening Mix #2
30003: VOA Tuning Comp.
30004: VOA Surr. Spike Mix
30005: VOA Matrix Spike Mix
30006: VOA Calib. Mix #1 (ketones)
30010: VOA Calib. Mix #5 (gases)
30011: VOA Int'l Standard Mix
30438: CLP VOA CAL2000 MegaMix™

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30440	30440-500

Volatile Organics Kit

30001: VOA Screening Mix #1
30002: VOA Screening Mix#2
30003: VOA Tuning Comp.
30004: VOA Surr. Spike Mix
30005: VOA Matrix Spike Mix
30006: VOA Calib. Mix #1 (ketones)
30007: VOA Calib. Mix #2
30008: VOA Calib. Mix #3
30009: VOA Calib. Mix #4
30010: VOA Calib. Mix #5 (gases)
30011: VOA Internal 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
bromodichloromethane
bromoform
carbon disulfide
carbon tetrachloride
chlorobenzene
chloroform
1,2-dibromo-3-chloropropane
cyclohexane
dibromochloromethane
1,2-dibromoethane
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
ethylbenzene
isopropylbenzene
methyl acetate
methylcyclohexane
methylene chloride
styrene
methyl *tert*-butyl ether (MTBE)
1,1,2,2-tetrachloroethane
tetrachloroethene
toluene
1,2,4-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethylene
1,1,2-trichlorotrifluoroethane (Freon® 113)
m-xylene
o-xylene
p-xylene

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
chloroethane
chloromethane
dichlorodifluoromethane
trichlorofluoromethane
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
chloroethane
chloromethane
dichlorodifluoromethane
trichlorofluoromethane
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
bromochloromethane
bromodichloromethane
bromoform
carbon disulfide
carbon tetrachloride
chlorobenzene
chloroform
cyclohexane
dibromochloromethane (chlorodibromomethane)
1,2-dibromo-3-chloropropane
1,2-dibromoethane (EDB)
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethylene
cis-1,2-dichloroethylene
trans-1,2-dichloroethylene
1,2-dichloropropane
cis-1,3-dichloropropene(Z)
trans-1,3-dichloropropene (E)
ethylbenzene
isopropylbenzene (cumene)
methyl acetate
methylcyclohexane
methyl *tert*-butyl ether (MTBE)
methylene chloride (dichloromethane)
styrene
1,1,2,2-tetrachloroethane
tetrachloroethylene
toluene
1,2,3-trichlorobenzene
1,2,4-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethylene
1,1,2-trichlorotrifluoroethane (Freon® 113)
m-xylene
o-xylene
p-xylene

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
bromoform-d
chloroethane-d5
chloroform-d
1,2-dichlorobenzene-d4
1,2-dichloroethane-d4
1,1-dichloroethene-d2
1,2-dichloropropane-d6
trans-1,3-dichloropropene-d4
1,1,2,2-tetrachloroethane-d2
toluene-d8
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, semivolaitle, 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.

Reach for Restek

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Lit. Cat. #59887A

Technical Guide

A Guide to the Analysis of Chiral Compounds by GC

Inside:

RESTEK CORPORATION

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

Index:

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 Laboratory 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 assymetric 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 importantly, 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 (l) or (-)}.

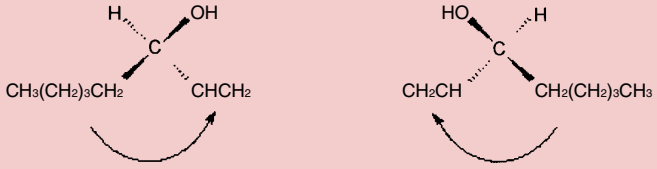
Enantiomers can be denoted by the specific configuration around the chiral center. Groups on the carbon center are assigned a "priority" based on atomic number of the first bonded atom (Cahn-Ingold-Prelog rules). The group with the highest atomic number is rated first. If priority cannot be established 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 assymmetric 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 assymmetric carbon center. The mirror images are not superimposable and so they are enantiomers.

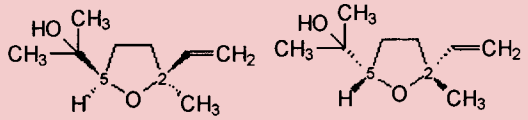
Enantiomers can be distinguished by configuration. Following groups from high to low priority in the clockwise direction is denoted R, and S for the counterclockwise direction.



S configuration **R configuration**

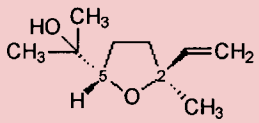
Figure 1B

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

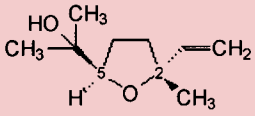


2S, 5R
(+)-cis-linalool oxide

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 assymetric 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.

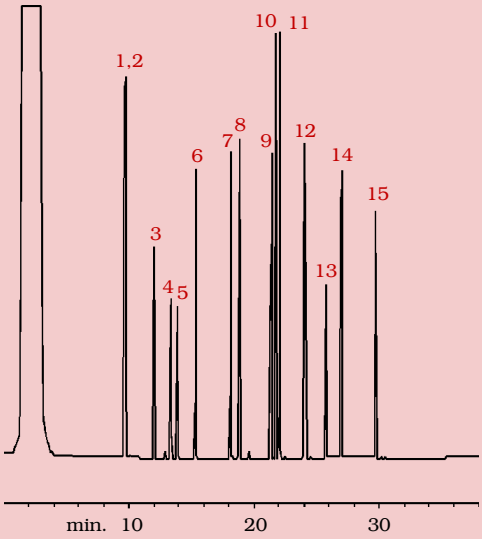
The permethylated derivative of beta-cyclodextrin in cyanopropyl-dimethylpolysiloxane liquid stationary phase is commonly used for such stereochemical separations, but it exhibits limited applications. Beta-cyclodextrins derivatized with alkyl substituents can enhance the enantiomeric resolution of various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

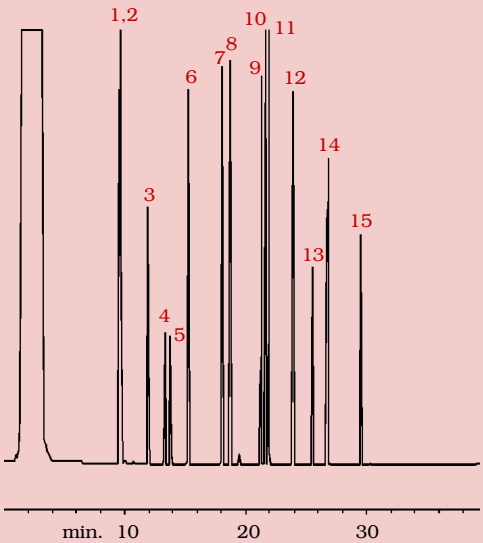
Figure 2
Restek's chiral columns demonstrate exceptional lifetime and stability for more than 250 injections without loss of resolution.

A: 1st injection



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

B: 250th injection

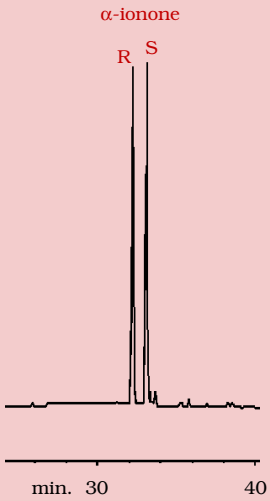


30m, 0.32mm ID, 0.25µm Rt-βDEXsa (cat.# 13108)
Oven temp.: 40°C (hold 1 min.) to 230°C @ 2°C/min.;
Carrier gas: hydrogen; 80cm/sec. set @ 40°C; **Detector:** FID set @ 220°C

RESTEK'S CHIRAL COLUMNS OFFER UNIQUE SELECTIVITY

Figure 3
The Rt-βDEXsm column provides the best chiral separation of isoborneol and α-terpineol.

Figure 4
The Rt-βDEXsm column offers complete resolution of α-ionone.



30m, 0.32mm ID, 0.25µm Rt-βDEXsm (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 posesses a specific combination of alkyl substituents on the derivatized β-cyclodextrins. These unique combinations provide a wide range of utilitization for each type of chiral column. Table I, on page 8, indicates that certain columns provide better resolution of specific compounds.

Rt-βDEXsm

Of the chiral columns evaluated, only the Rt-βDEXsm 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 mephobarbital (**Figures 3 and 4**).

Rt-βDEXse

The Rt-βDEXse is similar in performance to the Rt-βDEXsm, 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-βDEXse column resolves optical isomers of ethyl-2-methylbutyrate, styrene oxide, and camphor with some overlap.

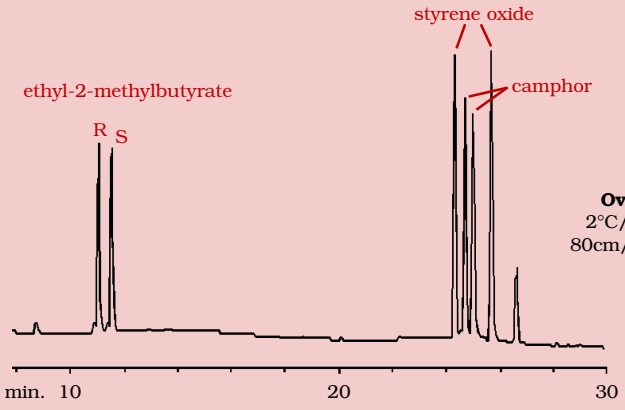
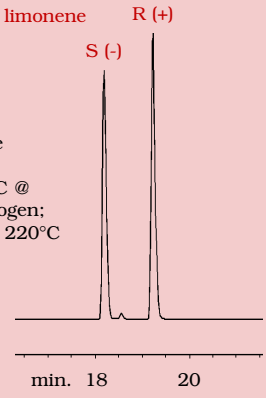


Figure 6
The Rt-βDEXse column resolves limonene enantiomers.



30m, 0.32mm ID, 0.25µm Rt-βDEXse
(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-βDEXsm and Rt-βDEXse

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-βDEXsm separates the linalool oxides, but it does not resolve linalyl acetate. Conversely, Rt-βDEXse 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**).

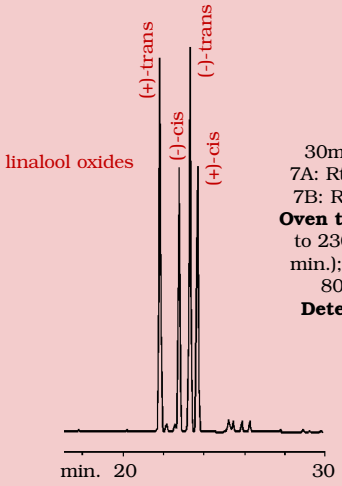
Rt-βDEXsp

Rt-βDEXsp is a specialized column that best resolves menthol. It would be useful in addition to the Rt-βDEXsm or Rt-βDEXse column for analyzing complete profiles of mint oils (**Figure 8**).

Rt-βDEXsa

The Rt-βDEXsa 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-βDEXsm column.



30m, 0.32mm ID, 0.25µm
7A: Rt-βDEXsm (cat.# 13104)
7B: Rt-βDEXse (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-βDEXse column.

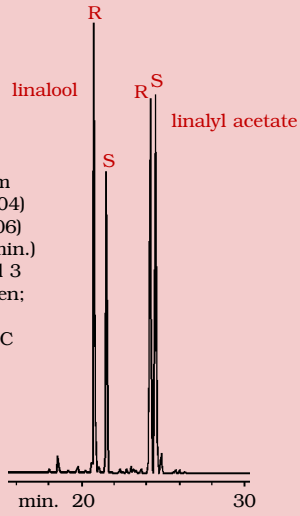


Figure 8
Menthol enantiomers are best resolved on the Rt-βDEXsp column.

30m, 0.32mm ID, 0.25µm Rt-βDEXsp (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

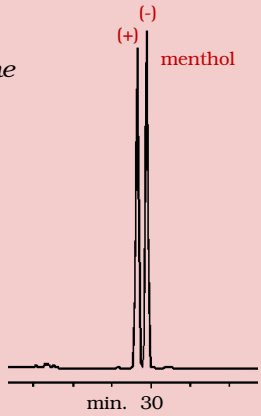


Figure 9A
1-octen-3-ol and carvone enantiomers are best resolved on an Rt-βDEXsa column.

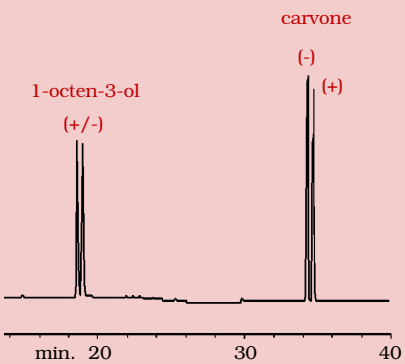
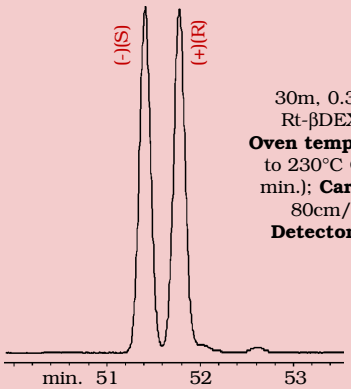


Figure 9B
An Rt-βDEXsa column provides baseline chiral resolution for β-citronellol.



30m, 0.32mm ID, 0.25µm
Rt-βDEXsa (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-βDEXsa column separates racemic cis and trans rose oxides.

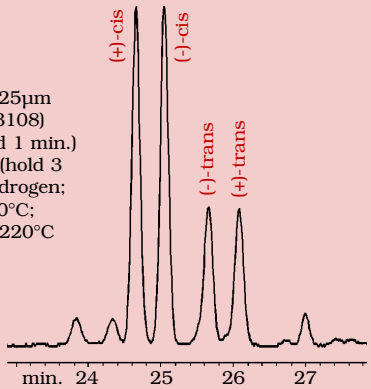


Figure 10
All of the irone isomers are resolved without overlapping of the enantiomeric pairs on the Rt-βDEXcst column.

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

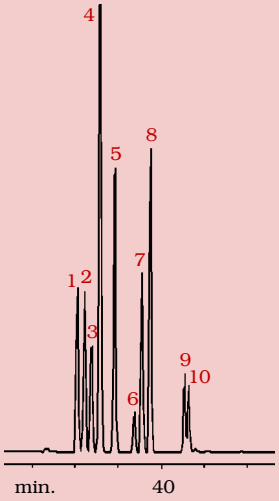
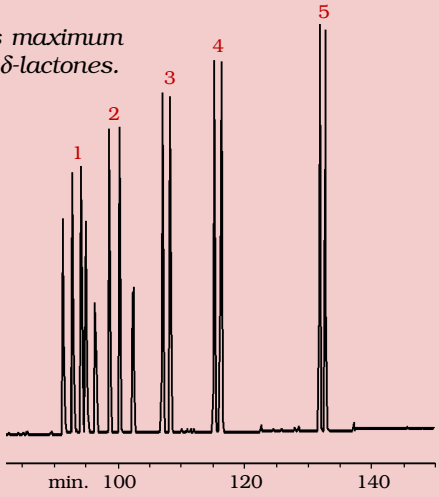


Figure 11
The Rt-βDEXcst column provides maximum resolution of the γ-lactones and δ-lactones.

A. *γ-lactones on the Rt-βDEXcst column*

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

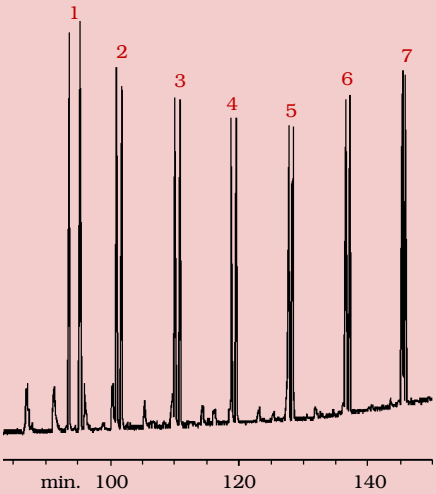
30m, 0.32mm ID, 0.25µm
Rt-βDEXcst (cat.# 13102)
Oven temp.: 60°C (hold 1 min.) to 200°C @ 1°C/min.; **Carrier gas:** hydrogen; 40cm/sec. set @ 60°C;
Detector: FID set @ 220°C



B. *δ-lactones on the Rt-βDEXcst column*

1. (+/-)δ-pentalactones
2. (+/-)δ-hexalactones
3. (+/-)δ-heptalactones
4. (+/-)δ-octalactones
5. (+/-)δ-nonalactones
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



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.

Visit Restek on-line at www.restekcorp.com, or call 800-356-1688, ext. 4, for technical assistance.

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 denoted 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-βDEXm (permethylated cyclodextrin) column. **Table I** shows the degree of enantiomeric separation by resolution factor for all twenty-five components on each column. The column 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

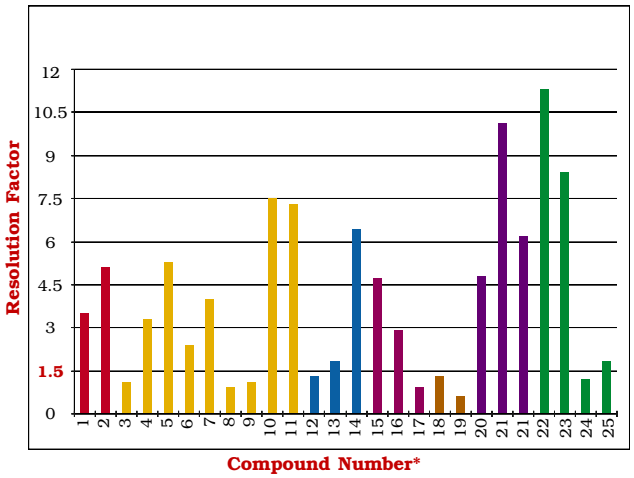
Resolution of common chiral compounds on Restek's cyclodextrin columns.

Compounds		Formula	m.w.	Column Resolution Factors					
				Rt-βDEX**sm**	Rt-βDEX**se**	Rt-βDEX**sp**	Rt-βDEX**sa**	Rt-βDEX**cst**	Rt-βDEX**m**
Terpenes	1. α-pinene	$C_{10}H_{16}$	136	3.50	0.80	*ns*	*ns*	0.90*b*	3.30
	2. limonene	$C_{10}H_{16}$	136	5.10	7.30	3.20	*ns*	2.70*b*	1.40
Alcohols	3. 1-octen-3-ol	$C_8H_{16}O$	128	1.10	*ns*	0.50	2.30	0.60*b*	0.50*t*
	4. linalool	$C_{10}H_{18}O$	154	3.30	6.00	4.30*b*	3.70	2.60*t*	1.00
	5. α-terpineol	$C_{10}H_{18}O$	154	5.30	5.50	2.20	4.00	4.30*t*	1.70
	6. terpinen-4-ol	$C_{10}H_{18}O$	154	2.40	2.20	*ns*	*ns*	1.20*t*	1.80*t*
	7. isoborneol	$C_{10}H_{18}O$	154	4.00	3.30*t*	*ns*	*ns*	1.90*t*	2.00
	8. β-citronellol	$C_{10}H_{20}O$	156	0.90	0.80	*ns*	1.00*t*	*ns*	*ns*
	9. menthol	$C_{10}H_{20}O$	156	1.10	1.10	2.20	*ns*	1.10	1.60*t*
	10. 2,3-butanediol	$C_4H_{10}O_2$	90	7.50	8.10	2.20	4.60	4.00	2.60
	11. 1-phenylethanol	$C_8H_{10}O$	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	2.10*t*	1.30*b*	4.30	2.50*t*	*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	$C_9H_{16}O_2$	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	$C_7H_{14}O_2$	130	1.30	3.50	1.10*b*	*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_8H_8O	120	4.80	10.20	6.50	1.00	1.40*t*	2.50*t*
	21. trans-linalooloxides	$C_{10}H_{18}O_2$	170	10.10	2.40	*ns*	1.50	4.30*b*	6.80*
	21. cis-linalooloxides			6.20	3.60	*ns*	1.00	3.50*b*	4.20*
Drugs	22. hexobarbital	$C_{12}H_{16}O_3N_2$	236	11.30	4.70	0.90	*ns*	8.90	6.30
	23. mephobarbital	$C_{13}H_{14}O_3N_2$	246	8.40	3.80	0.60	*ns*	6.20	6.10
	24. fenfluramine	$C_{12}H_{16}F_3N$	231	1.80	2.90*t*	*ad*	*ad*	*ns*	*ad*
	25. fenfluramine TFAA der.	$C_{14}H_{15}F_6NO$	327	1.20	*ns*	*ns*	2.40	3.40	*ns*

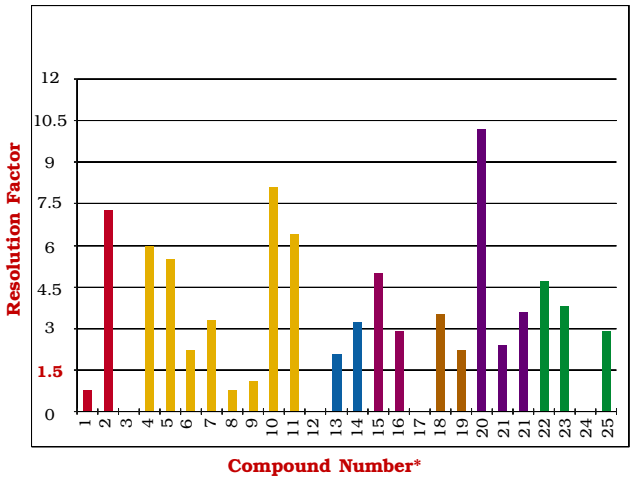
ns = no separation of enantiomers
t = peak tailing
b = peak broadening
ad = adsorption of nonderivatized drug compound
* = cis and trans linalool oxides analyzed @ 9.0 psi head pressure

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

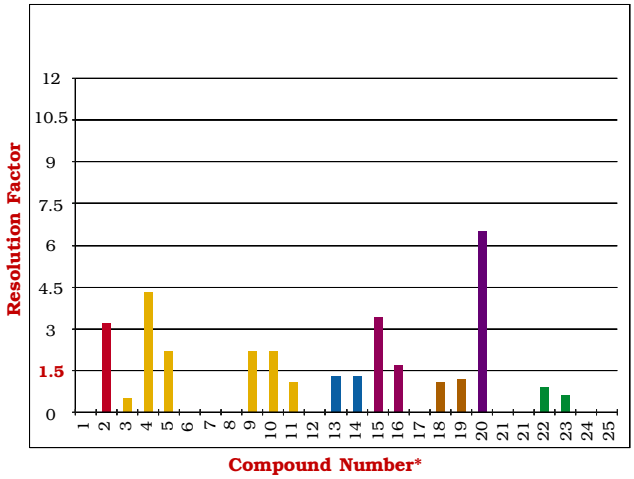
Rt-βDEXsm



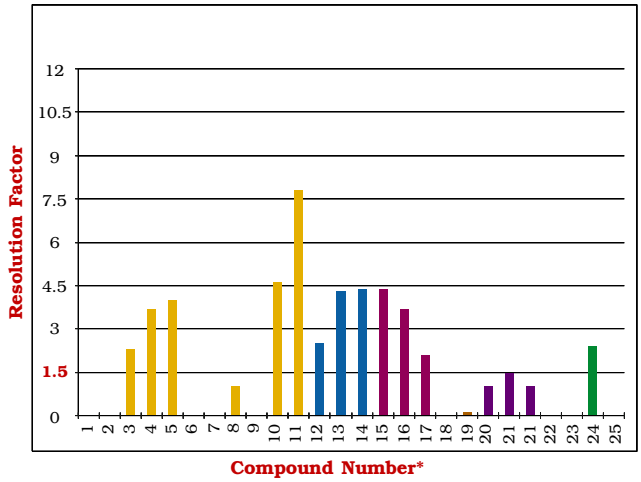
Rt-βDEXse



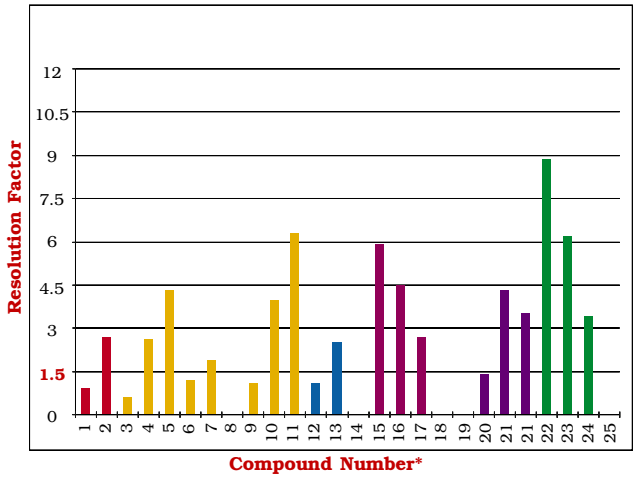
Rt-βDEXsp



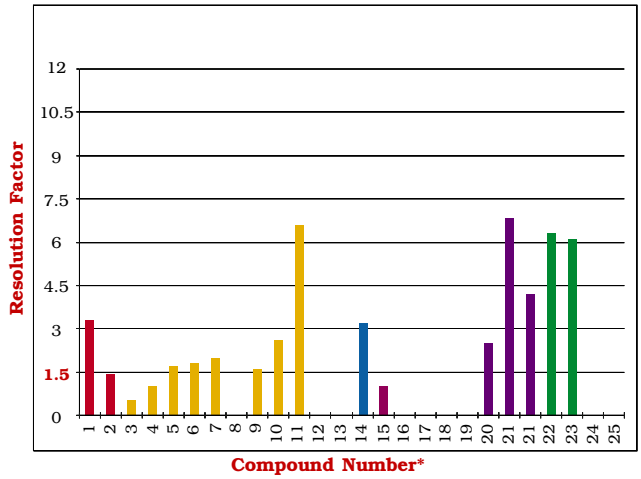
Rt-βDEXsa



Rt-βDEXcst



Rt-βDEXm



**Refer to Table I for compound identification.*

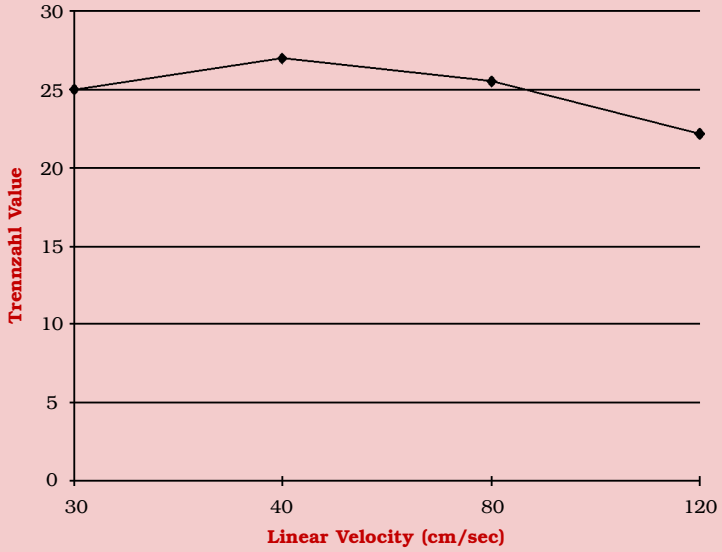
OPTIMIZATION OF CHIRAL SEPARATIONS

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)

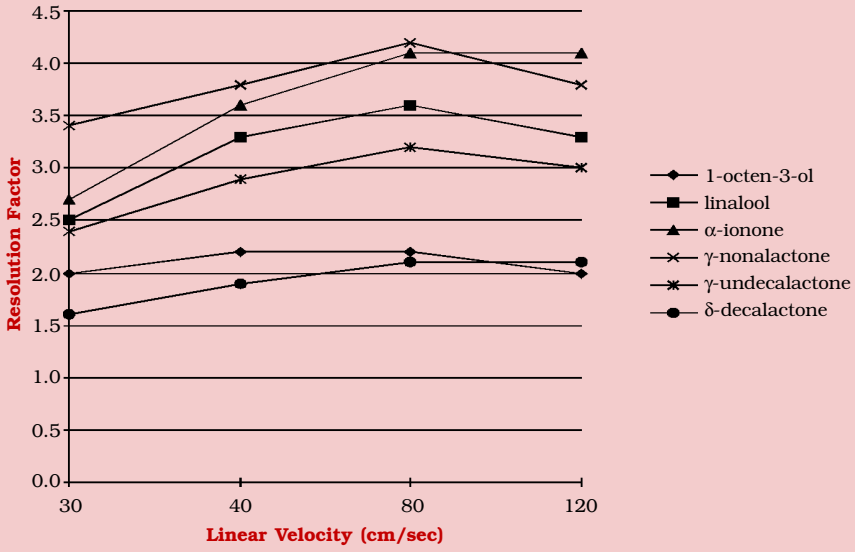
The resolution between the enantiomeric pairs can be improved by increasing the linear velocity. This is especially important if the resolution factor is below two for optical isomers (see Table I). Trennzahl values are measurements of column separation efficiency, which are often optimum at a linear 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-βDEXsa 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.



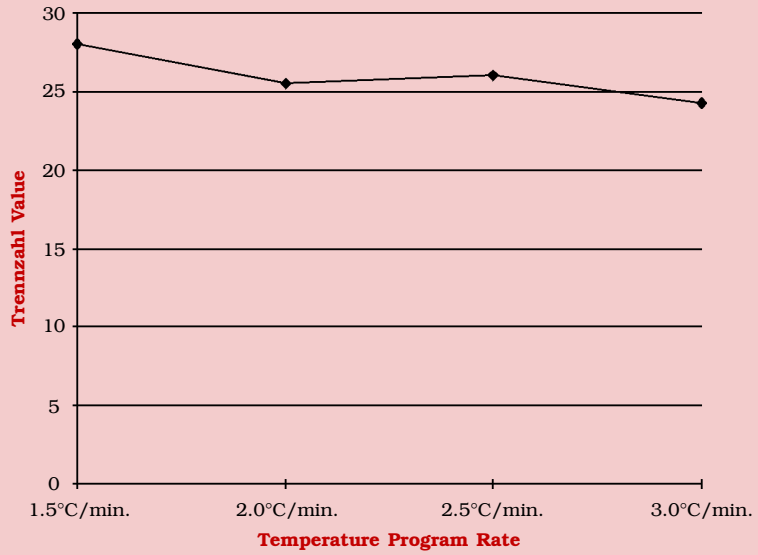
Rt-βDEXsa: 40°C to 200°C @ 2°C/min. (hold 1 min.). Hydrogen carrier gas.

Figure 12B
Faster linear velocities provide maximum resolution of chiral pairs.



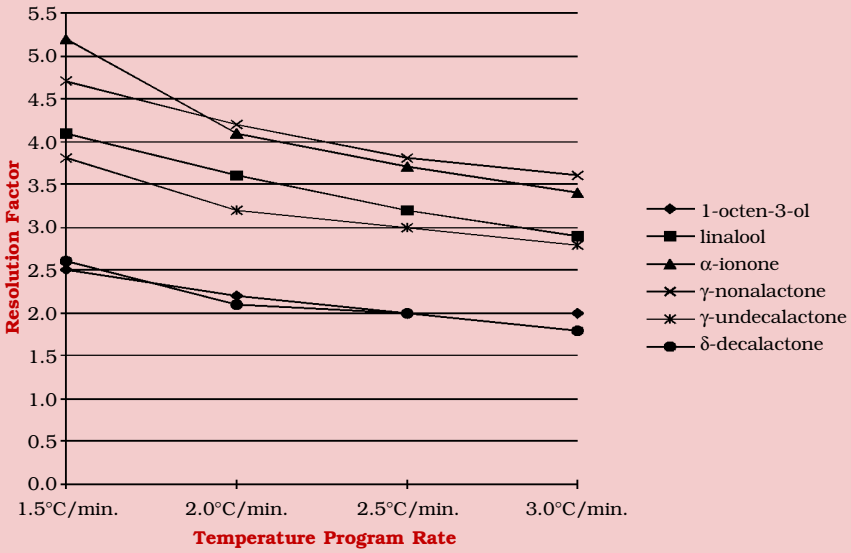
Rt-βDEXsa: 40°C (80°C for lactones) to 200°C @ 2°C/min. (hold 1 min.). Hydrogen carrier gas.

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.

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.

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.**

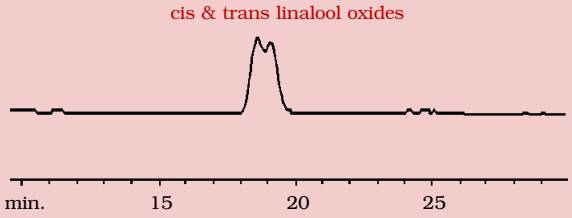
RESTEK CORPORATION

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.

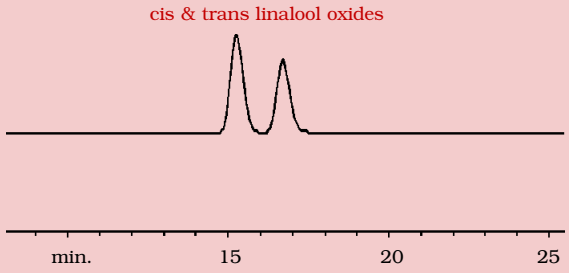
Higher initial temperatures do not always completely eliminate peak broadening for some components. However, the improvement in solvent peak shape indicates a physical transition of the cyclodextrin macromolecules from a crystalline structure to a liquid at this higher temperature.

Figure 14A
Linalool oxides exhibit extreme peak broadening and poor resolution on the Rt-βDEXcst column with an initial oven temperature of 40°C.



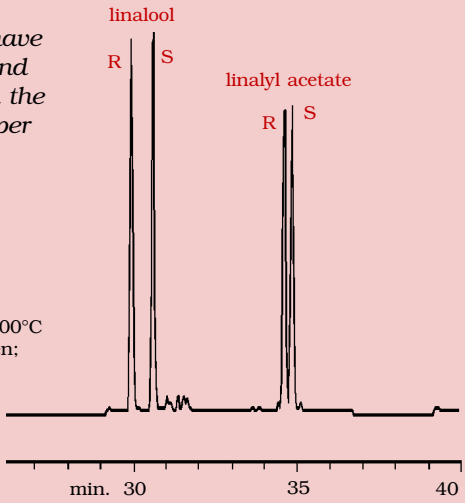
30m, 0.32mm ID, 0.25µm Rt-βDEXcst (cat.# 13102)
Oven temp.: 40°C (hold 1 min.) to 200°C @ 2°C/min.; **Carrier gas:** hydrogen; 80cm/sec. set @ 40°C;
Detector: FID set @ 220°C

Figure 14B
Linalool oxides exhibit improved peak shape and resolution on the Rt-βDEXcst column when initial oven temperature is increased to 70°C.



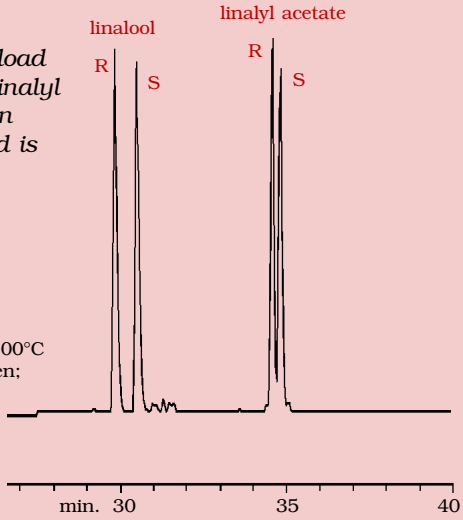
30m, 0.32mm ID, 0.25µm Rt-βDEXcst (cat.# 13102)
Oven temp.: 70°C (hold 1 min.) to 200°C @ 2°C/min.; **Carrier gas:** hydrogen; 80cm/sec. set @ 40°C;
Detector: FID set @ 220°C

Figure 15A
Linalool and linalyl acetate have symmetrical peak shapes and excellent chiral separation on the Rt-βDEXse column at 25ng per component on-column.



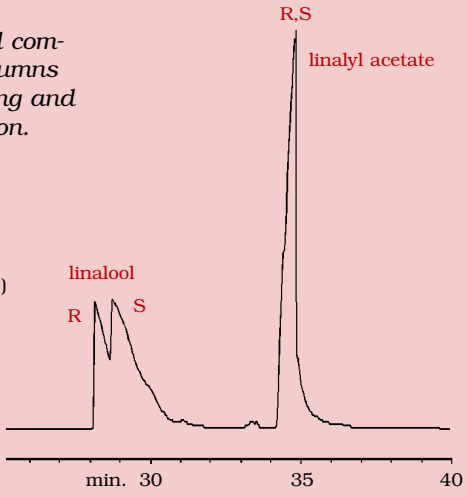
30m, 0.32mm ID, 0.25µm
Rt-βDEXse (cat.# 13106)
Oven temp.: 40°C (hold 1 min.) to 200°C @ 2°C/min.; **Carrier gas:** hydrogen; 80cm/sec. set @ 40°C;
Detector: FID set @ 220°C

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 to160ng.



30m, 0.32mm ID, 0.25µm
Rt-βDEXse (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, 0.25µm
Rt-βDEXse (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-βDEXse. 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 5µg 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.

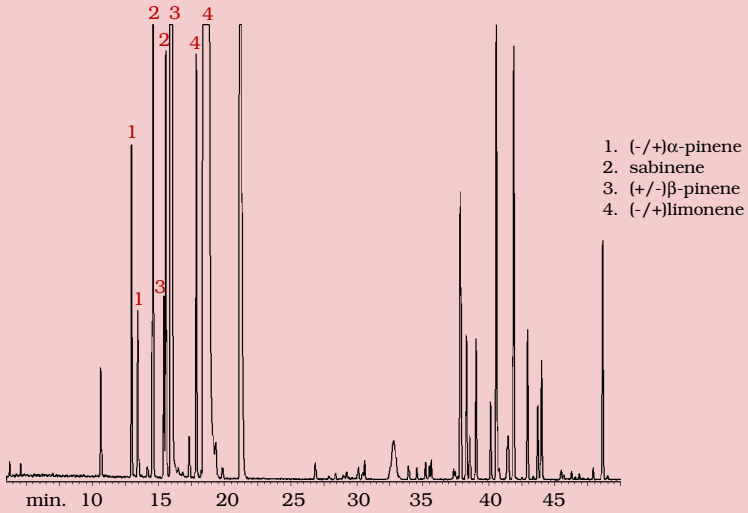
Since most chiral compounds naturally exist as one predominant isomer, resolution is more challenging, especially for components in higher concentrations. For primary constituents in essential oils, select a chiral column that provides a resolution factor value greater than two to overcome possible loss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

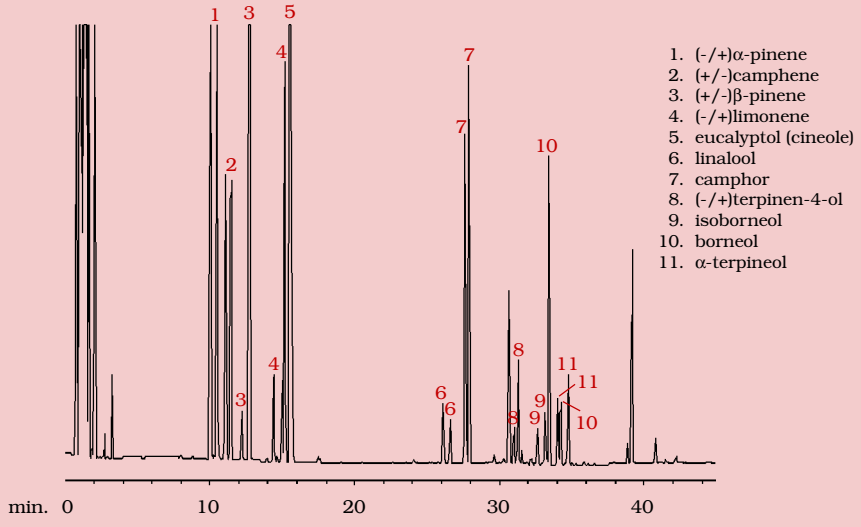
The Rt-βDEXsm column provides chiral resolution of the primary terpenes in Artificial Lemon oil.



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

Figure 17

The Rt-βDEXsm, 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.



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

Lemon Oil

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

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-βDEXsm column. The common permethylated β-cyclodextrin column cannot completely resolve the optical isomers of limonene, linalool, and camphor (**Figure 17**).

Visit Restek on-line at www.restekcorp.com, or call 800-356-1688, ext. 4, for technical assistance.

Peppermint Oil

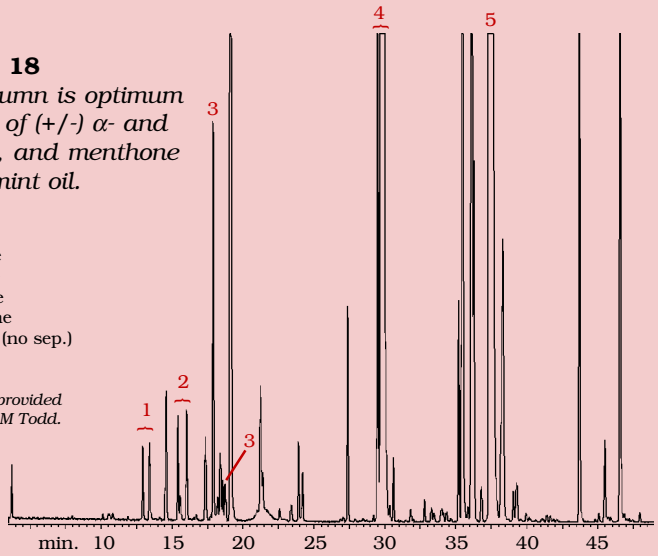
The Rt-βDEXsm column is optimum for the separation of (+/-) α- and β-pinene, limonene and menthone. Since menthone and menthol enantiomers are major constituents of peppermint oil, reducing the sample size to prevent overloading of these components and provide better enantiomeric resolution may be necessary.

An alternative solution is to use an Rt-βDEXsp 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-βDEXsm. The minimum temperatures of these phases differ by 20°C, which would minimize volatile terpene separation on the Rt-βDEXsm (**Figure 18**).

Figure 18
The Rt-βDEXsm 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.

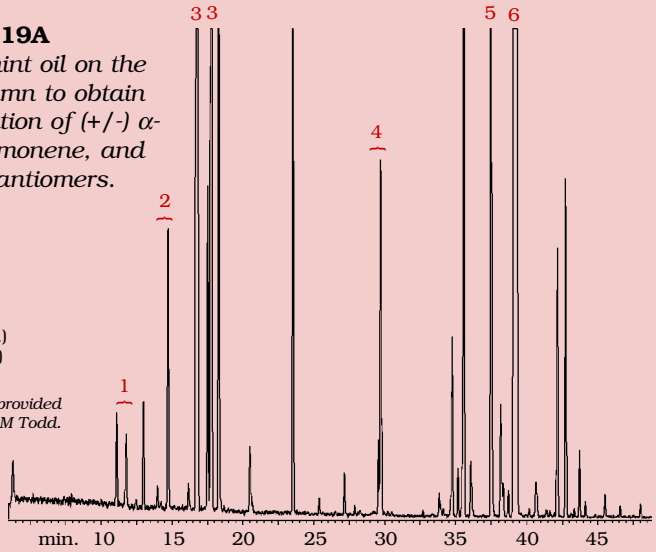


30m, 0.32mm ID, 0.25µm Rt-βDEXsm (cat.# 13104); **Oven temp.:** 40°C (hold 1 min.) to 200°C @ 2°C/min. (hold 3 min.); **Carrier gas:** helium; 60cm/sec.; **Detector:** MSD set @ 220°C

Figure 19A
Analyze spearmint oil on the Rt-βDEXsm 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.



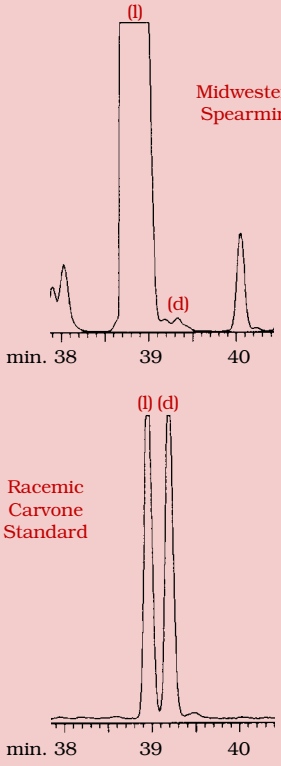
30m, 0.32mm ID, 0.25µm Rt-βDEXsm (cat.# 13104); **Oven temp.:** 40°C (hold 1 min.) to 200°C @ 2°C/min. (hold 3 min.); **Carrier gas:** helium; 60cm/sec.; **Detector:** MSD set @ 220°C

Spearmint Oil

The Rt-βDEXsm 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**.

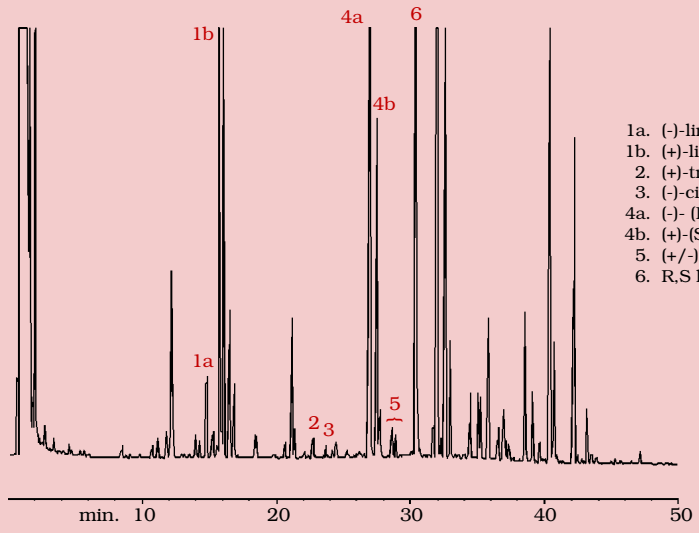
Although the optical isomers of carvone best separate on the Rt-βDEXsa column, α-pinene and limonene do not. For the separation of carvone, use a dual column system comprised of 30-meter Rt-βDEXsm and Rt-βDEXsa columns, since both have a minimum operating temperature of 40°C. **Figure 19B** compares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

Figure 19B
The optical isomers of carvone best separate on the Rt-βDEXsa column.



30m, 0.32mm ID, 0.25µm Rt-βDEXsa (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

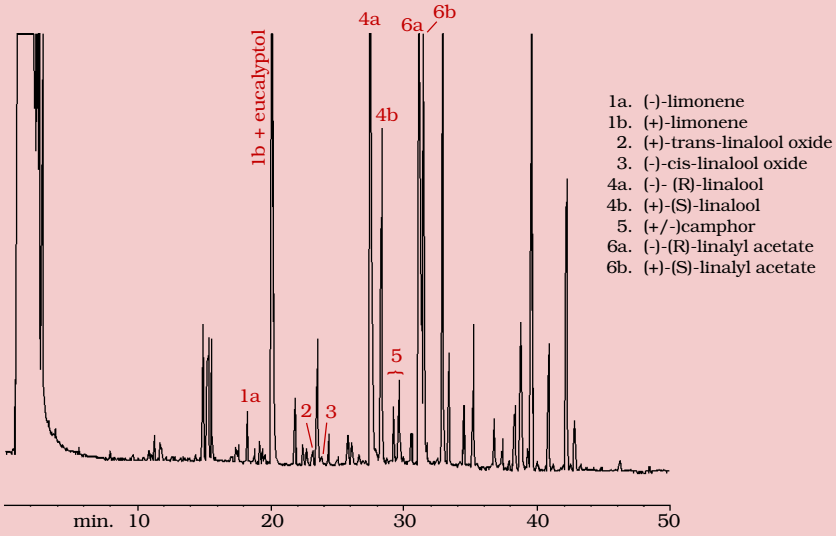
Figure 20A
Linalool and linalool oxides in lavender oils are stereochemically separated on the Rt-βDEXsm column.



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.

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.

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.

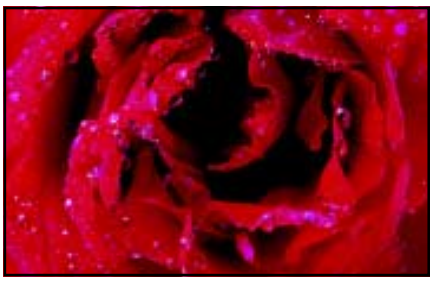
Visit Restek on-line at www.restekcorp.com, or call 800-356-1688, ext. 4, for technical assistance.

Geranium Oil

Chiral constituents in geranium oils include cis and trans rose oxides, linalool, and β-citronellol. The Rt-βDEXsa 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

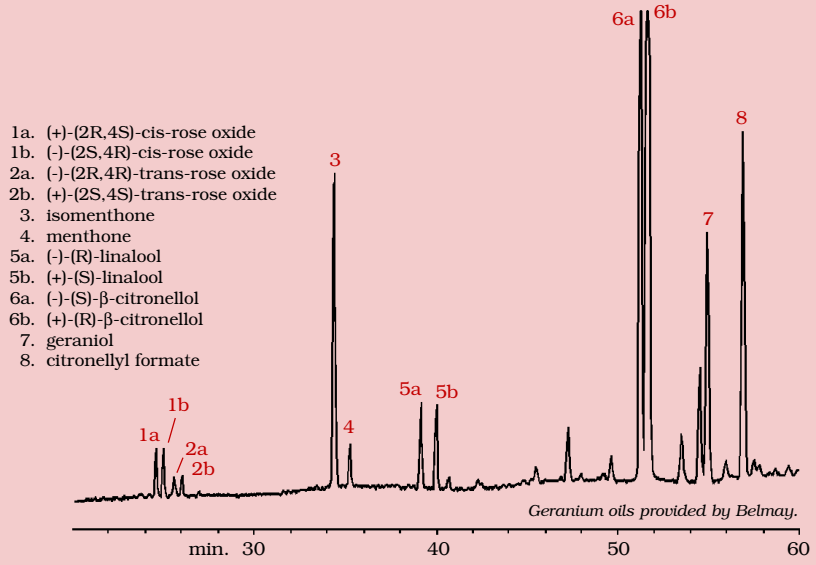
As with geranium oils, (-)-(2S,4R)-cis and (-)-(2R,4R)-trans rose oxides and (-)-(S)-β-citronellol are specific indicators of genuine rose oils.[5] Note the enantiomeric purity of these compounds in Rose Oil Maroc, as shown in **Figure 21B**.



Visit Restek on-line at www.restekcorp.com, or call 800-356-1688, ext. 4, for technical assistance.

Figure 21A

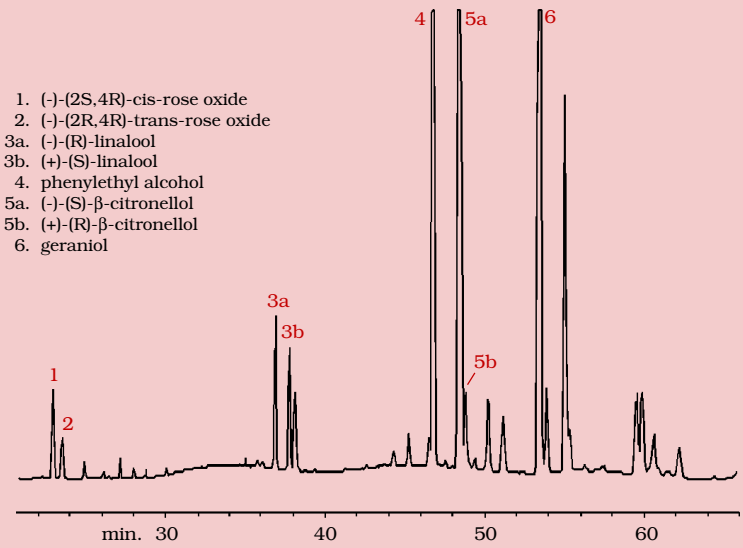
The Rt-βDEXsa column provides chiral resolution for cis and trans rose oxides, linalool, and β-citronellol in Chinese Geranium oil.



30m, 0.25mm ID, 0.25µm Rt-βDEXsa (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

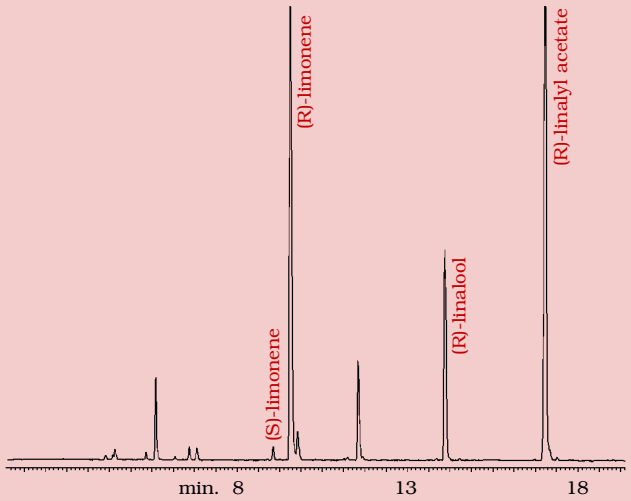
The Rt-βDEXsa column reveals authenticity of Rose oil Maroc.



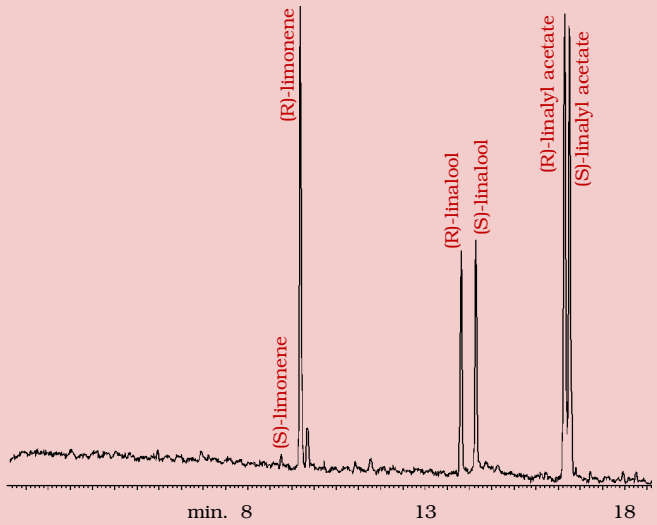
30m, 0.25mm ID, 0.25µm Rt-βDEXsa (cat.# 13109);
Oven temp.: 60°C to 110°C @ 1°C/min. (hold 30 min.);
Carrier gas: hydrogen; 40cm/sec. set @ 60°C; **Detector:** FID set @ 220°C

Figure 22
The Rt-βDEXse column can differentiate Bergamot extract from Bergamot flavor.

A: Bergamot Extract



B: Bergamot Flavor



30m, 0.32mm ID, 0.25µm Rt-βDEXse (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

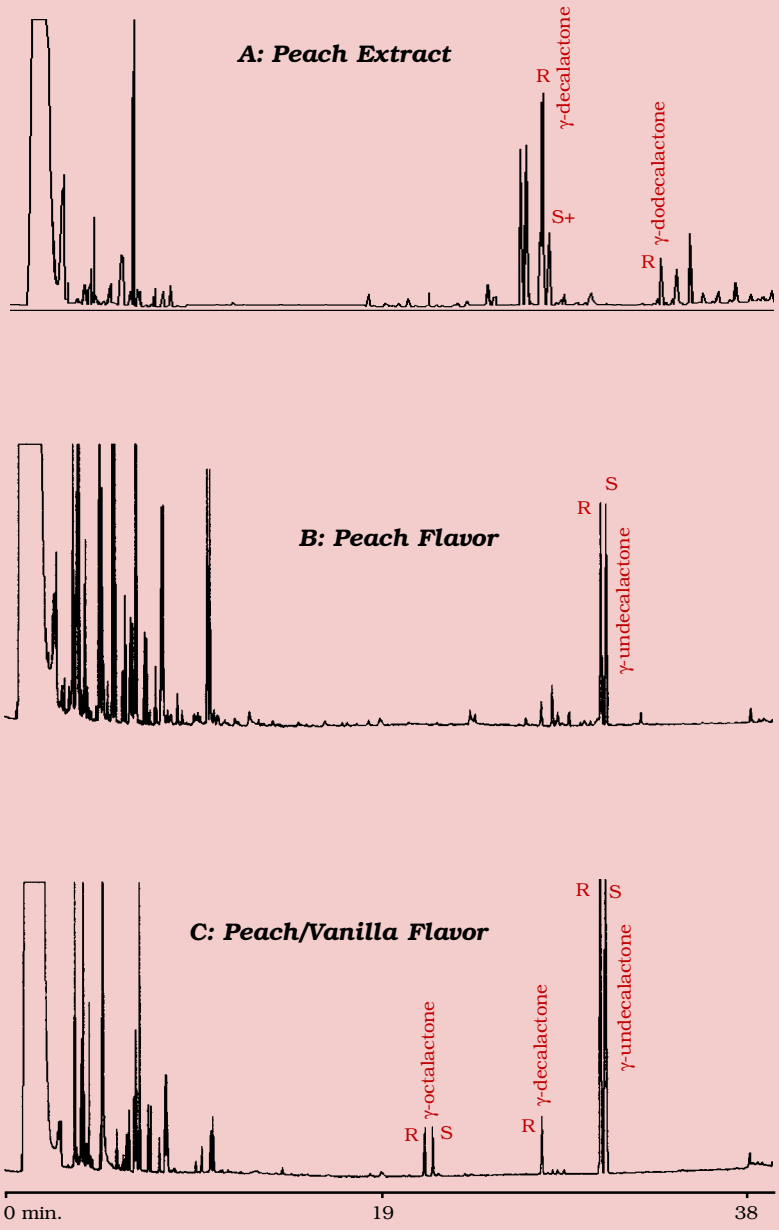
A genuine cold-pressed bergamot oil should contain only the (R)-isomers of linalool and linalyl acetate.[6] The enantiomeric purity of (R) limonene should also be considered.[7] Chromatogram A in **Figure 22** is a natural source of bergamot oil. Only the (R)-enantiomers of limonene, linalool and linalyl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

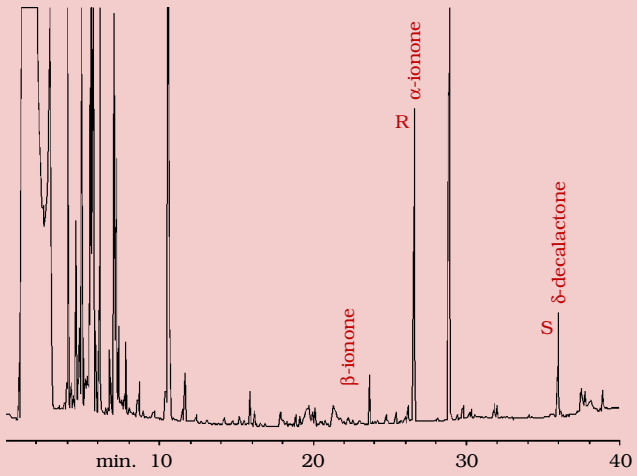
Figure 23
The γ-lactones are analyzed for the adulteration of peach flavor on the Rt-βDEXsa column.



30m, 0.32mm ID, 0.25µm Rt-βDEXsa (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.

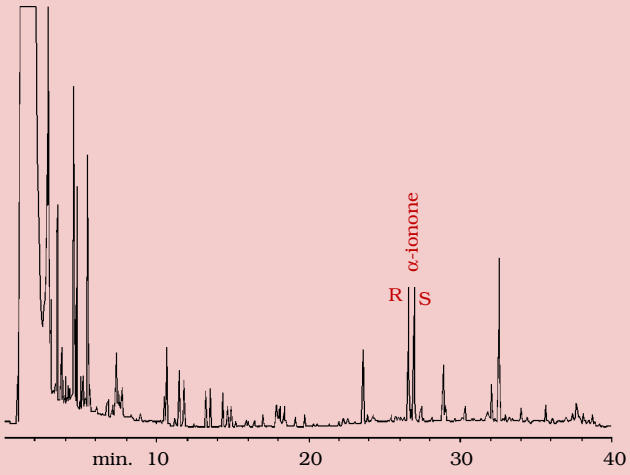
Figure 24
The Rt-βDEXsa column resolves isomers of α-ionone to determine raspberry authenticity.

A: Raspberry Extract



30m, 0.32mm ID, 0.25µm Rt-βDEXsa (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, 0.25µm Rt-βDEXsa (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-βDEXsa 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-βDEXcst 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-βDEXcst 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-βDEXcst column.

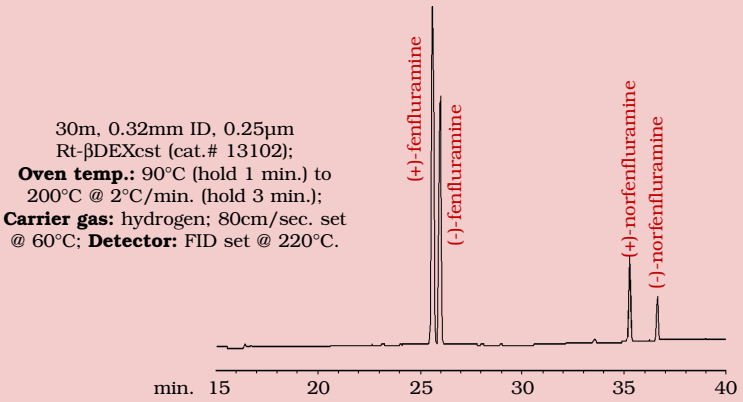


Figure 26

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

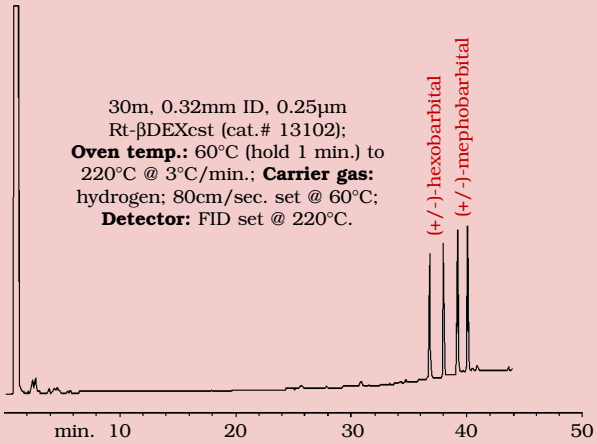
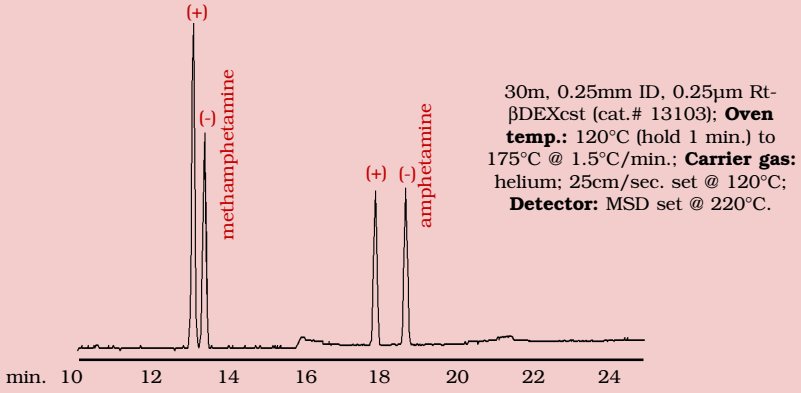
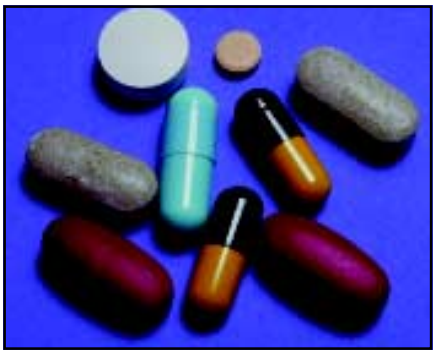


Figure 27

The Rt-βDEXcst 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-βDEXcst 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.

1. *John McMurray, Organic Chemistry, Brooks/Cole Pub., Monterey, CA, 1984.*
2. *C. Askari and A. Mosandl, Phytochem. Anal., Vol.2 p.211 (1991).*
3. *Uzi Ravid, Eli Putievsky, and Irena Katzir, "Chiral Analysis of Enantiomerically pure (R)-(-) Linalyl Acetate in some Lamiaceae, Myrtle, and petigrain Essential Oils,"* Flavour Fragrance J.*Vol.9, Pp. 275-276 (1994).*
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10. Physicians' Desk Reference, *46th ed., Pp. 2061-2062 (1992).*
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12. *W. Haefely, E. Kyburz, M. Gerecke, and H. Mohler. "Recent Advances in the molecular pharmacology of bezodiazepine receptors and in the structure-activity relationships of their agonists and antagonists."* Adv. Drug Res.*Vol. 14, Pp. 165-322 (1985).*
13. *J.T. Cody and R. Schwartzhoff, "Interpretation of Methamphetamine and Amphetamine Enantiomer Data"* J. Anal Toxicol. *Vol. 17, Pp. 321-326 (Oct. 1993).*

PRODUCT LIST

Rt-βDEXsm (30m, 0.25µm)				Rt-βDEXse (30m, 0.25µm)			
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-βDEXsa (30m, 0.25µm)				**Rt-βDEXsp (30m, 0.25µm)**			
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-βDEXcst (30m, 0.25µm)				**Rt-βDEXm (30m, 0.25µm)**			
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

Lit. Cat. #59889

RESTEK CORPORATION

Technical Guide

A Guide to Preparing and Analyzing Chlorinated Pesticides

Inside:

RESTEK

Table of Contents

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.*

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.

Removing water from the dichloromethane with sodium sulfate is critical before the extract is concentrated to final volume. Dichloromethane can hold approximately 11mL 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.

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

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 $^1/_3$ or $^1/_4$ 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.

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

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.

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.

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.

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-

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.

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 maintanence, 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.

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

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.

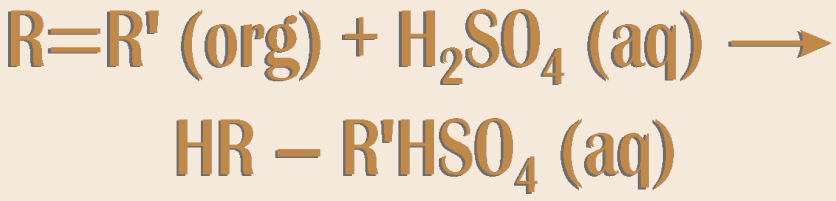
3. US EPA Contract Laboratory Program, *Statement of Work for Organic Analysis 0LM04.0*, Exhibit D Pesticides/Aroclors.

References not available from Restek.

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.

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



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 prepacked cartridges, which further increases performance reproducibility.

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

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

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.

Refer to page 19 for SPE tube ordering information.

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.

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.

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.

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.

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),

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 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.

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.

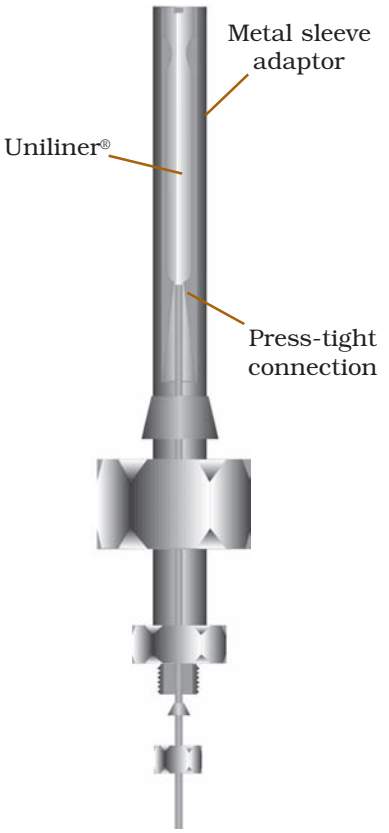
On-column injections are best suited for the analysis of water sample extracts, where analyte concentration levels are usually low and the amount of non-volatile material is relatively small. Both small and large volumes can be injected on-column, with the large-volume injections being even more sensitive to non-volatile residue. Conventional on-column injections are typically less than 1µL, and require the use of 0.53mm ID columns. Large-volume, on-column injections are typically 10µL to 100µL 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.

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.

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-

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 3µL to 4µL 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.

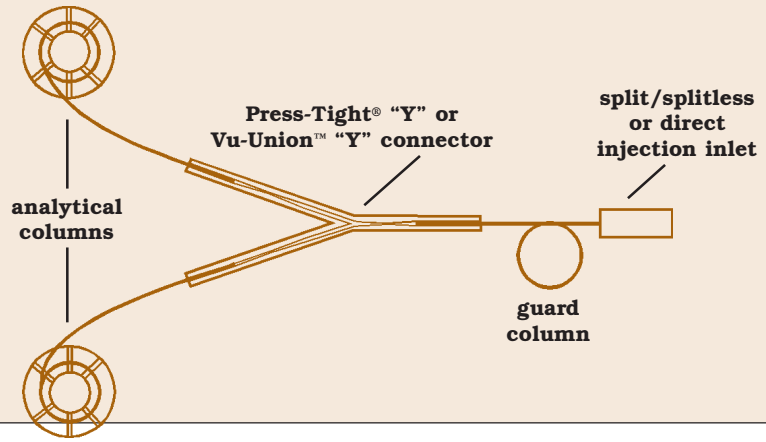
A third direct injection technique used for pesticide analysis involves large-volume injections into a cold injection port. The injected solvents and compounds are cold-trapped on the injection port liner walls. The injection port is heated to about the boiling point of the solvent, and the solvent is 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.

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



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

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®-

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).

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.

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.

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

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.

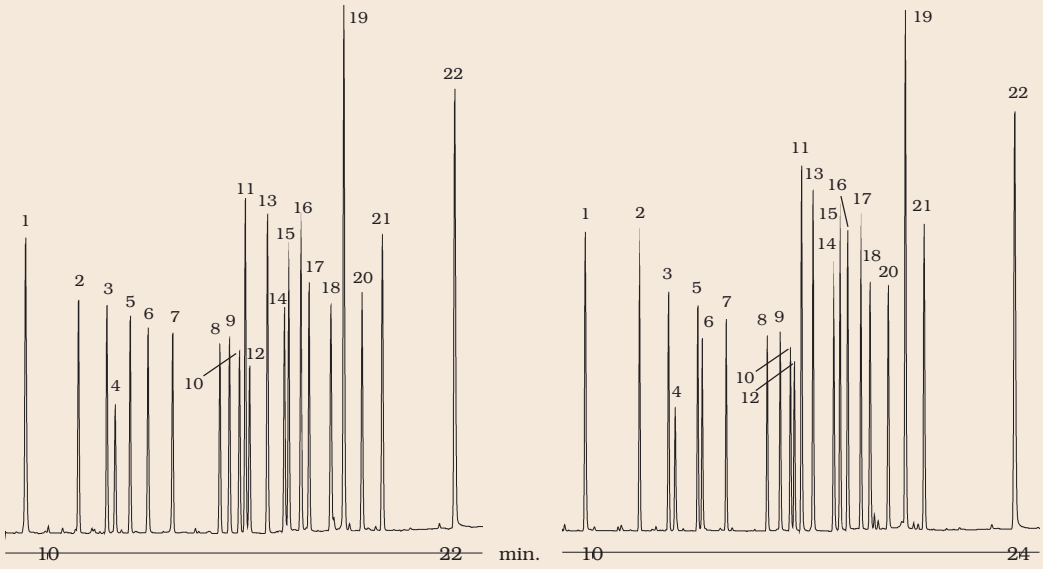
When problems occur, using proper troubleshooting and maintenance techniques can quickly reestablish system integrity. When faced with difficulties in your pesticide or PCB analysis, remember that the majority of problems occur during the sample preparation and cleanup step, or at the injection port 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. ensdosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl

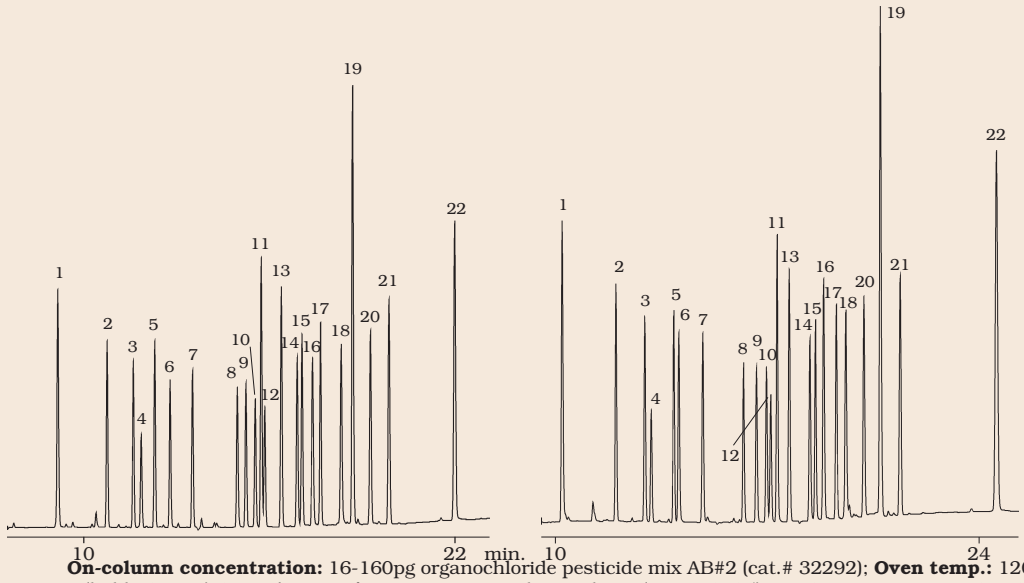
On-column concentration: 16-160pg organochloride pesticide mix AB#2 (cat.# 32292); **Oven temp.:** 120°C (hold 1 min.) to 300°C (hold 10 min.) @ 9°C/min.; **Inj. port:** Direct, Uniliner® sleeve (cat.# 20335), at 200°C; **Detector:** ECD, 300°C with Anode Purge; **Dead time:** 1.9 min.; **Head pressure:** 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. ensdosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl

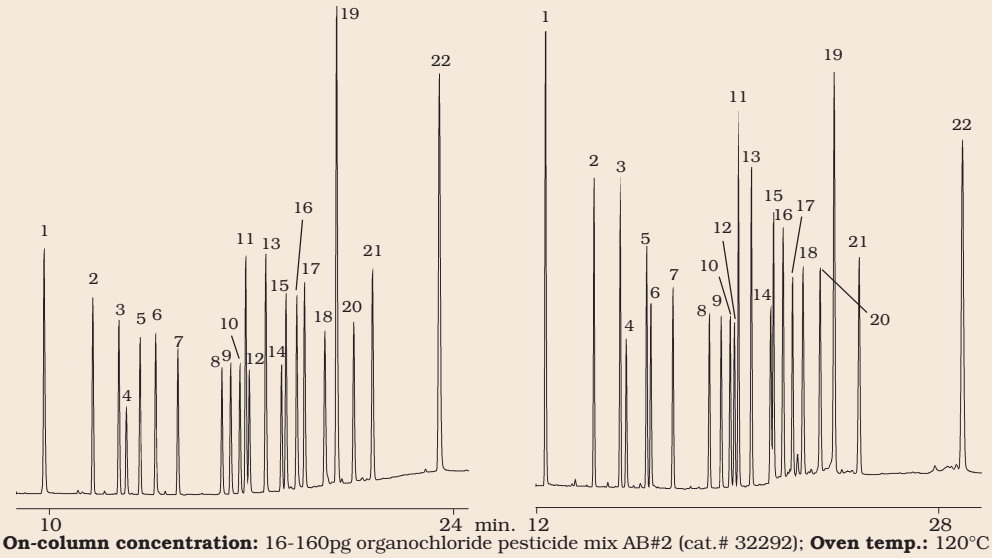
On-column concentration: 16-160pg organochloride pesticide mix AB#2 (cat.# 32292); **Oven temp.:** 120°C (hold 1 min.) to 300°C (hold 10 min.) @ 9°C/min.; **Inj. port:** Direct, Uniliner® sleeve (cat.# 20335), at 200°C; **Detector:** ECD, 300°C with Anode Purge; **Dead time:** 1.9 min.; **Head pressure:** 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. ensdosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl

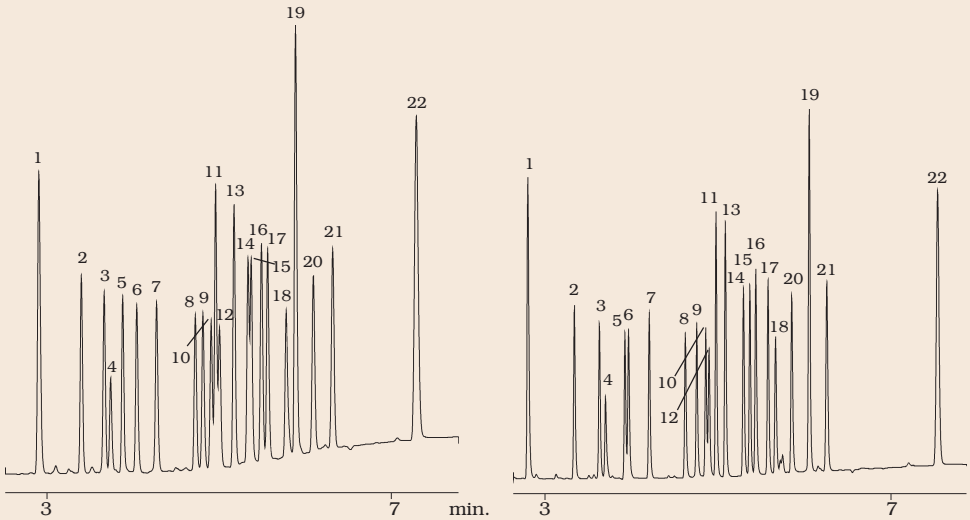
On-column concentration: 16-160pg organochloride pesticide mix AB#2 (cat.# 32292); **Oven temp.:** 120°C (hold 1 min.) to 300°C (hold 3 min.) @ 8.5°C/min.; **Inj. port:** Direct, Uniliner® sleeve (cat.# 20335), at 200°C; **Detector:** ECD, 300°C with Anode Purge; **Dead time:** 2.4 min.; **Head pressure:**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



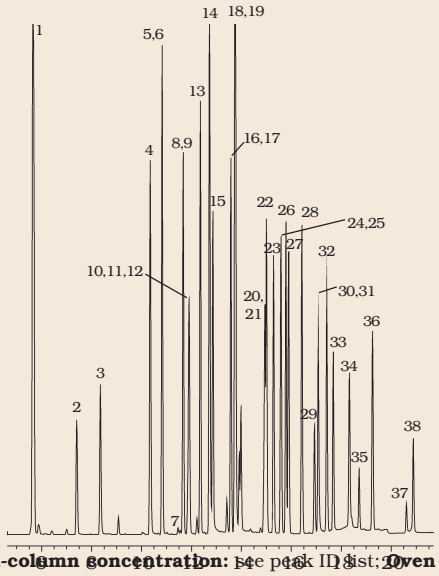
1. 2,4,5,6-tetrachloro-m-xylene
2. α-BHC (α-HCH)
3. γ-BHC (lindane)
4. β-BHC (β-HCH)
5. δ-BHC (δ-HCH)
6. heptachlor
7. aldrin
8. heptachlor epoxide
9. γ-chlordane
10. α-chlordane
11. 4,4'-DDE
12. endosulfan I
13. dieldrin
14. endrin
15. 4,4'-DDD
16. endosulfan II
17. 4,4'-DDT
18. endrin aldehyde
19.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

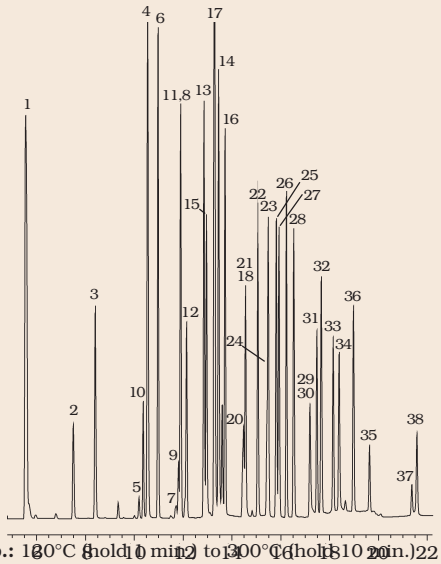
Figure 8

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)



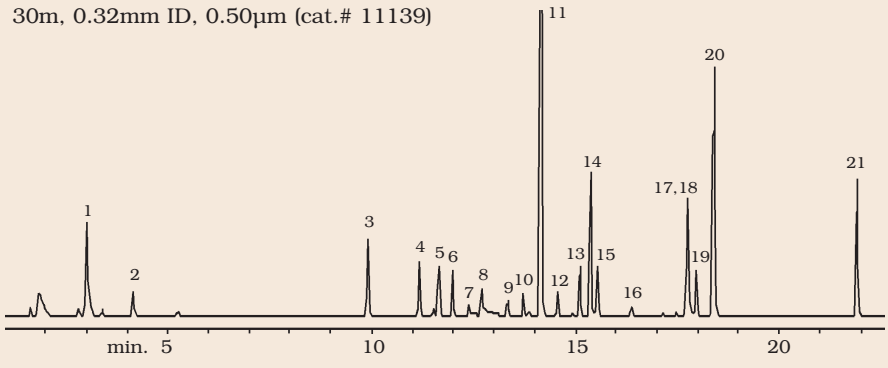
On-column concentration: see peak ID list; **Oven temp.:** 120°C (hold 10 min.) to 300°C (hold 10 min.) @ 9°C/min.; **Inj. port:** Direct, Uniliner® sleeve (cat.# 20335), at 200°C; **Detector:** ECD, 300°C with Anode Purge; **Dead time:** 1.7 min.; **Head pressure:** 12psi (constant); **Flow rate:**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

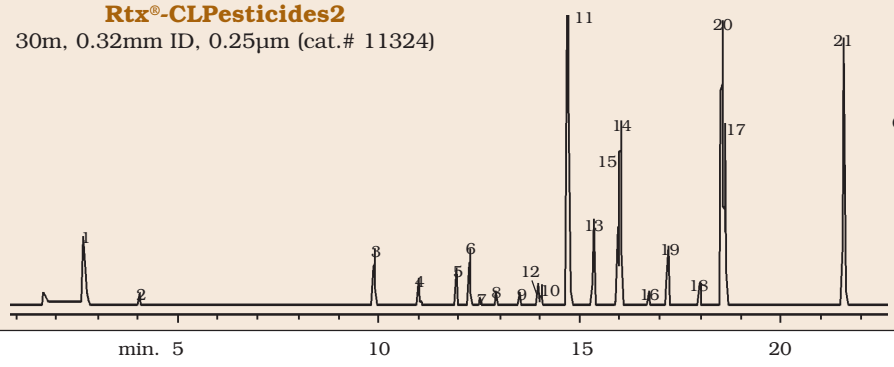
Figure 9

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/
2.	1,4-dichlorobenzene	500 µL
3.	3,5-dichlorobenzoic acid methyl ester	500
4.	4-nitroanisole	500
5.	DCAA methyl ester	500
6.	dicamba methyl ester	50
7.	MCPP 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.

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

Product Listings:

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

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

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

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.*

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

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

** 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
atrazine
chlorthalonil
cyanazine
hexachlorocyclopentadiene
metolachlor
metribuzin
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)

200µg/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	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

200µg/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

200µg/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

200µg/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
200µg/mL ea. in acetone, 1mL/ampul

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

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

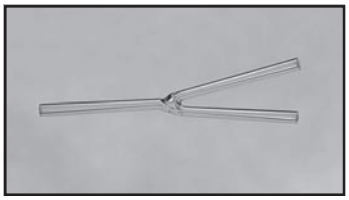
PCB Kit #2

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

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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Universal Angled "Y" Press-Tight® Connectors

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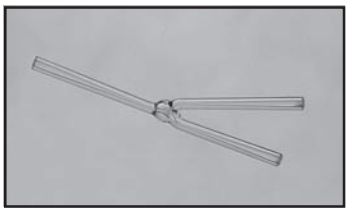
- Inlet and outlet ends conform to the column radius.
- Perform confirmational analysis with a single injection.



Universal Angled "Y" Press-Tight® Connectors

cat.# 20403 (ea.)	cat.# 20404 (3-pk.)

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- Preconditioned and ready to use.
- Do not adhere to hot metal surfaces.
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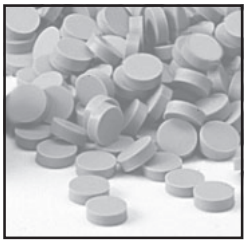
- Packaged in non-contaminating tins.

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
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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RESTEK

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Technical Guide

A Technical Guide for Static Headspace Analysis Using GC

Inside:

RESTEK

Table of Contents

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.

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.

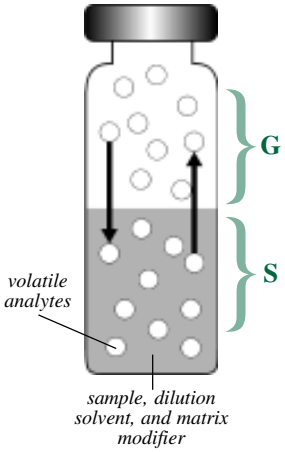


Figure 1

Phases of the headspace vial.

G=the gas phase (headspace).
The gas phase is commonly referred to as the headspace and lies above the condensed sample phase.

S=the sample phase.
The sample phase contains the compound(s) of interest and is 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, volatile components diffuse into the gas phase until the headspace has reached a state of equilibrium as depicted by the arrows. The sample is then taken from the headspace.

Figure 2

K and β are important variables in headspace analysis.

Equation 1
Partition Coefficient (K) = C_s/C_g

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**).

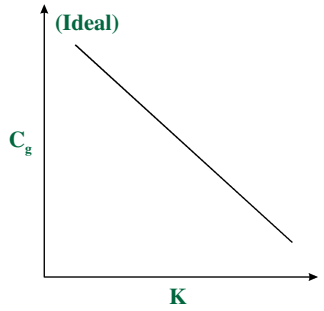
Compounds that have low K values will tend to partition more readily into the gas phase, and have relatively high responses and low limits of detection (**Figure 3**). An example of this would be hexane in water: at 40°C, hexane has a K value of 0.14 in an air-water system. Compounds that have high K 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.

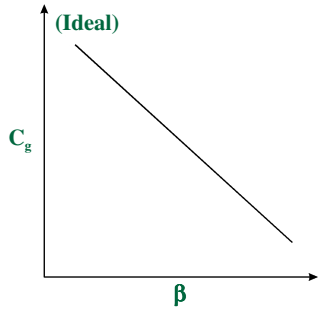


K can be lowered by changing the temperature at which the vial is equilibrated or by changing the composition of the sample matrix. In the case of ethanol, K can be lowered from 1355 to 328 by raising the temperature of the vial from 40°C to 80°C. It can be lowered even further by introducing inorganic 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

Figure 4
Sensitivity is increased when β is minimized.



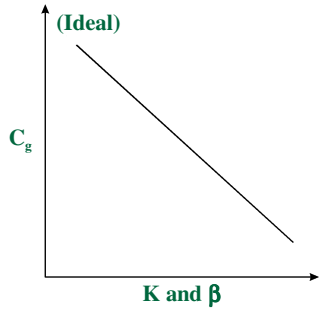
Phase Ratio

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.



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.

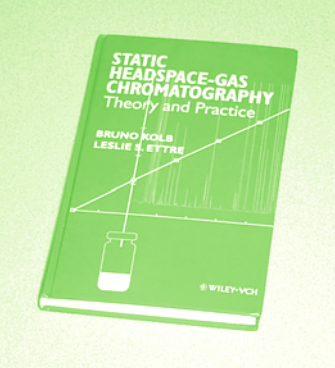
Compound of Interest	Derivatizing Reagent	Resulting Derivative
fatty acids	methanol with boron trifluoride	esterification
glycerol	acetic anhydride with sodium carbonate	acetylation

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

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.

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



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 thermostatting 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 6: *Gas syringe system*

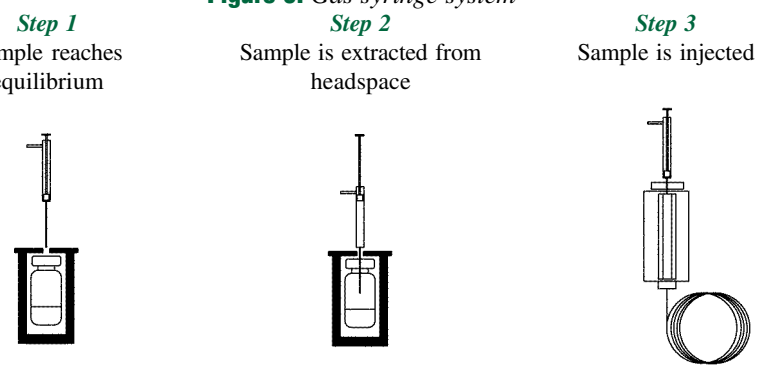
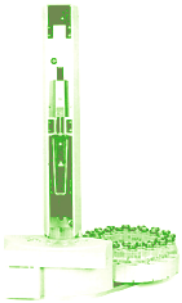


Figure 7
Gas-tight syringe autosampler TRACE HS850



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*

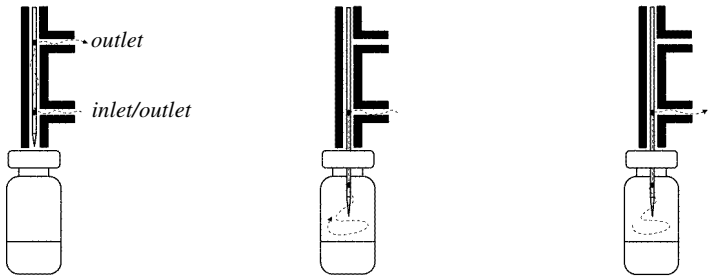
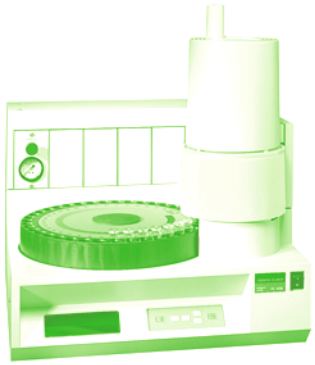


Figure 9
Balanced-pressure autosampler
Perkin-Elmer HS 40XL



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**).

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*

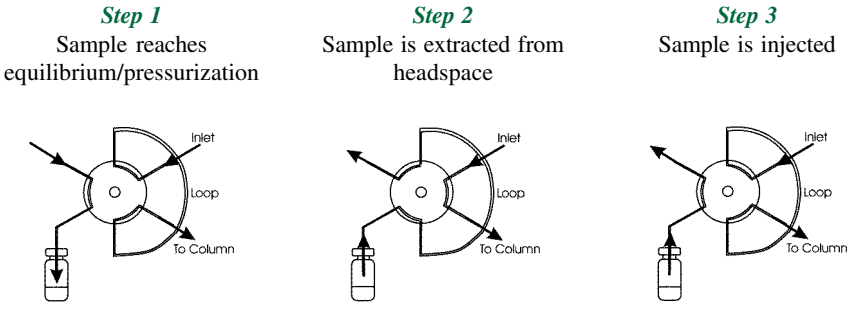


Figure 11
Pressure-loop system
OI Model 4632

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.

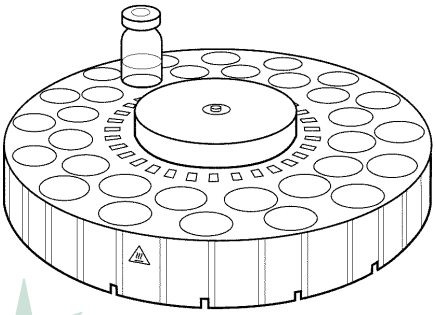
System Optimization (Troubleshooting)

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.

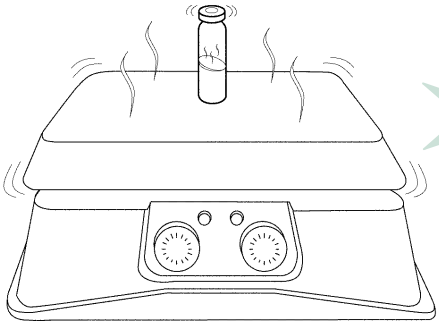
Sample Vial

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.

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).



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

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.

Sampling

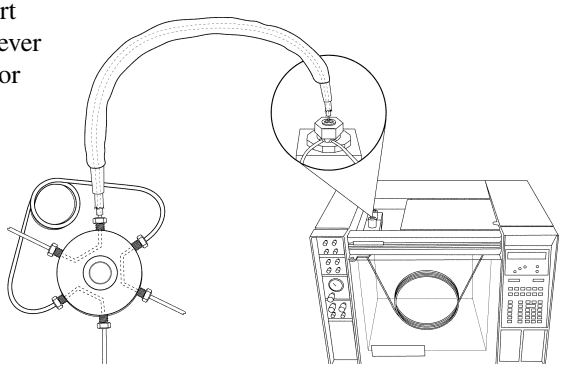
There are several techniques used to transfer samples from the vial to the GC. When using a *gas-tight syringe* for sampling, heat the syringe to a temperature comparable to the sample vial temperature. This minimizes pressure differences and condensation problems. To prevent carryover from inside the 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.

Transfer Line

After the headspace sample is withdrawn from the vial, it 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.

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.

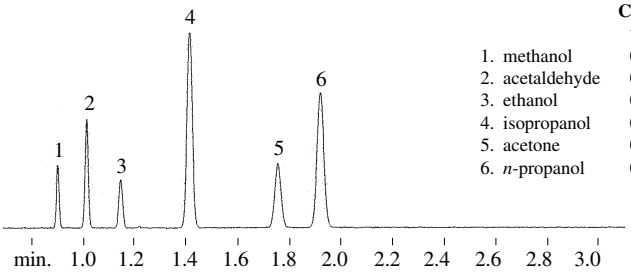
Quantitation Technique for Blood Alcohol Analysis (Internal Standard)

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

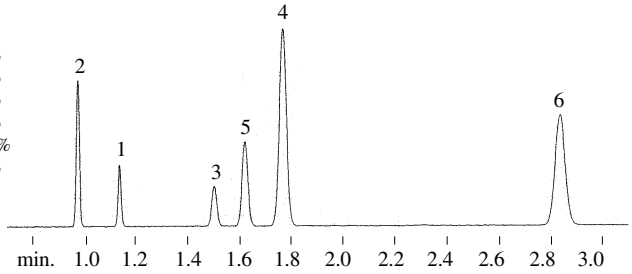
Achieve baseline resolution of all blood alcohol components in less than 3 minutes using the Rtx®-BAC1 and Rtx®-BAC2 columns and a Perkin-Elmer HS 40 headspace autosampler.

Rtx®-BAC1: 30m, 0.32mm ID, 1.8µm (cat.# 18003)



	Conc. w/v
1. methanol	0.1%
2. acetaldehyde	0.2%
3. ethanol	0.2%
4. isopropanol	0.1%
5. acetone	0.01%
6. *n*-propanol	0.1%

Rtx®-BAC2: 30m, 0.32mm ID, 1.2µm (cat.# 18002)



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**).

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 ±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>

New

benzene	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.

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

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

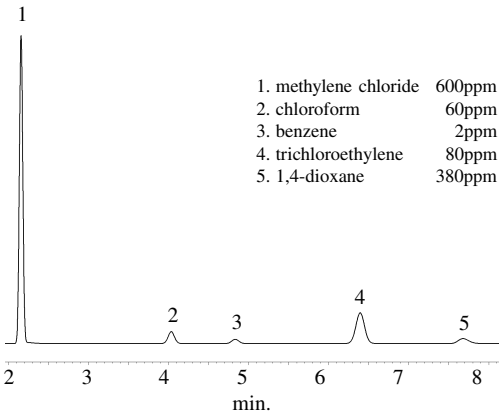
Method for Coated Tablets - Static Headspace
0.2% polyethylene glycol, MW 1500 (G39) on graphitized carbon (S7)
(0.2% Carbowax® 1500 on 80/100 CarboBlack™ C packed column, cat. # 80122)

USP also has clarified that a 5m phenylmethyl 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.

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 Forum[3] 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

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.

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.

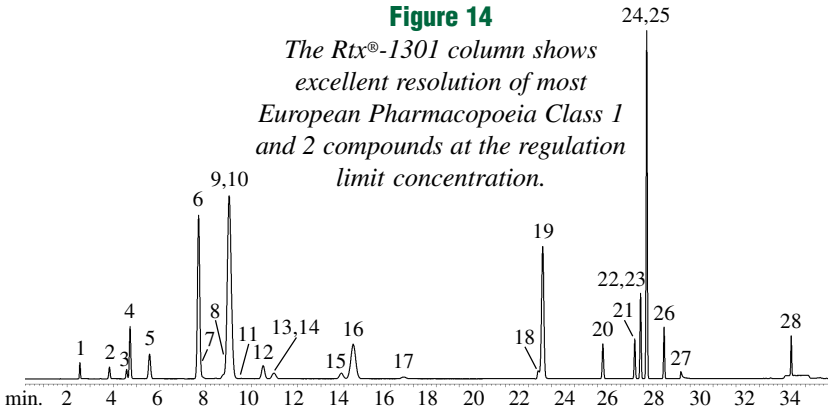
Peak List for Figure 15

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-trichlorethene)
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

Figure 14

The Rtx®-1301 column shows excellent resolution of most European Pharmacopoeia Class 1 and 2 compounds at the regulation limit concentration.



30m x .53mm ID x 3.0mm Rtx®-1301 w/5m phenyl methyl Integra-Guard™ guard column (cat.# 16085-126). **Oven temp.:** 40°C (hold 20 min.) to 240°C @ 10°C/min. (hold 20 min.); **Inj./det. temp.:** 200°C/250°C; **FID sensitivity:** 1.1 x 10^{-11} AFS; **Carrier gas:** H_2 @ 35cm/sec.; **Split ratio:** 2:1.

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®-BAC2 Capillary Columns

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

Rtx®-G27 Integra-Guard™ Column

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

Rtx®-G43 Integra-Guard™ Column

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

Rtx®-5 Capillary Columns

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

Rtx®-1301 Capillary Columns

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

Stabilwax® Capillary Columns

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

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

- 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-dimethyoxyethane	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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(814-353-1300, ext. 3)
or call your local
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GC Accessories

Headspace Autosampler Vials

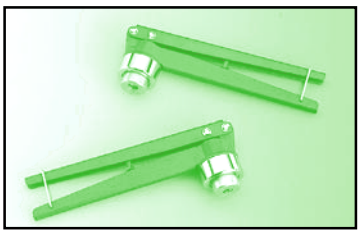
Description	100-pk.	1000-pk.
6mL Clear Vial	21166	21167
10mL Clear Vial, Flat Bottom	24683	24684
10mL Clear Vial, Rounded Bottom	21164	21165
20mL Clear Vial, Flat Bottom	24685	24686
20mL Clear Vial, Rounded Bottom	21162	21163
27mL Clear Vial	21160	21161

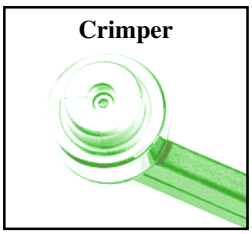
20mm Aluminum Seals w/Septa, Assembled

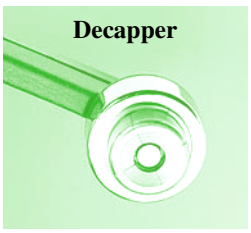
Description	100-pk.	1000-pk.
Silver Seal w/ PTFE/Gray Butyl Rubber	21761	21762
Silver Seal w/ PTFE/Silicone	21763	21764
Pressure Release, Silver Seal w/ PTFE/ Gray Butyl Rubber Septa <125°C	21765	21766
Pressure Release, Silver Seal w/ PTFE/ Silicone Septa >125°C	21767	21768

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

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Capillary Ferrules

(for 1/16" compression-type fittings)

Ferrule ID (mm)	Fits Column ID (mm)	Graphite 50-pk.	Vespel®/ 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.	Vespel®/ 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

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	——

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.

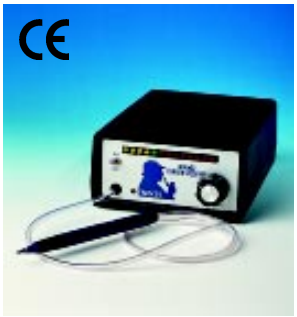
Description	Kit
LVI for HP 5890 Septum Packed Purge Port	cat.# 21698

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	Kit
LVI for HP Split/Splitless GC inlets	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 x 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	Each
Restek Leak Detective™ Electronic Leak Detector (110 volts)	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	Each
Restek Veri-Flow 500 Electronic Flowmeter (110 volts)	cat.# 21643
Restek Veri-Flow 500 Electronic Flowmeter (220 volts)	cat.# 21645

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Lit. Cat.# 59895A

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TECHNICAL GUIDE

A Guide to Passive Air Sampling

Equipment Needed and Practical Techniques for Collecting Air Samples

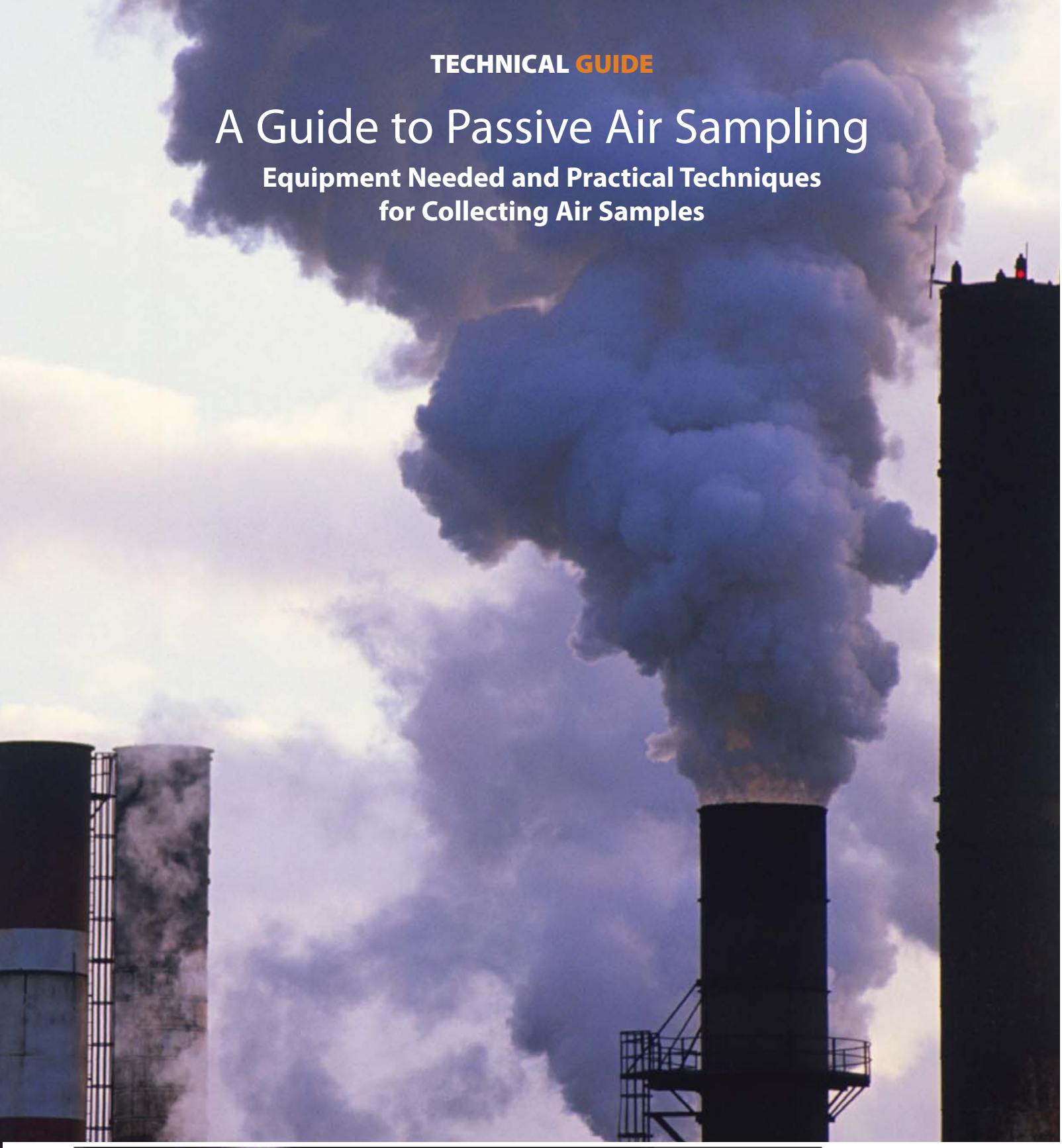


Table of Contents

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

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

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.

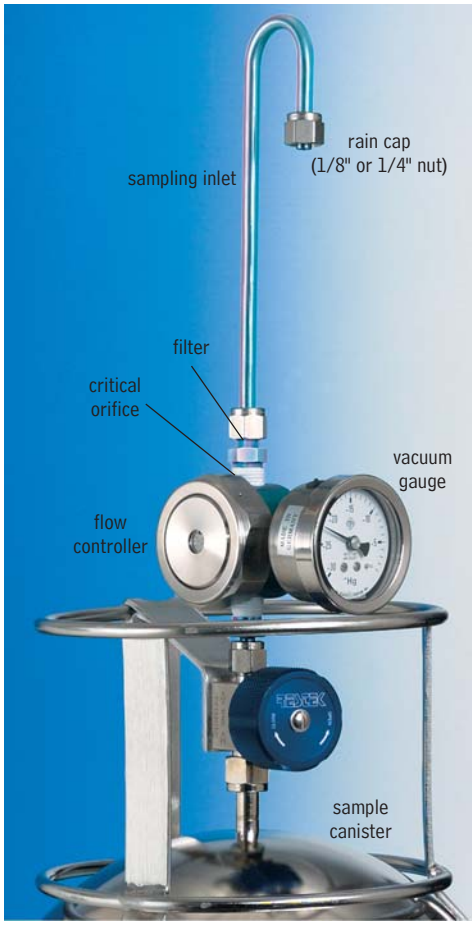


Figure 2 Filters used in sampling trains.

Figure 3 Flow controller & critical orifice.

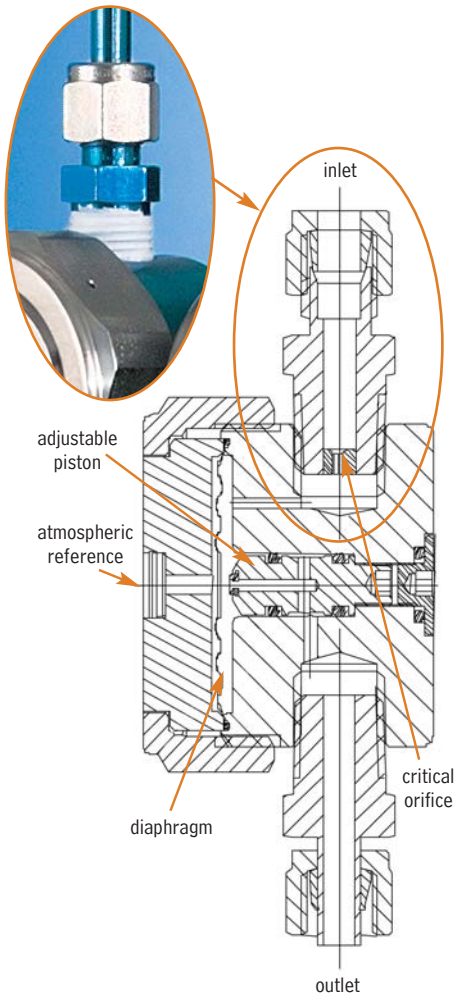


Figure 3 courtesy of Veriflo Corp., a division of Parker Hannifin Corp.

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.

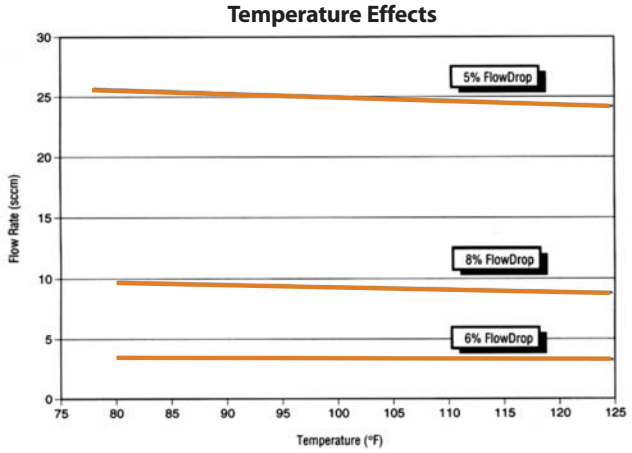
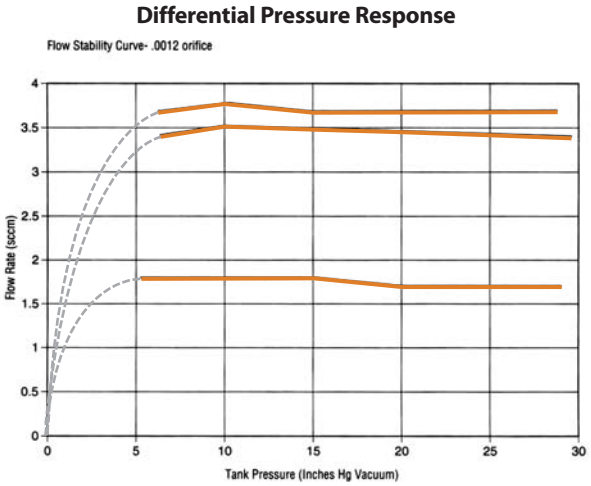


Figure 5 A flow controller will maintain a constant sample flow until it is unable to maintain a stable pressure differential across the critical orifice.



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 Molectrics) 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.

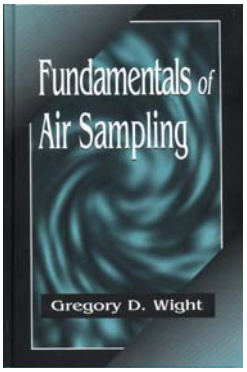
Barry Spicer, Jr.,
Restek Performance Coatings Technician

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.

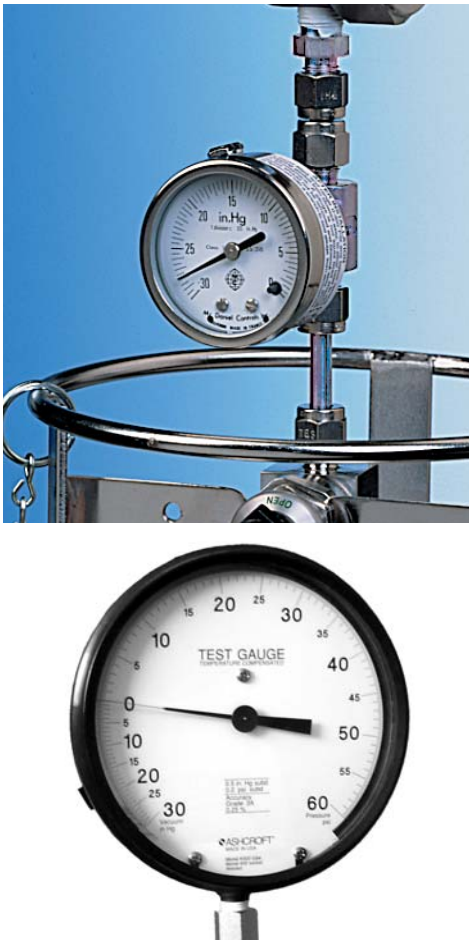
Certifying the cleanliness of the canister is important toward ensuring that results reported are solely from the site sampled, and not admixed with residue from a previous site or from contaminated laboratory air. To certify a canister clean, fill the canister with humidified air, pass the air from 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.



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.

did you **know**?

Restek canisters are shipped in boxes with handles for ease of carrying and transporting, eliminating costly carrying cases.

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 29.92" Hg vacuum; the final gauge pressure reading when the canister was returned to the lab was 7" Hg vacuum.

sample volume = [(29.92"Hg - 7"Hg) / 29.92"Hg] x 6L = 4.59 liters collected.

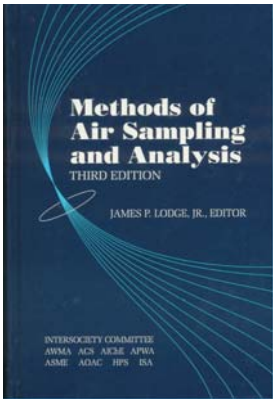
It is also a good practice to recheck the flow rate after sampling, because this will affect the sample volume (Equation 2). Laboratories typically allow a maximum deviation of +/-10% to +/-25% between the initial flow rate and the post-sampling flow rate.

Equation 2:

sample volume = [(initial flow rate + post-sampling flow rate)/2] x 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.

sample volume = [(3.3mL/min. + 3.0mL/min.) / 2] x 1440 min. = 4536mL.



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**

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:

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 x 0.491)) = 2.61

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**

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	0.58L	0.99L	1.39L	1.99L	2.59L	2.99L	3.59L	3.99L	4.60L	5.0L	5.40L	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 multipoint 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.

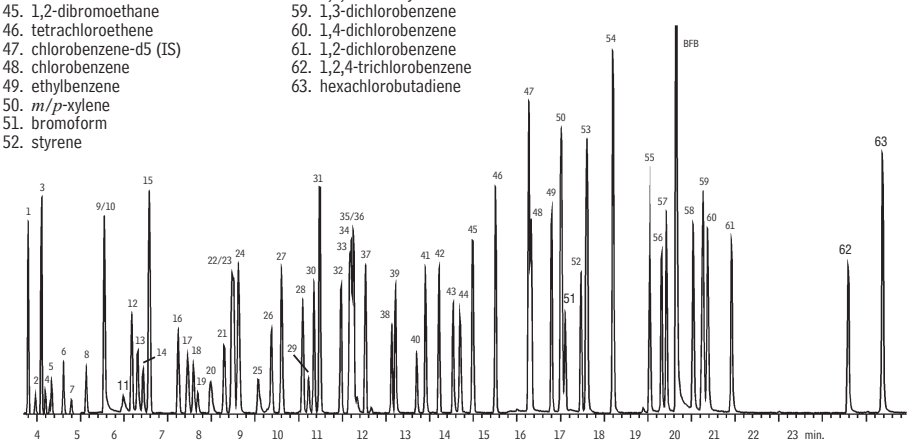
After analyzing the multipoint calibration standards and calculating peak area/concentration response factors, analyze the “real world” samples. If an "unknown" sample has not been diluted apply the corresponding response fac-

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	0.58L	0.99L	1.39L	1.99L	2.59L	2.99L	3.59L	3.99L	4.60L	5.0L	5.40L	6L
Dilution Factor	63.77	20.37	12.12	8.63	6.02	4.63	4.01	3.34	3.00	2.61	2.40	2.22	2.00

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.

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 subsampling.
- 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.

3. 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.

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, 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.

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, 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.

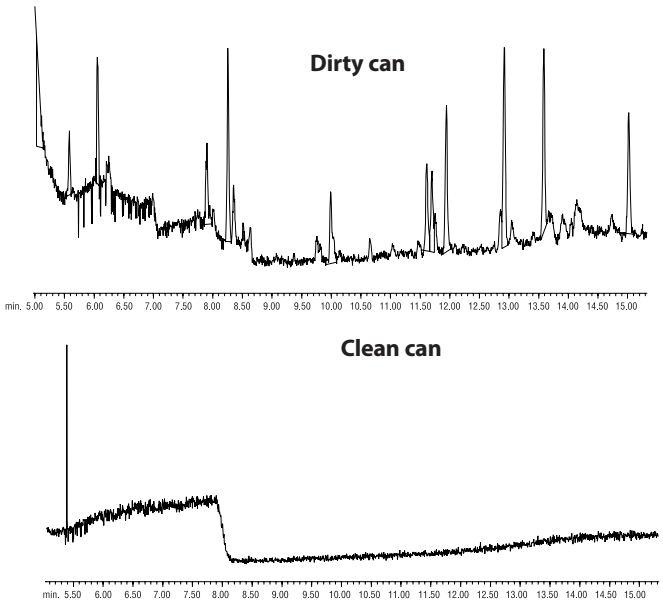
To certify a canister for analyte stability, introduce a low working concentration of a characterized test mix into the canister. Analyze an aliquot of the contents of the canister immediately after introducing the test mixture and at periodic intervals. We recommend monitoring for changes for a minimum 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

Pressure Conversion Table

Pressure	PSI	atm	kg/cm²	torr	kP*a*	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/cm² =	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
kP*a* =	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

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

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

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

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.

1/4" Replacement Valves for Air Monitoring Canisters*

	Stainless Steel Valve		Siltek®-Treated Valve	
Description	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^{-9}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.

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

Metal-seated diaphragm valve

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
bromomethane
carbon tetrachloride
chlorobenzene
chloroform
chloromethane
1,2-dibromoethane
m-dichlorobenzene
o-dichlorobenzene
p-dichlorobenzene
dichlorodifluoromethane
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethene
cis-1,2-dichloroethene
1,2-dichloropropane
cis-1,3-dichloropropene
trans-1,3-dichloropropene
dichlorotetrafluoroethane
ethyl benzene
ethyl chloride
hexachloro-1,3 butadiene
methylene chloride
styrene
1,1,2,2-tetrachloroethane
tetrachloroethylene
toluene
1,2,4-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethene
trichlorofluoromethane
1,1,2 trichlorotrifluoroethane
1,2,4-trimethylbenzene
1,3,5-trimethylbenzene
vinyl chloride
m-xylene
o-xylene
p-xylene

In nitrogen, 104 liters @ 1800psig

1ppm	100ppb
34400 (ea.)	34421 (ea.)

TO-15 62 Component Mix (62 components)

acetone
benzene
benzyl chloride*
bromodichloromethane
bromoform
bromomethane
1,3-butadiene
2-butanone (MEK)
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-dichlorethene
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
ethyl dibromide (1,1-dibromoethane)
4-ethyltoluene
trichlorofluoromethane (Freon® 11)
dichlorodifluoromethane (Freon® 12)
1,1,2-trichloro-1,2,2-trifluoroethane (Freon® 113)
1,2-dichlorotetrafluoroethane (Freon® 114)
heptane
hexachloro-1,3-butadiene
hexane
2-hexanone (MBK)
4-methyl-2-pentanone (MIBK)
methylene chloride
methyl *tert*-butyl ether (MTBE)
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

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

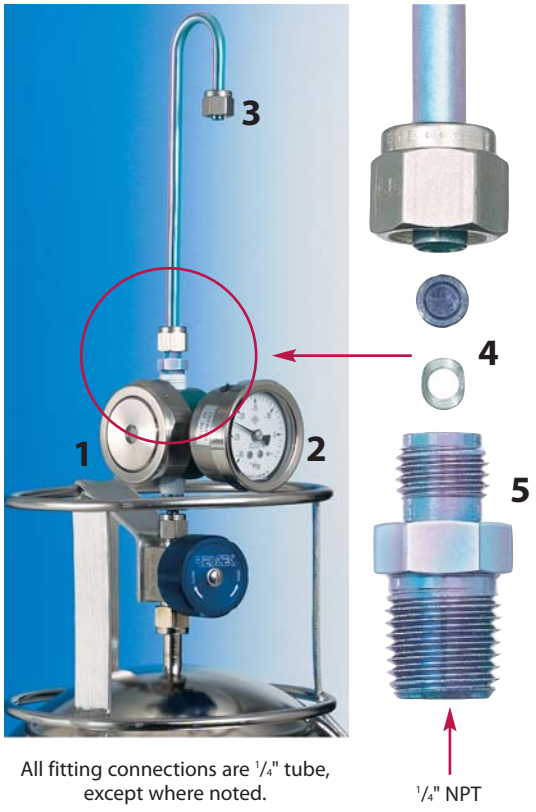
Canister Volume*/Sampling Time					Flow	Orifice	Siltek®-Treated	Stainless Steel
400cc	1 Liter	3 Liter	6 Liter	15 Liter	(sccm)	size	Sampling Kits	Sampling Kits
8 hour	24 hour	48 hour	125 hour	—	0.5–2	0.0008"	24217	24216
2 hour	4 hour	12 hour	24 hour	60 hour	2–4	0.0012"	24160	24165
1 hour	2 hour	6 hour	12 hour	30 hour	4–8	0.0016"	24161	24166
—	1 hour	4 hour	8 hour	20 hour	8–20	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

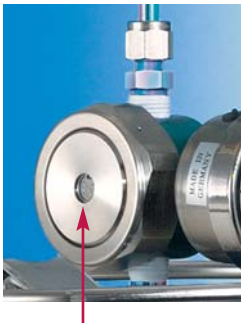
For use in critical orifice fitting. Includes washers.

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® 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

High-quality
vacuum gauge

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.

RESTEK 20 YEARS

ISO 9001:2000 cert.# FM80397

Lit. Cat.# 59977B

2006/07 Edition

CLINICAL/ FORENSICS

Products & Applications for GC & HPLC

Introduction

In the clinical and forensic markets, chromatography encompasses a wide range of application areas, including:

- hospital & forensic toxicology
- drugs of abuse
- alcohol & driving
- workplace drug testing (illicit drugs)
- drug abuse in sports (steroids, stimulants, analgesics, hormones, diuretics, narcotics)
- therapeutic drug monitoring (prescription drugs)
- natural toxins (hallucinogenic mushrooms)
- pesticide poisoning
- volatile substance abuse (adhesives, dry cleaning/degreasing agents, hydrocarbon fuels, solvents, paint strippers/thinners, vasodilators)
- solid drug identification

Example chromatograms for a variety of these applications, obtained using Restek GC and HPLC columns and accessories, are illustrated in this guide.

Throughout this guide you also will find references to application notes that detail specific analyses, technical guides that explain various analytical techniques, product information pieces that highlight aspects of our products, and reference books that offer detailed information on topics from sample preparation to chromatographic methods. All Restek publications are available free on request; order by contacting our Technical Service Department, our Customer Service Department, or your Restek distributor by e-mail, telephone, or fax, and providing the literature catalog numbers for the publications you want. For fastest access, you can review and download these publications from our website. For prices for reference books, refer to our general catalog or website - or simply call our Customer Service Department.

Kristi Sellers
Clinical/Forensic
Innovations Chemist

Kristi brings 18 years of experience in GC and 4 years of experience in HPLC to her role as our principal clinical and forensic applications chemist. She holds a B.S. in chemistry from Lock Haven University, Lock Haven, PA.

E-mail: kristi.sellers@restek.com
Phone: 800-356-1688 or 814-353-1300, ext. 2150

Table of Contents:

Arson Accelerants

Arson Accelerants

Analyses of fire debris for accelerants commonly are performed on non-polar columns, like the Rtx®-1 column. Thick film columns give better resolution for the low boiling point compounds that are components of gasoline. Thinner film columns provide better resolution and shorter analysis times for higher molecular weight compounds in diesel fuels. However, by choosing the appropriate column dimensions and analytical parameters, a wide range of petroleum products can be tested for on a single column.

Accelerants in fire debris are identified through chromatographic pattern comparison. Accelerants and that have been exposed to high heat exhibit a "weathered" pattern. Large proportions of the lower boiling components in any accelerant will have disappeared in samples that have undergone severe weathering. We have an extensive line of petroleum products that have been weathered to various stages of weight loss to aid in the identification of accelerants in fire debris.

free literature

Weathered Petroleum Analytical Reference Materials

lit. cat.# 59215

Fire Debris Analysis

lit. cat.# 59574

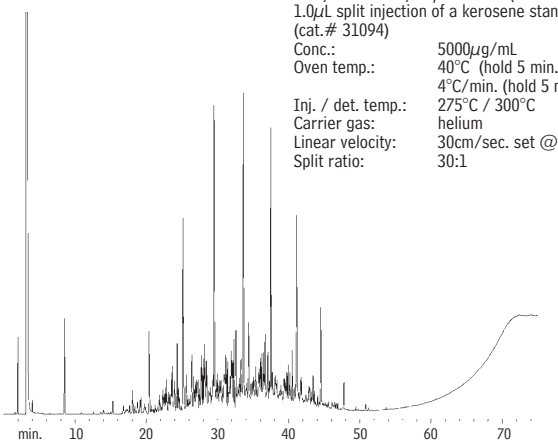
Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

GC

Kerosene
Rtx®-1

30m, 0.32mm ID, 1.5µm Rtx®-1 (cat.# 10169)
1.0µL split injection of a kerosene standard (cat.# 31094)

Conc.:	5000µg/mL
Oven temp.:	40°C (hold 5 min.) to 300°C @ 4°C/min. (hold 5 min.)
Inj. / det. temp.:	275°C / 300°C
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 40°C
Split ratio:	30:1



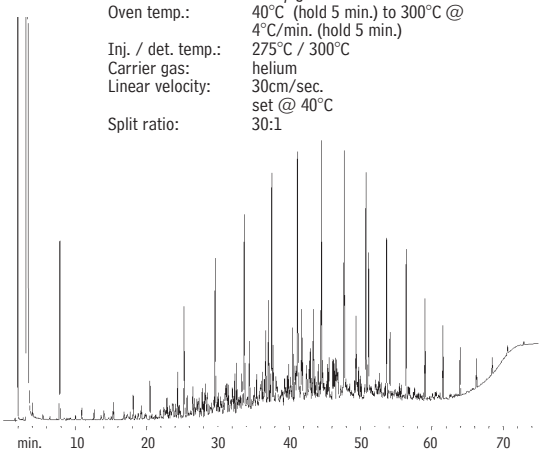
GC_MS00274

GC

Diesel Fuel
Rtx®-1

30m, 0.32mm ID, 1.5µm Rtx®-1 (cat.# 10169)
1.0µL split injection of a diesel fuel #2 standard (cat.# 31093)

Conc.:	5000µg/mL
Oven temp.:	40°C (hold 5 min.) to 300°C @ 4°C/min. (hold 5 min.)
Inj. / det. temp.:	275°C / 300°C
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 40°C
Split ratio:	30:1



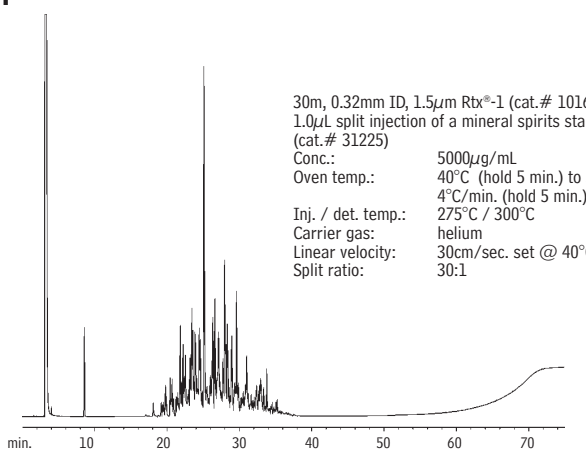
GC_MS00275

GC

Mineral Spirits
Rtx®-1

30m, 0.32mm ID, 1.5µm Rtx®-1 (cat.# 10169)
1.0µL split injection of a mineral spirits standard (cat.# 31225)

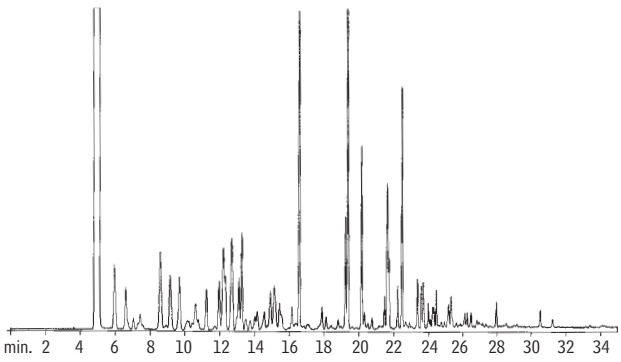
Conc.:	5000µg/mL
Oven temp.:	40°C (hold 5 min.) to 300°C @ 4°C/min. (hold 5 min.)
Inj. / det. temp.:	275°C / 300°C
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 40°C
Split ratio:	30:1



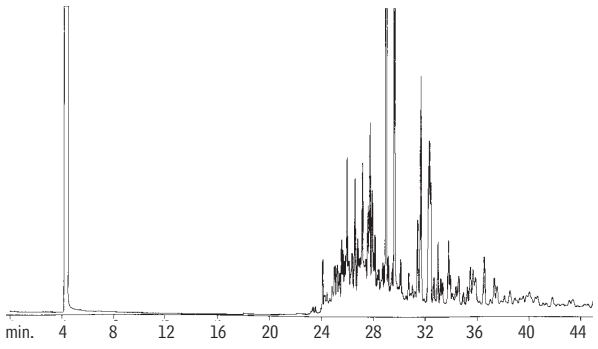
Unleaded Gasoline
Rtx®-1

GC

Unweathered



99% Weathered

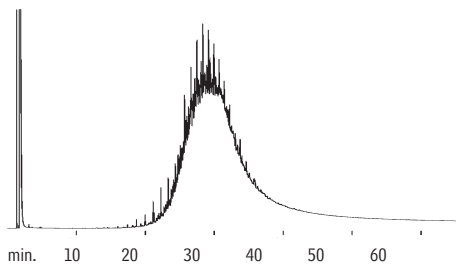


GC_MS00270

30m, 0.53mm ID, 1.50μm Rtx®-1 (cat.# 10170)
Oven temp.: 40°C (hold 3 min.) to 75°C @ 15°C/min. to 275°C @ 20°C/min. (hold 5 min.)
Inj./det. temp.: 250°C/285°C
Carrier gas: hydrogen
Linear velocity: 50cm/sec. set @ 40°C
FID sensitivity: 4.10 x 10⁻⁹ AFS
Split ratio: 30:1

Motor Oil
Rtx®-5

GC

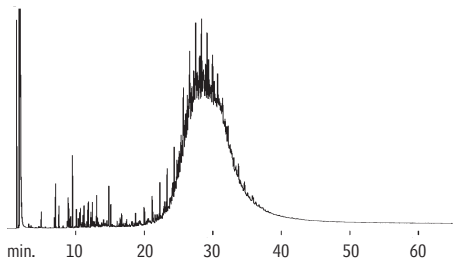


GC_MS00272

30m, 0.53mm ID, 1.0μm Rtx®-5 (cat.# 10255)
1.0μL split injection of Motor Oil Composite Standard (cat.# 31464)*
Conc.: 50,000ppm
Oven temp.: 40°C (hold 2 min.) to 330°C @ 10°C/min. (hold 60 min.)
Inj./det. temp.: 300/330°C
Carrier gas: hydrogen
Linear velocity: 41cm/sec.
Split ratio: 3:1

*Prepared from an equal volume blend of 5w30, 10w30, 10w40, and 20w50 motor oil, precisely weighed to produce a mixture at 50,000μg/mL in methylene chloride.

Motor Oil (Used)
Rtx®-5



GC_MS00273

30m, 0.25mm ID, 0.25μm Rtx®-5 (cat.# 10223)
1.0μL split injection of Used Motor Oil Composite Standard (cat.# 31465)*
Conc.: 50,000ppm
Oven temp.: 40°C to 340°C @ 10°C/min. (hold 15 min.)
Inj./det. temp.: 250/340°C
Carrier gas: hydrogen
Linear velocity: 40cm/sec.
Split ratio: 15:1

*Prepared from an equal volume blend from five gasoline powered vehicles, precisely weighed to produce a mixture at 50,000μg/mL in methylene chloride.

Abused Inhalants; Anesthetics

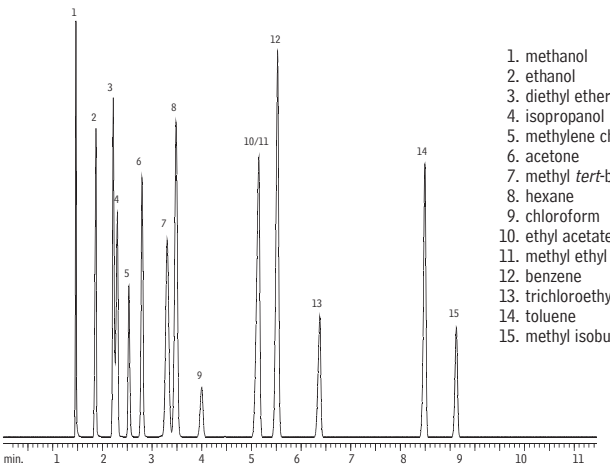
Abused Inhalants/ Anesthetics

Inhalant abuse can be detected during screening of whole blood, serum, or urine samples, using headspace (GC) with flame ionization detection (FID). Here, we used a GC equipped with an automated headspace sampler that simultaneously introduces a sample into two analytical columns. A dual-column configuration provides screening and confirmational data from the same injection. We used an Rtx®-BAC1 column (30m, 0.53mm ID, 3.00µm df) and an Rtx®-BAC2 column (30m, 0.53mm ID, 2.00µm df)—columns typically used in combination as a screening and confirmation column set for blood alcohol analysis. A useful extension of blood alcohol analysis using this column set is the detection of other volatile organic compounds (VOCs), such as those in inhalants and anesthetics.

Abused Inhalants
Rtx®-BAC1 & Rtx®-BAC2

GC

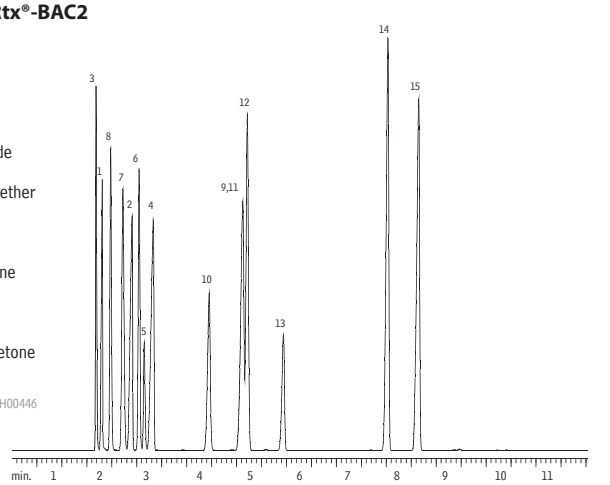
Rtx®-BAC1



1. methanol
2. ethanol
3. diethyl ether
4. isopropanol
5. methylene chloride
6. acetone
7. methyl *tert*-butyl ether
8. hexane
9. chloroform
10. ethyl acetate
11. methyl ethyl ketone
12. benzene
13. trichloroethylene
14. toluene
15. methyl isobutyl ketone

GC_PH00446

Rtx®-BAC2



30m, 0.32mm ID, 1.80µm Rtx®-BAC1 (cat.# 18003)
30m, 0.32mm ID, 1.20µm Rtx®-BAC2 (cat.# 18002)

Oven temp.:	40°C (hold 4 min.) to 120°C @ 10°C/min.
Carrier gas:	helium
Linear velocity:	50cm/sec.
Detector:	FID

Sample:	250µL headspace
Transfer line temp.:	125°C
Sample loop temp.:	125°C
Vial temp.:	70°C

Acknowledgement: Analyses performed using a Tekmar 7000 Headspace Autosampler on loan courtesy of Tekmar-Dohrmann.

for **more** info

GC Analysis of Commonly Abused Inhalants in Blood, Using Rtx®-BAC1 and Rtx®-BAC2 Columns

lit. cat.# 59548

A Technical Guide for Static Headspace Analysis, Using GC

lit. cat.# 59895A

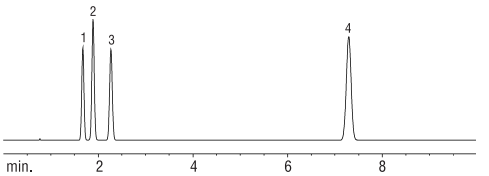
Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

Abused Anesthetics
Rtx®-BAC1 & Rtx®-BAC2

GC

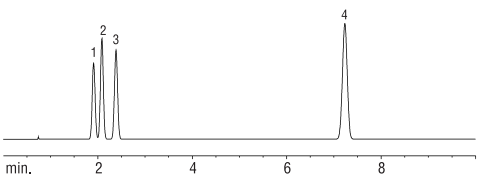
Rtx®-BAC1

1. isoflurane
2. enflurane
3. halothane
4. methoxyflurane



GC_PH00501

Rtx®-BAC2



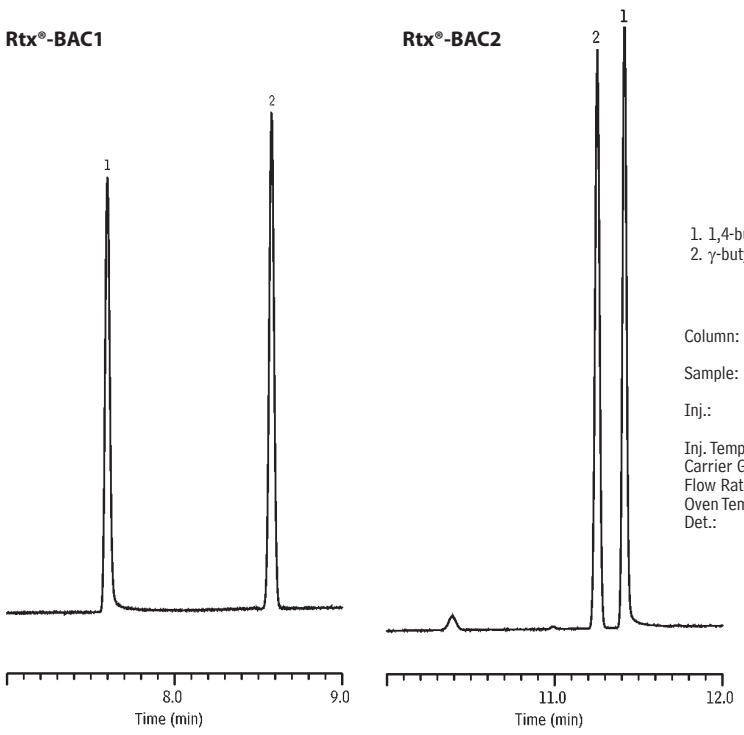
GC_PH00502

30m, 0.53mm ID, 3.0µm Rtx®-BAC1 (cat.# 18001)
30m, 0.53mm ID, 2.0µm Rtx®-BAC2 (cat.# 18000)
1.0mL headspace sample

Oven temp.:	40°C (hold 5 min.) to 240°C @ 5°C/min.
Inj. & det. temp.:	240°C
Carrier gas:	helium
Linear velocity:	65cm/sec.

GC

γ-butyrolactone & 1,4-butanediol
Rtx®-BAC1 & Rtx®-BAC2



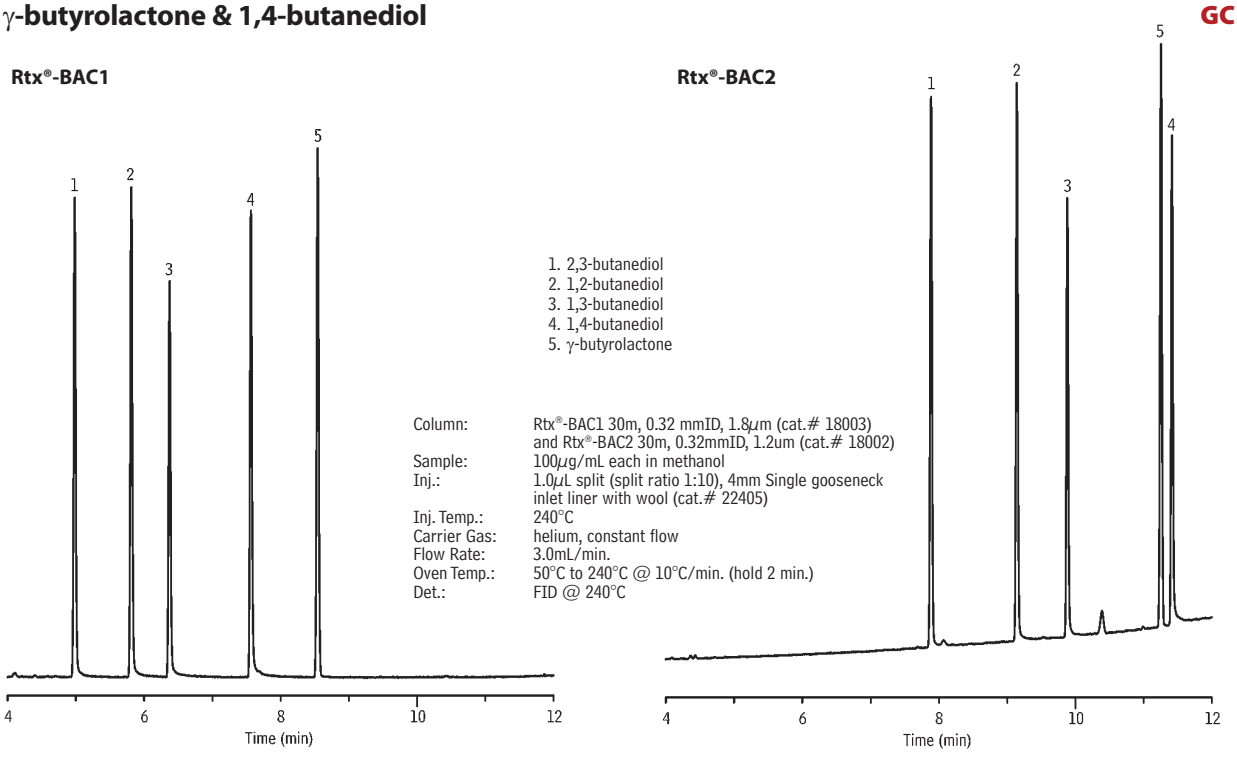
GC_PH00783 GC_PH00784

1. 1,4-butanediol
2. γ-butyrolactone

Column:	Rtx®-BAC1 30m, 0.32 mm ID, 1.8μm (cat.# 18003) and Rtx®-BAC2 30m, 0.32 mmID, 1.2μm (cat.# 18002)
Sample:	100μg/mL each γ-butyrolactone (GBL) and 1, 4-butanediol in methanol
Inj.:	1.0μL split (split ratio 1:10), 4mm single gooseneck inlet liner with wool (cat. # 22405)
Inj. Temp.:	240°C
Carrier Gas:	helium, constant flow
Flow Rate:	3.0mL/min.
Oven Temp.:	50°C to 240°C @ 10°C/min. (hold 2 min.)
Det.:	FID @ 240°C

GC

γ-butyrolactone & 1,4-butanediol



GC_PH00808 GC_PH00809

1. 2,3-butanediol
2. 1,2-butanediol
3. 1,3-butanediol
4. 1,4-butanediol
5. γ-butyrolactone

Column:	Rtx®-BAC1 30m, 0.32 mmID, 1.8μm (cat.# 18003) and Rtx®-BAC2 30m, 0.32mmID, 1.2um (cat.# 18002)
Sample:	100μg/mL each in methanol
Inj.:	1.0μL split (split ratio 1:10), 4mm Single gooseneck inlet liner with wool (cat.# 22405)
Inj. Temp.:	240°C
Carrier Gas:	helium, constant flow
Flow Rate:	3.0mL/min.
Oven Temp.:	50°C to 240°C @ 10°C/min. (hold 2 min.)
Det.:	FID @ 240°C

Barbiturates

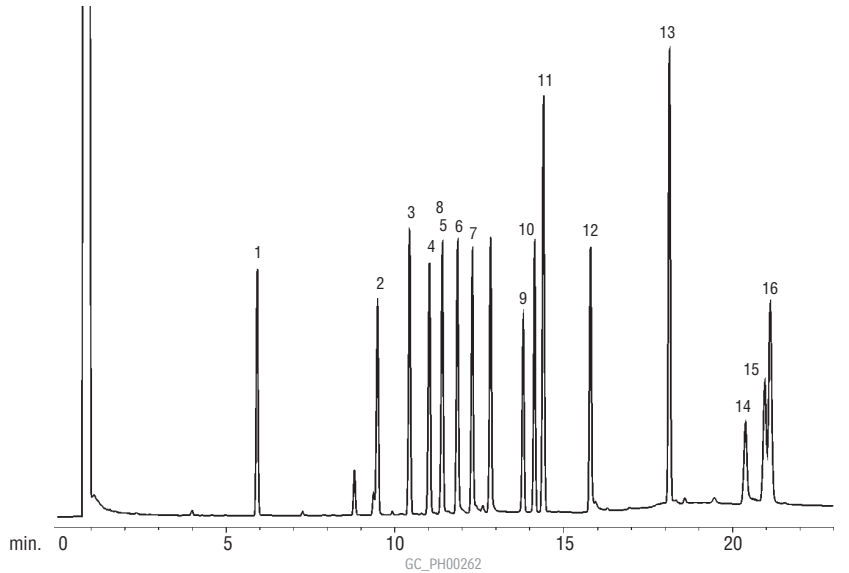
Barbiturates

These acidic and neutral drugs normally are analyzed in their underivatized forms. Symmetric peaks and baseline resolution for many compounds can be achieved by using intermediate polarity Restek columns, Rtx®-35 and Rtx®-1701. In drug overdose or drug abuse testing, these two columns can be used in combination, in dual column analysis for screening and tentatively confirming the presence or absence of specific compounds. Differences in polarity for each stationary phase produce shifts in retention time and elution order. High thermal stability and low column bleed allow these drugs to be detected by FID with on-column concentrations in the low nanogram range. When analyzing underivatized barbiturates, a deactivated inlet liner should be used in the injection port to prevent adsorption onto the surface of the liner. Liners should be changed frequently to prevent the accumulation of non-volatile contamination.

To avert injection port adsorption problems, barbiturates also can be analyzed by gas chromatography in their derivatized forms. They can be derivitized on-column, using methylating reagents like TMAH or TMPAH.

GC

Barbiturates (Underivitized)
Rtx®-35

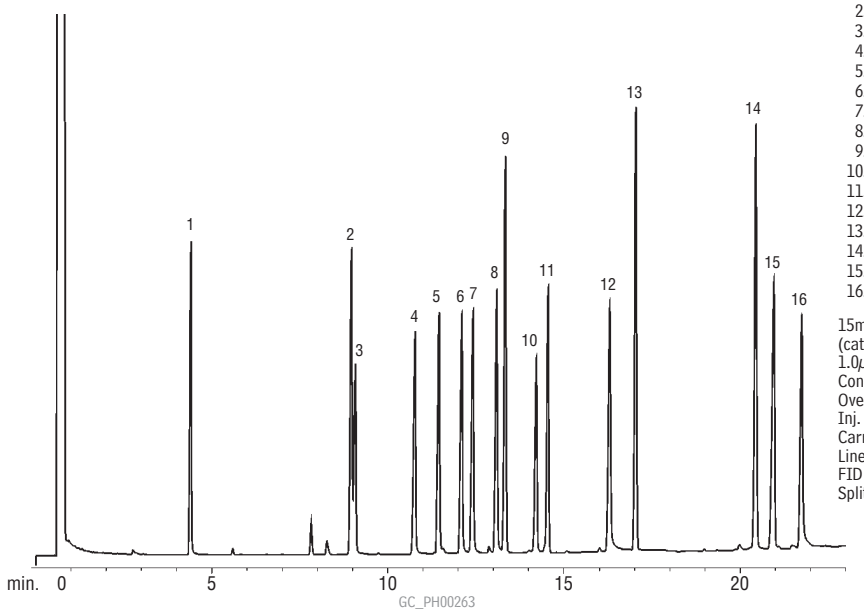


1. ethosuximide
2. barbital
3. methyprylon
4. aprobarbital
5. butalbital
6. amobarbital
7. pentobarbital
8. secobarbital
9. meprobamate
10. carisoprodal
11. glutethimide
12. phenobarbital
13. methaqualone
14. primidone
15. carbamazepine
16. diphenylhydantoin

30m, 0.53mm ID, 1.0µm Rtx®-35 (cat.# 10455)
1.0µL splitless injection

Conc.:	50µg/mL
Oven temp.:	100°C to 280°C @ 10°C/min. (hold 5 min.)
Inj. & det. temp.:	250°C/ 275°C
Carrier gas:	helium
Linear velocity:	40cm/sec. set @ 100°C
FID sensitivity:	5.12 x 10^{-10} AFS
Splitless hold time:	0.5 min.

Barbiturates (Underivatized)
Rtx®-1701



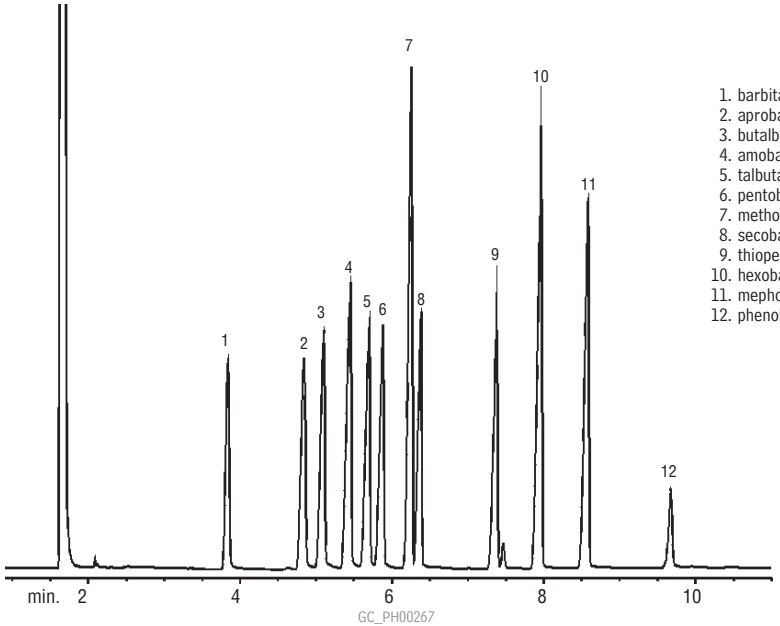
1. ethosuximide
2. methyprylon
3. barbital
4. aprobarbital
5. butalbital
6. amobarbital
7. pentobarbital
8. secobarbital
9. glutethimide
10. meprobamate
11. carisoprodal
12. phenobarbital
13. methaqualone
14. carbamazepine
15. primidone
16. diphenylhydantoin

15m, 0.53mm ID, 0.50µm Rtx®-1701 (cat.# 12037)
1.0µL splitless injection

Conc.:	50µg/mL
Oven temp.:	100°C to 280°C @ 7°C/min.
Inj. / det. temp.:	250°C / 275°C
Carrier gas:	helium
Linear velocity:	40cm/sec. set @ 100°C
FID sensitivity:	5.12 x 10^{-10} AFS
Splitless hold time:	0.5 min.

Barbiturates (Underivatized)
Rtx®-35

GC



1. barbital
2. aprobarbital
3. butalbital
4. amobarbital
5. talbutal
6. pentobarbital
7. methohexital
8. secobarbital
9. thiopental
10. hexobarbital
11. mephobarbital
12. phenobarbital

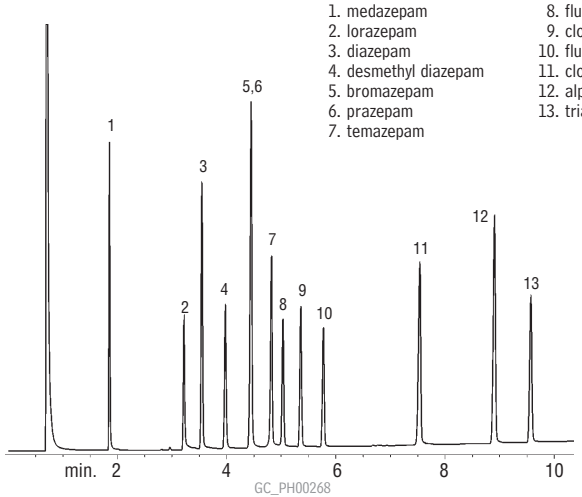
GC_PH00267

30m, 0.32mm ID, 0.50µm Rtx®-35 (cat.# 10439)
1.0µL split injection of barbiturates

Oven temp.:	210°C (hold 2 min.) to 300°C @ 7°C/min. (hold 2 min.)
Inj. & det. temp.:	300°C
Carrier gas:	helium
Linear velocity:	35cm/sec. set @ 210°C
FID sensitivity:	5.12 x 10^{-10} AFS
Split vent:	30:1

Benzodiazepines (Underivatized)
Rtx®-200

GC



1. medazepam
2. lorazepam
3. diazepam
4. desmethyl diazepam
5. bromazepam
6. prazepam
7. temazepam
8. flurazepam
9. clobazam
10. flunitrazepam
11. clonazepam
12. alprazolam
13. triazolam

GC_PH00268

15m, 0.32mm ID, 0.25µm Rtx®-200 (cat.# 15021)
1.0µL split injection of benzodiazepines

Conc.:	15ng/component
Oven temp.:	225°C to 325°C @ 8°C/min.
Inj. / det. temp.:	250°C / 320°C
Carrier gas:	helium
Linear velocity:	35cm/sec. set @ 225°C
FID sensitivity:	5.12 x 10^{-10} AFS
Split ratio:	60:1

Benzodiazepines

The Rtx®-200 stationary phase provides excellent resolution and peak shape for benzodiazepines. The unique selectivity of the Rtx®-200 trifluoropropyl methyl polymer helps to resolve this group of compounds, which are very closely related structurally. The high thermal stability of the stationary phase provides a stable baseline at high temperature, to facilitate analysis of picogram quantities of these materials.

free literature

Barbituate Analysis

lit. cat.# 59575

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Basic Drugs (Mixed)

Mixed Basic Drugs

Many basic compounds can be screened for in any given analysis, using a single Restek column. Our bonded polysiloxane stationary phases exhibit low column bleed and high thermal stability while offering a variety of compositions that yield selective retention for specific analytes. Screening and confirmation for individual drugs can be performed simultaneously by using two dissimilar stationary phases in a dual column configuration.

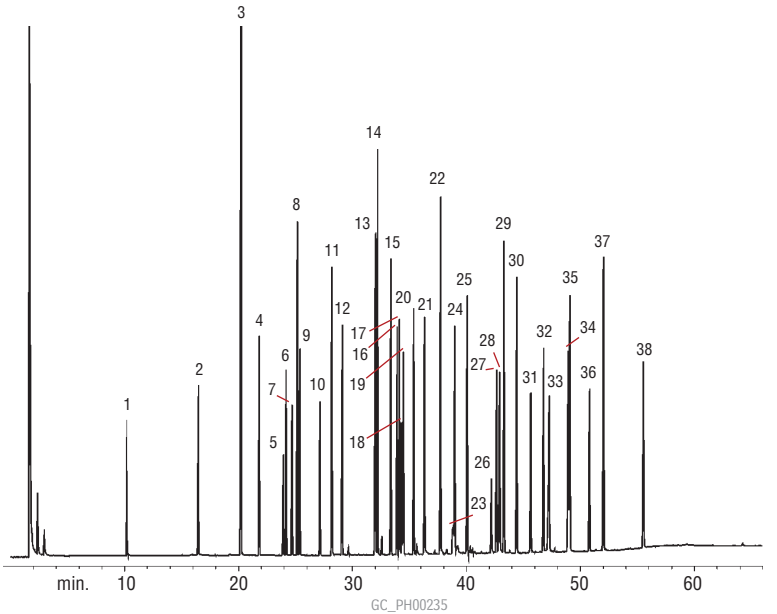
Rtx®-5 and Rtx®-35 phases are 5% and 35% phenyl/methyl polysiloxanes, respectively. The Rtx®-5 column has been used as the primary analytical column by many laboratories and can resolve all 38 compounds in this mixture of basic drugs in less than 60 minutes. The higher phenyl content of the Rtx®-35 phase increases its polarity and retention of more polar compounds, resulting in several elution order and retention time shifts. Under the same conditions used for the Rtx®-5 column, the Rtx®-35 column also resolves all 38 basic drugs, with the exception of pyrilamine and bupivacaine.

Rtx®-200 is a trifluoropropyl polysiloxane that exhibits unique selectivity for compounds with lone pairs of electrons. This column displays very different retention times and elution orders in comparison to the Rtx®-5 or Rtx®-35 column. Under the same analytical conditions, several coelutions occur with the Rtx®-200 column, including: cotinine/caffeine, bupivacaine/chlorpromazine, bromazepam/prazepam, and flurazepam/papaverine.

Because all three columns can be operated under the same temperature program conditions, simultaneous dual column confirmation analysis can be accomplished by using either an Rtx®-35 column or an Rtx®-200 column in conjunction with an Rtx®-5 column. This increases qualitative and quantitative reliability without sacrificing analysis time.

GC

Mixed Basic Drugs (Underivatized)
Rtx®-5



1. nicotine
2. benzocaine
3. cotinine
4. meperidine
5. caffeine
6. benzphetamine
7. ketamine
8. diphenhydramine
9. lidocaine
10. phenyltoloxamine
11. tripelennamine
12. phenothiazine
13. dextromethorphan
14. methadone
15. amitriptyline
16. trimipramine
17. tetracaine
18. pyrilamine
19. medazepam
20. bupivacaine
21. scopolamine
22. codeine
23. morphine
24. diazepam
25. chlorpromazine
26. temazepam
27. flunitrazepam
28. bromazepam
29. prazepam
30. acetopromazine
31. flurazepam
32. papaverine
33. clonazepam
34. haloperidol
35. alprazolam
36. triazolam
37. thioridazine
38. trazodone

30m, 0.25mm ID, 0.25µm Rtx®-5 (cat.# 10223)
1.0µL split injection of a basic drug sample.

Conc.:	1000ng/µL
Oven temp.:	100°C to 325°C @ 4°C/min. (hold 10 min.)
Inj. / det. temp.:	250°C / 320°C
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 100°C
FID sensitivity:	1.28 x 10^{-10} AFS
Split ratio:	50:1

free literature

Improved GC Analysis of Basic Organic Compounds Using Base Deactivated Columns & Liners.

lit. cat.# 59108

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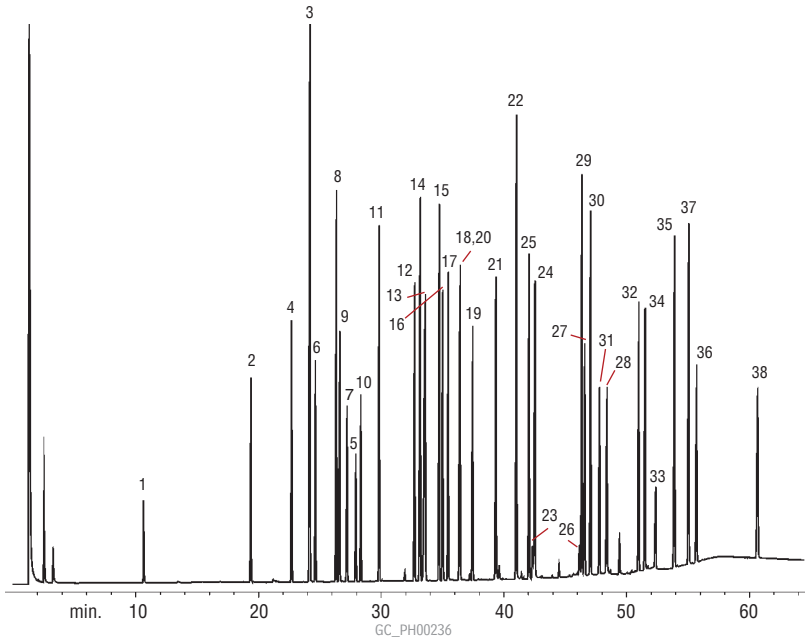
Mixed Basic Drugs (Underivatized)
Rtx®-35

GC

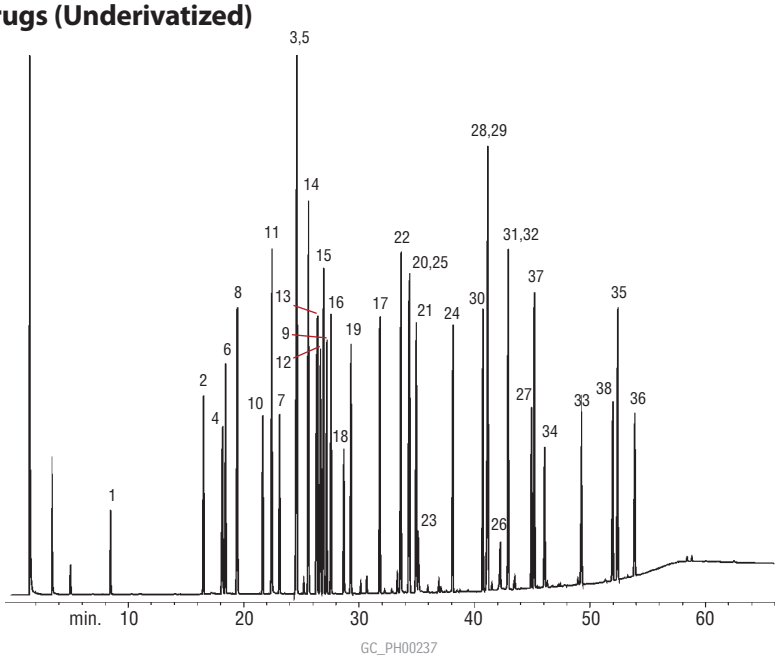


30m, 0.25mm ID, 0.25µm Rtx®-35 (cat.# 10423)
1.0µL split injection of a basic drug mix (1mg/mL)

Conc.:	1000ng/µL
Oven temp.:	100°C to 325°C @ 4°C/min. (hold 10 min.)
Inj. / det. temp.:	250°C / 320°C
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 100°C
FID sensitivity:	1.28 x 10^{-10} AFS
Split ratio:	50:1

1. nicotine
2. benzocaine
3. cotinine
4. meperidine
5. caffeine
6. benzphetamine
7. ketamine
8. diphenhydramine
9. lidocaine
10. phenyltoloxamine
11. tripelennamine
12. phenothiazine
13. dextromethorphan
14. methadone
15. amitriptyline
16. trimipramine
17. tetracaine
18. pyrilamine
19. medazepam
20. bupivacaine
21. scopolamine
22. codeine
23. morphine
24. diazepam
25. chlorpromazine
26. temazepam
27. flunitrazepam
28. bromazepam
29. prazepam
30. acetopromazine
31. flurazepam
32. papaverine
33. clonazepam
34. haloperidol
35. alprazolam
36. triazolam
37. thioridazine
38. trazodone

Mixed Basic Drugs (Underivatized)
Rtx®-200



30m, 0.25mm ID, 0.25µm Rtx®-200 (cat.# 15023)
1.0µL split injection of a basic drug mix (1mg/mL)

Conc.:	1000ng/µL
Oven temp.:	100°C to 325°C @ 4°C/min. (hold 10 min.)
Inj. / det. temp.:	250°C / 320°C
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 100°C
FID sensitivity:	1.28 x 10^{-10} AFS
Split ratio:	50:1

Blood Alcohol

Blood Alcohol

Resolution and analysis time are two critical factors to consider when developing an assay for ethanol and other volatiles in biological samples. We have developed two stationary phases specifically for blood alcohol analysis; each has the ability to baseline resolve all of the low molecular weight alcohols and their metabolites. Elution order of this analyte set differs on the two stationary phases, enabling screening and confirmation of volatile compounds to be performed with one injection. These columns also were designed for maximum sample throughput, with total analysis time under 3.5 minutes for an isothermal run. In addition to blood alcohol analysis, these columns can be used to test whole blood, serum, or urine for volatile compounds, using temperature programming.

Blood Alcohol
Rtx®-BAC1 & Rtx®-BAC2 (0.32mm ID Columns)

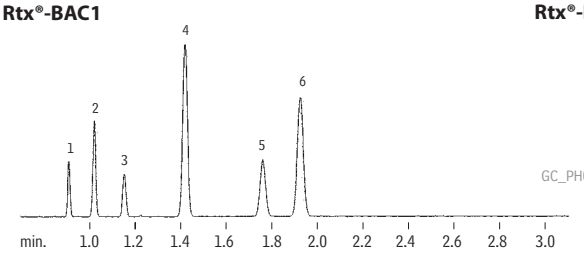
GC

	Conc. w/v
1. methanol	0.1%
2. acetaldehyde	0.2%
3. ethanol	0.2%
4. isopropanol	0.1%
5. acetone	0.01%
6. *n*-propanol	0.1%

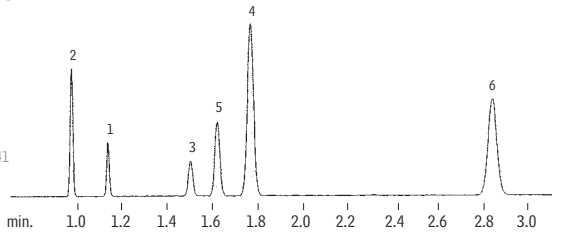
30m, 0.32mm ID, 1.8µm Rtx®-BAC1 (cat.# 18003)
30m, 0.32mm ID, 1.2µm Rtx®-BAC2 (cat.# 18002)
Dual-column analysis using a two-hole ferrule.
1.0mL headspace sample of a blood alcohol mix in water

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

Rtx®-BAC1



Rtx®-BAC2



GC_PH00241

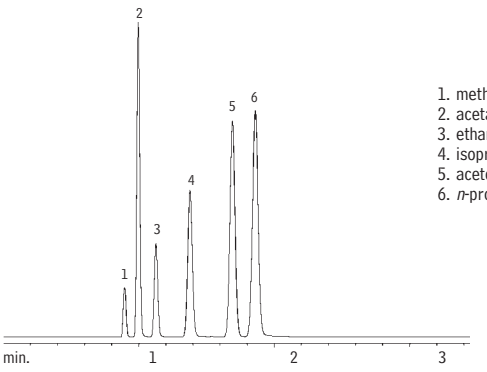
Blood Alcohol
Rtx®-BAC1 & Rtx®-BAC2 (0.53mm ID columns)

GC

30m, 0.53mm ID, 3.0µm Rtx®-BAC1 (cat.# 18001)
30m, 0.53mm ID, 2.0µm Rtx®-BAC2 (cat.# 18000)
1.0mL headspace sample of a blood alcohol mix in water

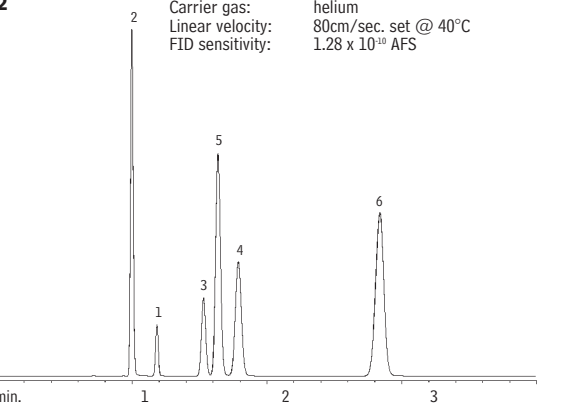
Sample conc.:	0.1% per compound
Oven temp.:	40°C
Inj. & det. temp.:	200°C
Carrier gas:	helium
Linear velocity:	80cm/sec. set @ 40°C
FID sensitivity:	1.28 x 10^{-10} AFS

Rtx®-BAC1



1. methanol
2. acetaldehyde
3. ethanol
4. isopropanol
5. acetone
6. *n*-propanol

Rtx®-BAC2

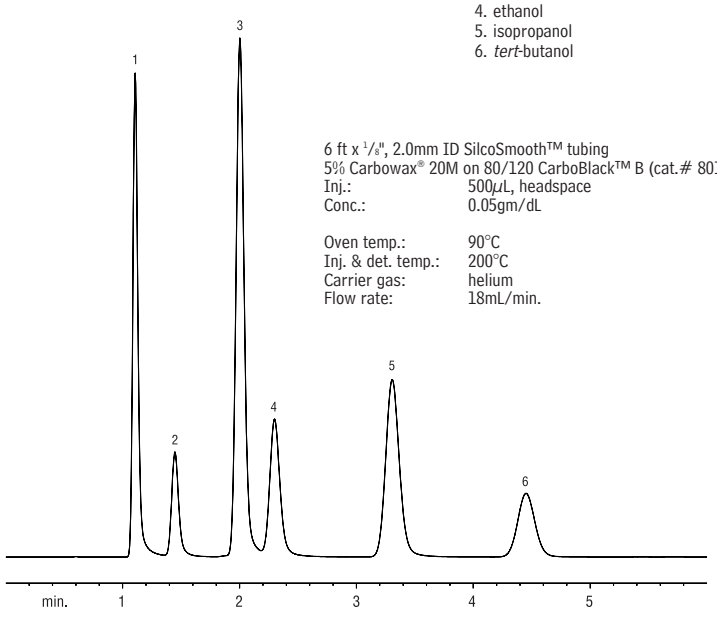


GC_PH00239

Blood Alcohol
5% Carbowax® 20M on 80/120 CarboBlack™ B

GC

1. acetaldehyde
2. methanol
3. acetone
4. ethanol
5. isopropanol
6. *tert*-butanol



GC_PH00478

6 ft x 1/8", 2.0mm ID SilcoSmooth™ tubing
5% Carbowax® 20M on 80/120 CarboBlack™ B (cat.# 80105)

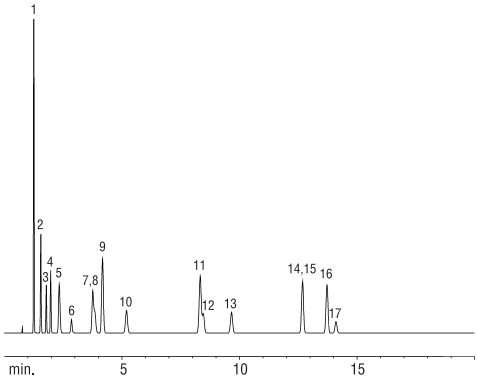
Inj.:	500µL, headspace
Conc.:	0.05gm/dL
Oven temp.:	90°C
Inj. & det. temp.:	200°C
Carrier gas:	helium
Flow rate:	18mL/min.

Solvents
Rtx®-BAC1 & Rtx®-BAC2

GC

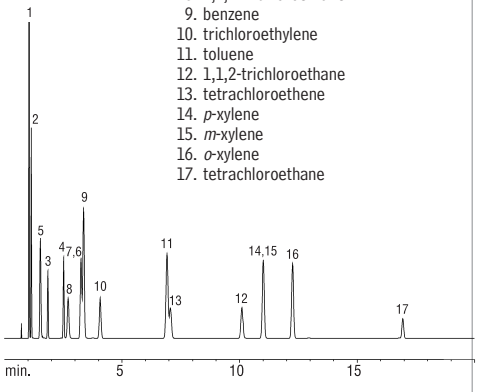
1. ethyl chloride
2. diethyl ether
3. methylene chloride
4. acetonitrile
5. methyl *tert*-butyl ether
6. chloroform
7. methyl ethyl ketone
8. 1,1,1-trichloroethane
9. benzene
10. trichloroethylene
11. toluene
12. 1,1,2-trichloroethane
13. tetrachloroethene
14. *p*-xylene
15. *m*-xylene
16. *o*-xylene
17. tetrachloroethane

Rtx®-BAC1



GC_PH00509

Rtx®-BAC2



GC_PH00510

30m, 0.53mm ID, 3.0µm Rtx®-BAC1 (cat.# 18001)
30m, 0.53mm ID, 2.0µm Rtx®-BAC2 (cat.# 18000)
1.0mL headspace sample

Oven temp.:	40°C (hold 5 min.) to 240°C @ 5°C/min.
Inj. & det. temp.:	240°C
Carrier gas:	helium
Linear velocity:	65cm/sec.

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.

free literature

Ethanol Analytical Reference Standards for Blood Alcohol Testing

lit. cat.# 59382

Dual-Column Confermation GC Analysis of Blood Alcohols Using Rtx®-BAC1 and Rtx®-BAC2 Columns Optimized for the Perkin Elmer HS 40 Headspace Autosampler

lit. cat.# 59598

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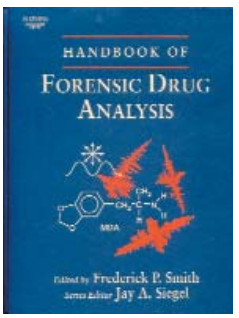
Cannabinoids; Opiates

Cannabinoids

Analysis of cannabinoids includes screening for parent compounds as well as metabolites of the active constituents in marijuana. Derivatization is commonly employed to help improve peak shape and resolution. Derivatization also can aid in the identification of specific cannabinoids by GC/MS by producing unique high mass ion fragments. Columns with low bleed and high thermal stability are required for trace level analysis. Low polarity columns, like the Rtx®-5 column, provide sufficient resolution for derivatized cannabinoids without extending the run time unnecessarily.

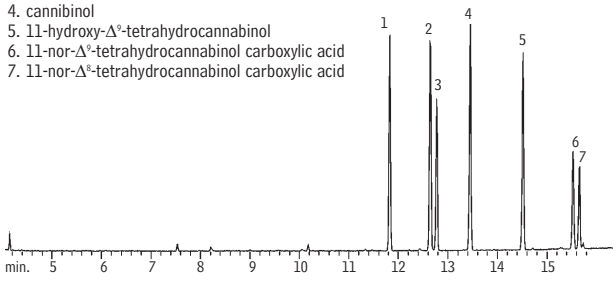


Handbook of Forensic Drug Analysis
cat.# 23055 (ea.)

Cannabinoids (TMS Derivatives) Rtx®-5

GC

1. cannabidiol
2. Δ^8-tetrahydrocannabinol
3. Δ^9-tetrahydrocannabinol
4. cannibinol
5. 11-hydroxy-Δ^9-tetrahydrocannabinol
6. 11-nor-Δ^9-tetrahydrocannabinol carboxylic acid
7. 11-nor-Δ^8-tetrahydrocannabinol carboxylic acid



GC_PH00253

15m, 0.25mm ID, 0.25µm Rtx®-5 (cat.# 10220)
1.0µL splitless injection of cannabinoids

Conc.:	100µg/mL
Oven temp.:	50°C (hold 0.5 min.) to 225°C @ 30°C/min., to 325°C @ 10°C/min.
Inj. temp.:	225°C
Interface temp.:	320°C
Det.:	MSD
Ionization:	EI
Carrier gas:	helium
Scan range:	40-500 AMU
Linear velocity:	40cm/sec. set @ 50°C
Splitless hold time:	0.75 min.

Opiates

Opiates are comprised of a multiple ring structure, substituted at various sites, producing compounds with different degrees of potency. When substitution is with a hydroxyl group, derivatization prior to analysis by GC is necessary to improve peak shape and response. Derivatization also can aid in the identification of opiates during GC/MS analysis by forming unique high mass ion fragments. An Rtx®-5 column can efficiently separate trimethylsilyl or fluoroacyl derivatives of the opiates.

free literature

Opiate Analysis

lit. cat.# 59576

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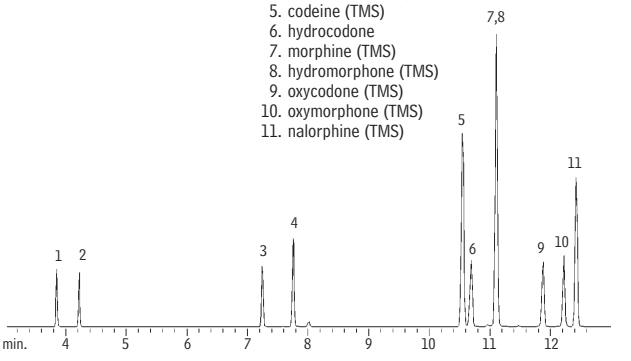
Opiates (TMS Derivatives) Rtx®-5

GC

1. meperidine
2. alphaprodine
3. methadone
4. levorphanol (TMS)
5. codeine (TMS)
6. hydrocodone
7. morphine (TMS)
8. hydromorphone (TMS)
9. oxycodone (TMS)
10. oxymorphone (TMS)
11. nalorphine (TMS)



GC_PH00233

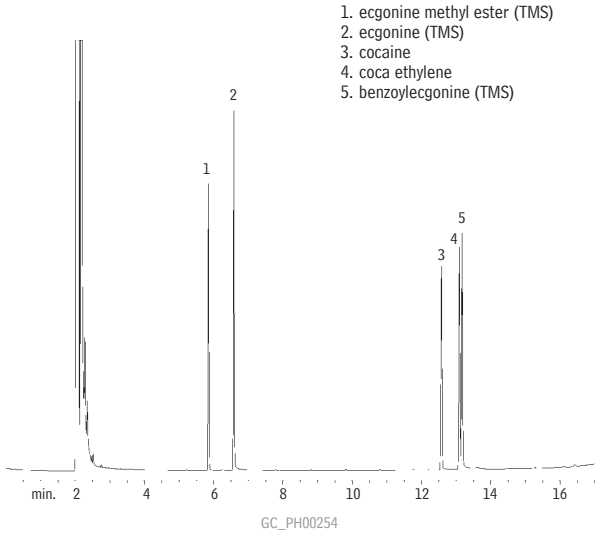
30m, 0.25mm ID, 0.25µm Rtx®-5 (cat.# 10223)
2.0µL split injection of opiates

Conc:	2000ng/µL
Oven temp.:	200°C to 325°C @ 7°C/min.
Inj. / det. temp.:	250°C / 300°C
Det. type:	MS
Ionization:	EI
Carrier gas:	helium
Mode:	full scan
Linear velocity:	30cm/sec. set @ 200°C
Split ratio:	50:1

Cocaine

Benzoylecgonine is the major metabolite found in urine after cocaine abuse. Monitoring for benzoylecgonine is required in the testing protocol established by the Department of Health and Human Services and the National Institute on Drug Abuse. Benzoylecgonine and other cocaine metabolites should be derivatized prior to analysis. Parent cocaine and its derivatized metabolites can be analyzed together on an Rtx®-5 column with good peak shape and resolution. The analysis also can be accomplished by HPLC.

Cocaine & Metabolites (TMS Derivatives) GC
Rtx®-5



30m, 0.25mm ID, 0.25µm Rtx®-5 (cat.# 10223)
1.0µL split injection of cocaine and cocaine metabolites

Oven temp.:	150°C to 320°C @ 10°C/min.
Inj. / det. temp.:	250°C / 300°C
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 50°C
FID sensitivity:	2.56 x 10^{-10} AFS
Split ratio:	30:1

Cocaine and Ecgonine Methyl Ester HPLC
Allure™ PFP Propyl

Peak List:
1. EME (ecgonine methyl ester)
2. COC (cocaine)

Sample:

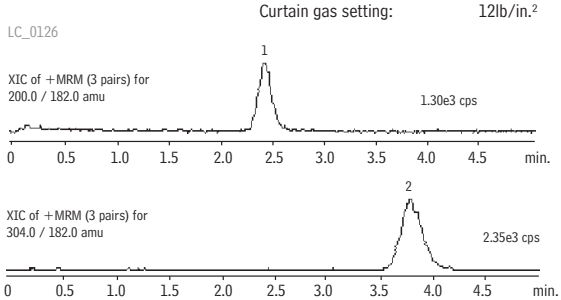
Inj.:	10µL
Conc.:	1µg/mL
Solvent:	water
Temp.:	4°C

Column: Allure™ PFP Propyl

Cat. #:	9169532
Dimensions:	30 x 2.1mm
Particle size:	5µm
Pore size:	60Å

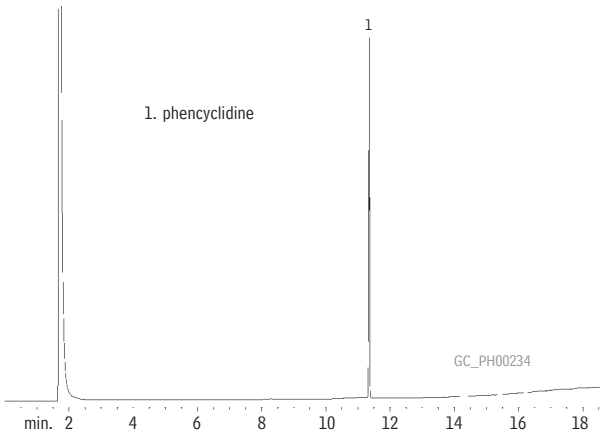
Conditions:

Mobile phase:	5mM ammonium formate, pH 3.0: acetonitrile (10:90, v/v)
Flow:	0.6mL/min.
Column temp.:	ambient
Det.:	PE/Sciex API 3000
Interface:	turbo ion spray, ESI
Interface temp.:	150°C
Ion mode:	positive
ESI probe voltage:	5000V
Orifice:	+ 71V
Ring:	+ 265V
Collision gas:	nitrogen
Collision gas pressure:	2.2 mTorr
Collision gas energy:	28 eV (COC) 26 eV (EME)
Electron multiplier:	2100 volts
Auxilary gas flow:	7000cc/min.
Nebulizer gas setting:	15lb/in.²
Curtain gas setting:	12lb/in.²



Data Courtesy of Shane Needham, Pfizer Inc.

Phencyclidine (PCP) GC
Rtx®-5



30m, 0.25mm ID, 0.25µm Rtx®-5 (cat.# 10223)
1.0µL split injection of phencyclidine

Conc.:	1000ng/µL
Oven temp.:	50°C (hold 1 min.) to 250°C @ 25°C/min., then to 325°C @ 10°C/min. (hold 2 min.)
Inj. / det. temp.:	250°C / 325°C
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 50°C
FID sensitivity:	2.56 x 10^{-10} AFS
Split ratio:	30:1

Phencyclidine

Phencyclidine can be screened for using immunoassay techniques. However, prior to reporting positive results, the presence of phencyclidine should be confirmed by an alternative chemical test. Phencyclidine easily can be chromatographed on phenylmethyl stationary phases like Rtx®-5. Low picogram amounts of the drug can be detected by NPD or GC/MS.

free literature

HPLC Column Selection Guide

lit. cat.# 59454B

Allure™ PFP Propyl and Ultra PFP Columns Provide Improved Analyses of Basic Compounds

lit. cat.# 59118A

Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

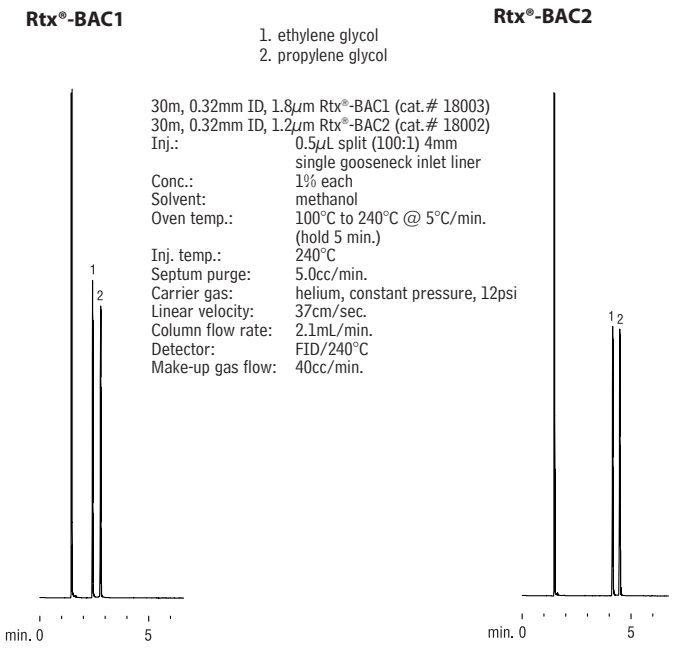
Glycols

Ethylene Glycol

Ethylene glycol, a major component of automotive antifreeze products, is a poison to humans and animals. It can be analyzed by GC on an Rtx®-BAC1, Rtx®-BAC2, or Stabilwax® column.

GC

Glycols
Rtx®-BAC1 & Rtx®-BAC2



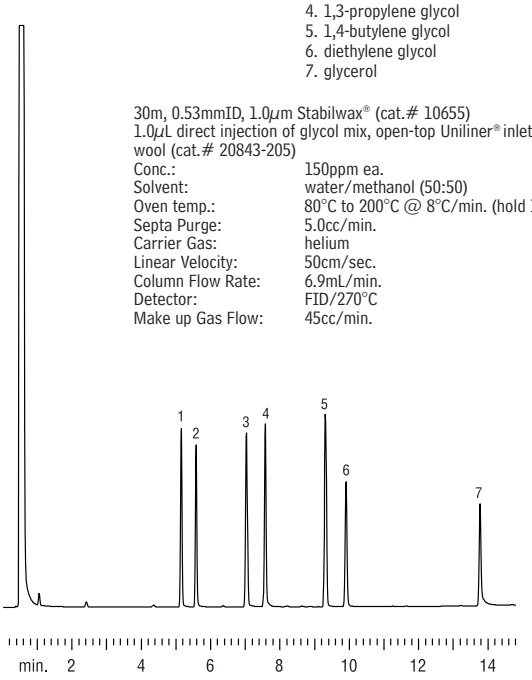
30m, 0.32mm ID, 1.8µm Rtx®-BAC1 (cat.# 18003)
30m, 0.32mm ID, 1.2µm Rtx®-BAC2 (cat.# 18002)

Inj.:	0.5µL split (100:1) 4mm single gooseneck inlet liner
Conc.:	1% each
Solvent:	methanol
Oven temp.:	100°C to 240°C @ 5°C/min. (hold 5 min.)
Inj. temp.:	240°C
Septum purge:	5.0cc/min.
Carrier gas:	helium, constant pressure, 12psi
Linear velocity:	37cm/sec.
Column flow rate:	2.1mL/min.
Detector:	FID/240°C
Make-up gas flow:	40cc/min.

GC_EV00474

Glycols
Stabilwax®

1. 1,2-propylene glycol
2. ethylene glycol
3. 1,3-butylene glycol
4. 1,3-propylene glycol
5. 1,4-butylene glycol
6. diethylene glycol
7. glycerol



30m, 0.53mmID, 1.0µm Stabilwax® (cat.# 10655)
1.0µL direct injection of glycol mix, open-top Uniliner® inlet liner without wool (cat.# 20843-205)

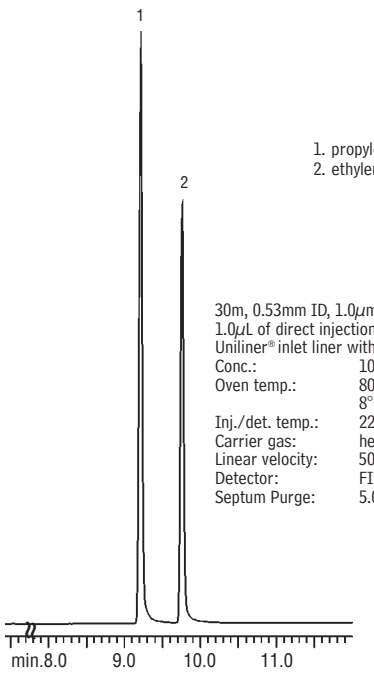
Conc.:	150ppm ea.
Solvent:	water/methanol (50:50)
Oven temp.:	80°C to 200°C @ 8°C/min. (hold 10 min.)
Septa Purge:	5.0cc/min.
Carrier Gas:	helium
Linear Velocity:	50cm/sec.
Column Flow Rate:	6.9mL/min.
Detector:	FID/270°C
Make up Gas Flow:	45cc/min.

GC_EV00476

GC

Glycols
Stabilwax®

1. propylene glycol
2. ethylene glycol



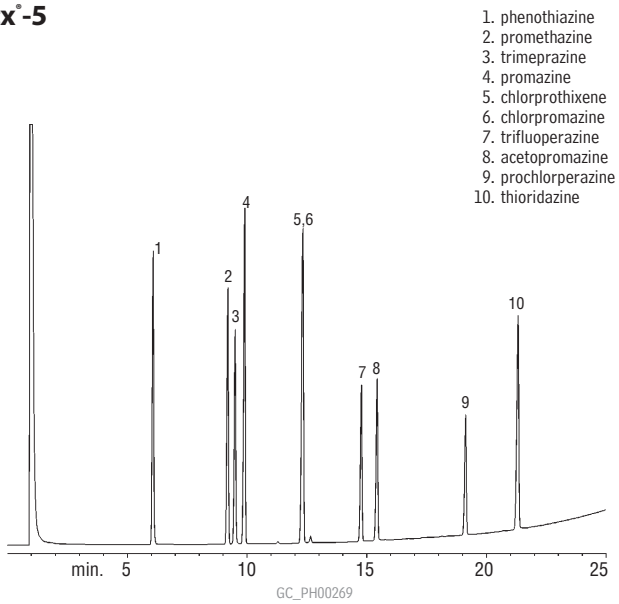
30m, 0.53mm ID, 1.0µm Stabilwax® (cat.# 10655)
1.0µL of direct injection of glycols in water. Open-top Uniliner® inlet liner without wool (cat.# 20843-205).

Conc.:	100ppm
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
Septum Purge:	5.0cc/min.

GC_EV00546

Phenothiazines (Underivatized) Rtx®-5

GC



15m, 0.32mm ID, 0.50µm Rtx®-5 (cat.# 10236)
1.0µL split injection of phenothiazines
Conc.: 2000ng/µL

Oven temp.: 200°C to 325°C @ 5°C/min.
Inj. / det. temp.: 250°C / 315°C
Carrier gas: helium
Linear velocity: 25cm/sec. set @ 200°C
FID sensitivity: 2.56 x 10^{-10} AFS
Split ratio: 30:1

Phenothiazines

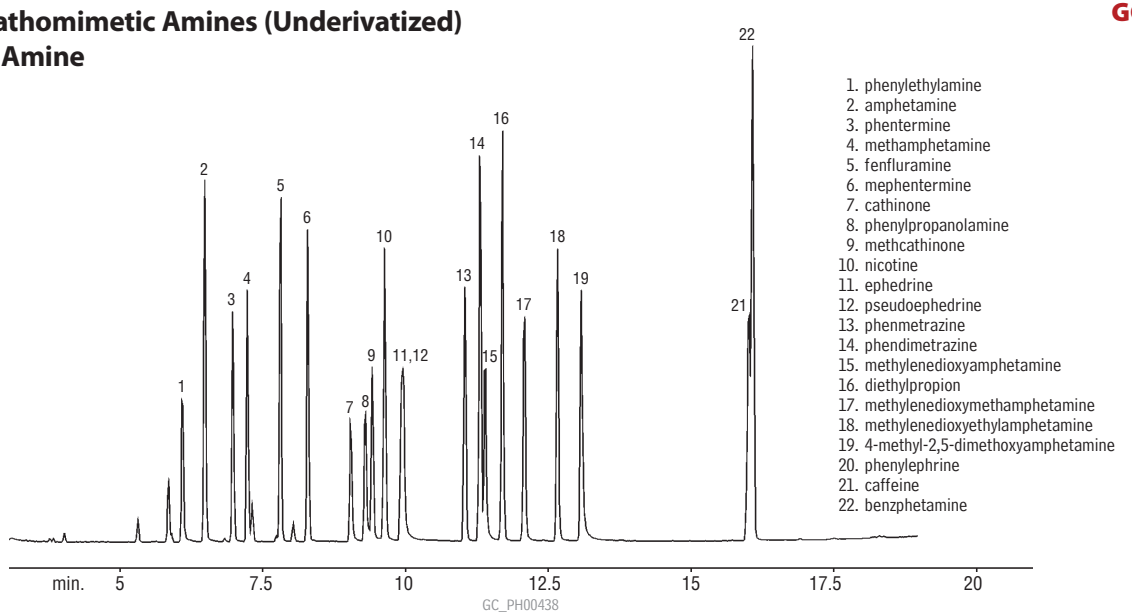
Phenothiazines are high molecular weight compounds based on a three ring structure. When analyzing phenothiazines by gas chromatography, retention times are typically long with elution temperatures at or near the maximum operating temperature of the column. Using shorter columns will help to reduce the effective elution temperature and overall analysis time.

Sympathomimetic Amines

Sympathomimetic amine drugs include both controlled and non-controlled substances based on phenylethylamine, with various functional groups substituted at key positions on the molecule. Active hydrogens on the amine portion of the molecule necessitate derivatization to improve peak shape and response. Acylating reagents have been used to produce less polar and less reactive compounds that chromatograph well on low polarity stationary phases like Rtx®-5. A carefully chosen derivatizing reagent can produce differences in relative retention time for specific compounds and help to resolve coelutions. Identification by GC/MS can be improved by using a reagent that produces unique high mass ion fragments. Deactivated vials, glassware and inlet liners should be used to maintain consistent recovery during sample preparation and analysis.

Sympathomimetic Amines (Underivatized) Rtx®-5 Amine

GC



30m, 0.25mm ID, 0.50µm Rtx®-5 Amine (cat.# 12338)
split mode, split vent flow rate 45mL/min.
Oven temp.: 100°C to 310°C @ 10°C/min.

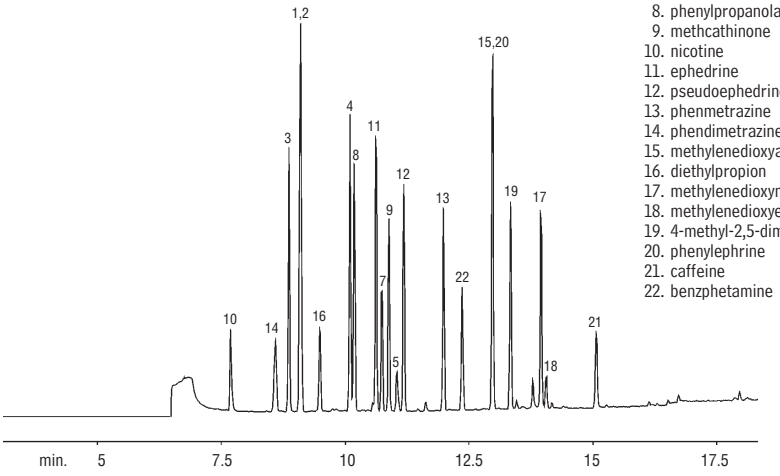
Varian 3400 GC coupled with Varian Saturn 2000 MS detector
Mass spectroscopy data collected using a scan range of 40 amu through 600 amu.
Ionization performed in the EI Auto mode.

CLINICAL/FORENSICS

GC

Sympathomimetic Amines (Derivatized)
Rtx®-200

1. phenylethylamine
2. amphetamine
3. phentermine
4. methamphetamine
5. fenfluramine
6. mephentermine
7. cathinone
8. phenylpropanolamine
9. methcathinone
10. nicotine
11. ephedrine
12. pseudoephedrine
13. phenmetrazine
14. phendimetrazine
15. methylenedioxyamphetamine
16. diethylpropion
17. methylenedioxymethamphetamine
18. methylenedioxyethylamphetamine
19. 4-methyl-2,5-dimethoxyamphetamine
20. phenylephrine
21. caffeine
22. benzphetamine



GC_PH00439

30m, 0.25mm ID, 0.50µm Rtx®-200 (cat.# 15038)
split mode, split vent flow rate 45mL/min.
Oven temp.: 100°C to 310°C @ 10°C/min.

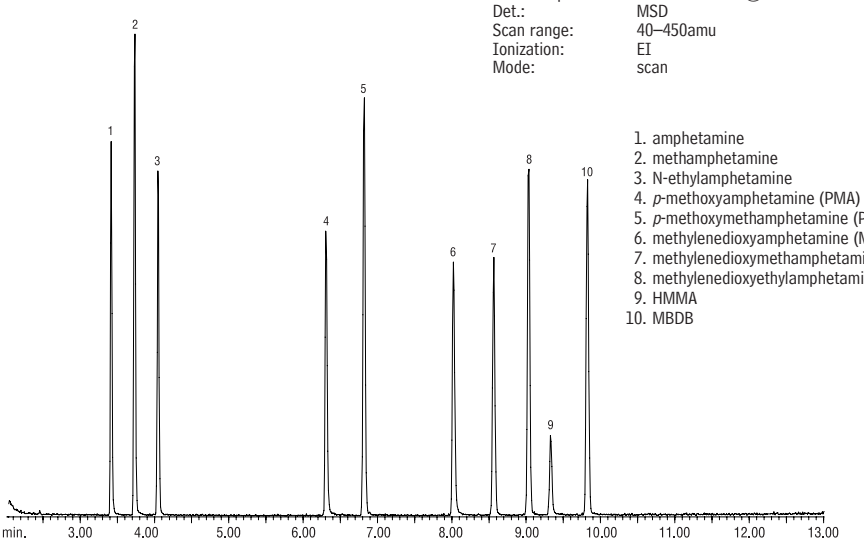
Varian 3400 GC coupled with Varian Saturn 2000 MS detector
Mass spectroscopy data collected using a scan range of 40 amu through 600 amu.
Ionization performed in the EI Auto mode.

Sympathomimetic Amines (Underivatized)
Rtx®-35 Amine

Rtx®-35 Amine 30m, 0.25mm ID, 0.50µm (cat.# 11338)
1.0µL split injection of underivatized sympathomimetic amines

Conc.	1000ng/µL
Inj. temp:	250°C
Carrier gas:	helium
Linear velocity:	30cm/sec.
Oven temp.:	150°C to 240°C @ 7°C/min.
Det.:	MSD
Scan range:	40–450amu
Ionization:	EI
Mode:	scan

1. amphetamine
2. methamphetamine
3. N-ethylamphetamine
4. *p*-methoxyamphetamine (PMA)
5. *p*-methoxymethamphetamine (PMMA)
6. methylenedioxyamphetamine (MDA)
7. methylenedioxymethamphetamine (MDMA)
8. methylenedioxyethylamphetamine (MDEA)
9. HMMA
10. MBDB

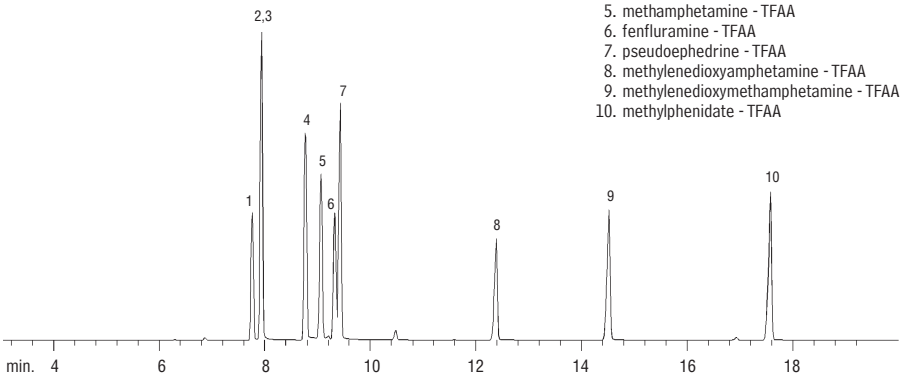


GC_PH00574

GC

Sympathomimetic Amines (TFAA Derivatives)
Rtx®-5

1. amphetamine - TFAA
2. phentermine - TFAA
3. phenylpropanolamine - TFAA
4. ephedrine - TFAA
5. methamphetamine - TFAA
6. fenfluramine - TFAA
7. pseudoephedrine - TFAA
8. methylenedioxyamphetamine - TFAA
9. methylenedioxymethamphetamine - TFAA
10. methylphenidate - TFAA



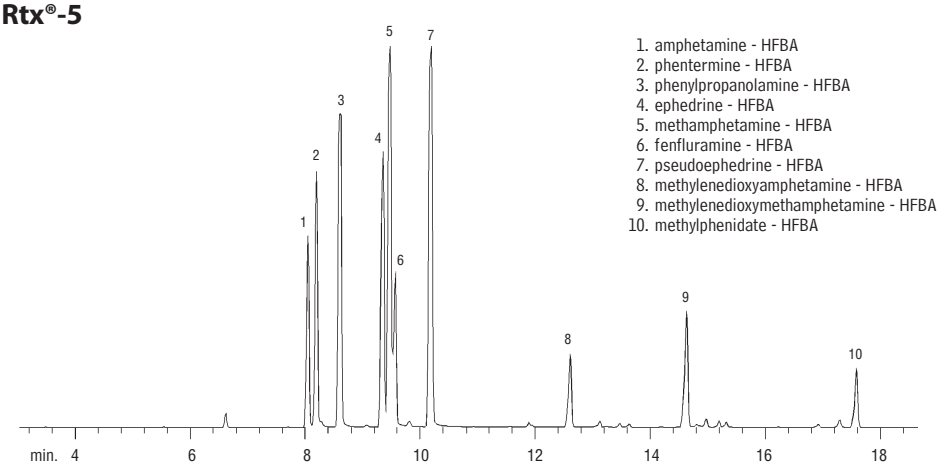
GC_PH00251

30m, 0.25mm ID, 0.25µm Rtx®-5 (cat.# 10223)
1.0µL splitless injection of derivatized sympathomimetic amines

Conc.:	approximately 2.5ng/µL
Oven temp.:	40°C (hold 1 min.) to 150°C @ 30°C/min., to 300°C @ 5°C/min.
Inj. temp.:	225°C
Interface temp.:	275°C
Det.:	MSD
Ionization:	EI
Carrier gas:	helium
Linear velocity:	20cm/sec. set @ 100°C
Splitless hold time:	1 min.

Sympathomimetic Amines (HFBA Derivatives)
Rtx®-5

1. amphetamine - HFBA
2. phentermine - HFBA
3. phenylpropanolamine - HFBA
4. ephedrine - HFBA
5. methamphetamine - HFBA
6. fenfluramine - HFBA
7. pseudoephedrine - HFBA
8. methylenedioxyamphetamine - HFBA
9. methylenedioxymethamphetamine - HFBA
10. methylphenidate - HFBA



GC_PH00252

30m, 0.25mm ID, 0.25µm Rtx®-5 (cat.# 10223)
1.0µL splitless injection of sympathomimetic amines

Conc.:	approximately 2.5ng/µL.
Oven temp.:	40°C (hold 1 min.) to 150°C @ 30°C/min., to 300°C @ 5°C/min.
Inj. temp.:	225°C
Interface temp.:	275°C
Det.:	MSD
Ionization:	EI
Carrier gas:	helium
Linear velocity:	20cm/sec. set @ 100°C
Splitless hold time:	1 min.

free literature

GC Column Installation

lit. cat.# 59668A

USP Column Cross-Reference Chart

lit. cat.# 59253

Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

Analgesic Formulations

Analgesics

Popular components for multi-active analgesic formulations include aspirin, salicylic acid, salicylamide, acetaminophen, ibuprofen, naproxen, guaifenesin, codeine, oxycodone, hydrocodone, and caffeine. Many of these compounds can be analyzed simultaneously using high performance liquid chromatography (HPLC), thereby improving laboratory efficiency and productivity. With the selection of the proper LC phase, separation becomes a simple and manageable task that does not rely upon extensive preparation procedures or use of ion pairing agents, which often are described in pharmaceutical compenda.

Ultra C18, Ultra Phenyl, and Allure™ Basix HPLC column phases separate mixtures of these pharmaceuticals in a productive and cost effective manner. The selective chemistries of these phases provide powerful separation mechanisms.

free literature

Improved HPLC Analysis of Analgesics

lit. cat.# 59511A

Genuine Restek Replacement Parts for HPLC Systems

lit. cat.# 59012A

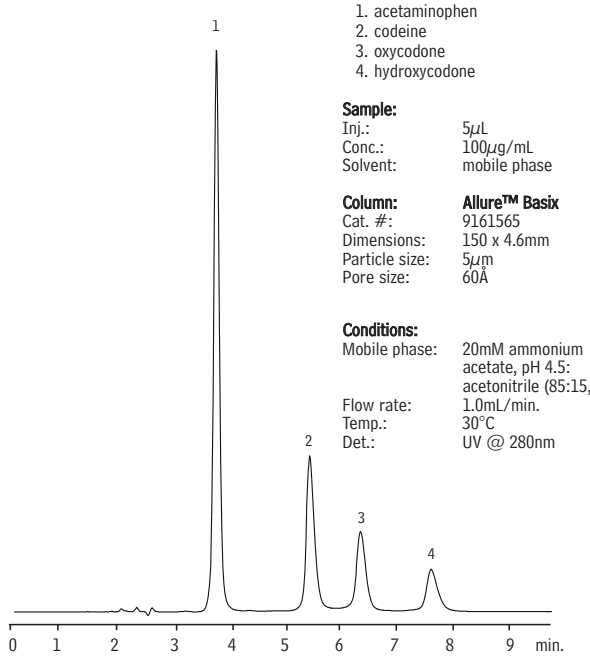
HPLC Tech Tips Wall Chart

lit. cat.# 59894A

Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

Acetaminophen and Narcotic Analgesics
Allure™ Basix

HPLC



LC_0082

Peak List:
1. acetaminophen
2. codeine
3. oxycodone
4. hydroxycodone

Sample:
Inj.: 5µL
Conc.: 100µg/mL
Solvent: mobile phase

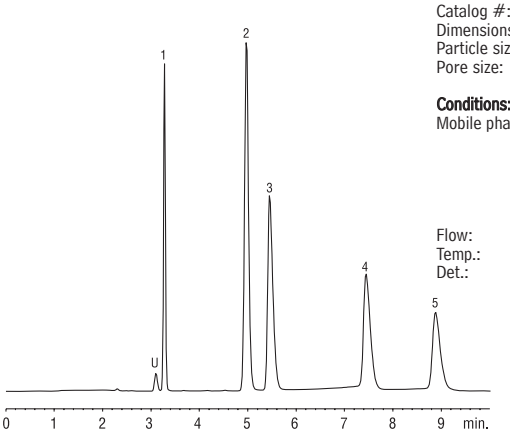
Column: **Allure™ Basix**
Cat. #: 9161565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 60Å

Conditions:
Mobile phase: 20mM ammonium acetate, pH 4.5: acetonitrile (85:15, v/v)
Flow rate: 1.0mL/min.
Temp.: 30°C
Det.: UV @ 280nm

Acetaminophen and Narcotic Analgesics
Ultra C18

HPLC

Peak List:	Conc. (µg/mL)	Ret. Time (min.)	Tailing	Resolution
U. unknown	unknown	3.1	NA	NA
1. morphine sulfate	204	3.3	1.0	1.8
2. acetaminophen	92	5.0	1.1	14.1
3. codeine phosphate	216	5.5	1.4	2.6
4. oxycodone HCl	206	7.5	1.4	8.5
5. hydrocodone bitartrate	218	8.9	1.4	5.0



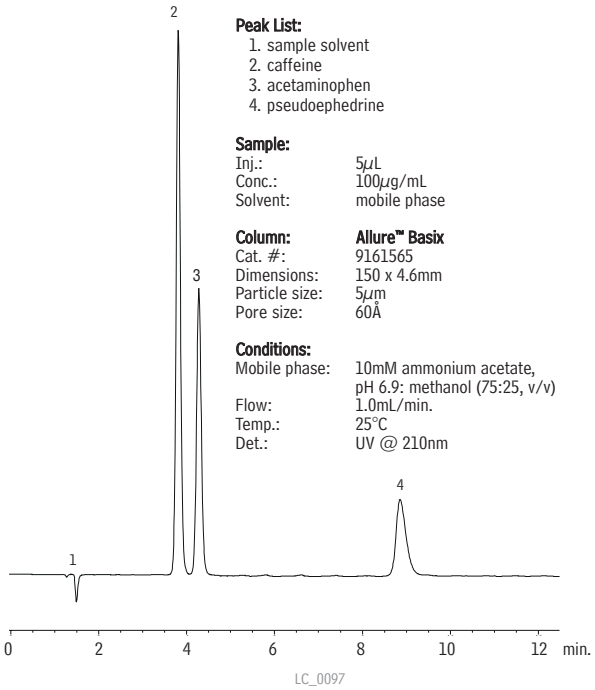
LC_0218

Sample:
Inj.: 4.0µL
Sample: raw material mix
Solvent: mobile phase

Column: **Ultra C18**
Catalog #: 9174575
Dimensions: 250 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:
Mobile phase: A: 10mm potassium phosphate, pH 2.8
B: acetonitrile: methanol (90:10 v/v)
(85A:15B, v/v)
Flow: 1.0mL/min.
Temp.: 27°C
Det.: UV @ 235nm

Acetaminophen (Analgesic), Pseudoephedrine (Decongestant), and Caffeine (Stimulant)
Allure™ Basix



Peak List:
1. sample solvent
2. caffeine
3. acetaminophen
4. pseudoephedrine

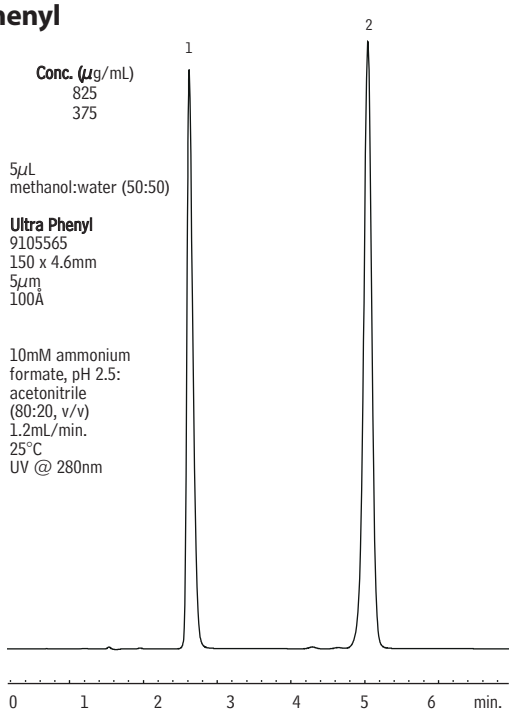
Sample:
Inj.: 5µL
Conc.: 100µg/mL
Solvent: mobile phase

Column: **Allure™ Basix**
Cat. #: 9161565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 60Å

Conditions:
Mobile phase: 10mM ammonium acetate, pH 6.9: methanol (75:25, v/v)
Flow: 1.0mL/min.
Temp.: 25°C
Det.: UV @ 210nm

Guaifenesin (Expectorant/Antitussive) and Codeine (Narcotic Analgesic)
Ultra Phenyl

HPLC



Peak List: **Conc. (µg/mL)**
1. codeine 825
2. guaifenesin 375

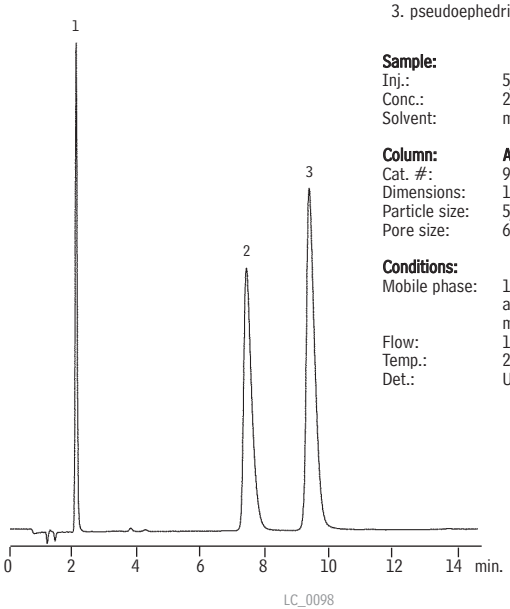
Sample:
Inj.: 5µL
Solvent: methanol:water (50:50)

Column: **Ultra Phenyl**
Cat. #: 9105565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:
Mobile phase: 10mM ammonium formate, pH 2.5: acetonitrile (80:20, v/v)
Flow rate: 1.2mL/min.
Temp.: 25°C
Det.: UV @ 280nm

Ibuprofen (Analgesic) and Pseudoephedrine (Decongestant)
Allure™ Basix

HPLC



Peak List:
1. uracil
2. ibuprofen
3. pseudoephedrine

Sample:
Inj.: 5µL
Conc.: 200µg/mL
Solvent: mobile phase

Column: **Allure™ Basix**
Cat. #: 9161565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 60Å

Conditions:
Mobile phase: 10mM ammonium acetate, pH 6.9: methanol (75:25, v/v)
Flow: 1.0mL/min.
Temp.: 25°C
Det.: UV @ 210nm

free literature

HPLC Columns & Accessories (catalog)

lit. cat.# 59241B

Analysis of Narcotics & Narcotic/Acetaminophen Admixtures: What to do When Compendium Methods Don't Work

lit. cat.# 59453

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Antidepressants (Tricyclic)

Tricyclic Antidepressants

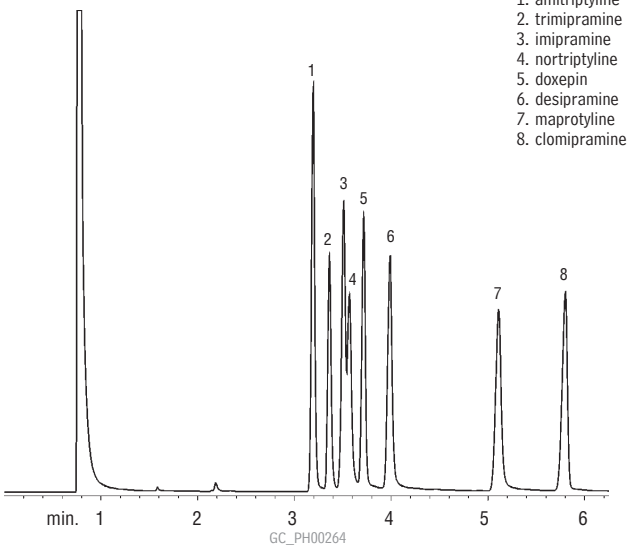
Antidepressants are similar to phenothiazines in structure except that the center ring is seven membered instead of six, and does not contain sulfur. Short GC columns operated at elevated temperatures produce the best separations in the shortest time. Rtx®-1701 columns provide a unique selectivity for the antidepressants, performing the separation isothermally in less than six minutes.

Tricylic antidepressants also are rapidly separated on an HPLC column specially developed for basic compounds: Allure™ Basix.

Antidepressants (Basic Drugs)
Rtx®-1701

GC

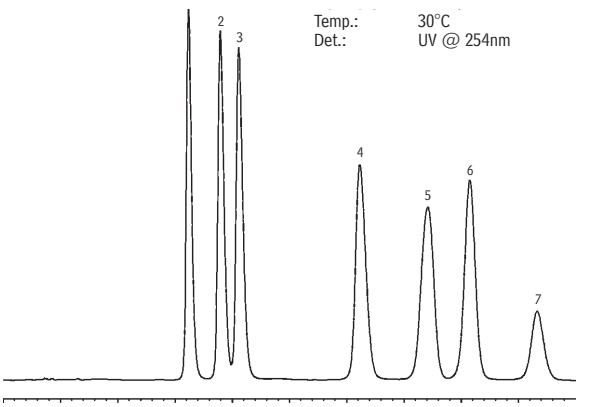
1. amitriptyline
2. trimipramine
3. imipramine
4. nortriptyline
5. doxepin
6. desipramine
7. maprotyline
8. clomipramine



15m, 0.25mm ID, 0.25µm Rtx®-1701 (cat.# 12020)
1.0µL split injection of antidepressants

Conc.:	25ng/component
Oven temp.:	225°C
Inj. / det. temp.:	250°C / 260°C
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 225°C
FID sensitivity:	2.56 x 10^{-10} AFS
Split ratio:	40:1

HPLC



Antidepressants (Basic Drugs)
Allure™ Basix (LC/MS)

HPLC

Peak List:
1. desipramine
2. trimipramine

Sample:

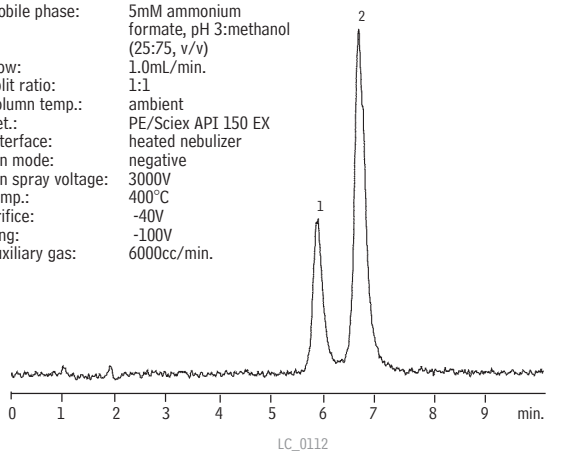
Inj.:	10µL
Conc.:	10µg/mL
Solvent:	water:methanol (1:1, v/v)

Column: **Allure™ Basix**

Cat. #:	9161565
Dimensions:	150 x 4.6mm
Particle size:	5µm
Pore size:	60Å

Conditions:

Mobile phase:	5mM ammonium formate, pH 3:methanol (25:75, v/v)
Flow:	1.0mL/min.
Split ratio:	1:1
Column temp.:	ambient
Det.:	PE/Sciex API 150 EX
Interface:	heated nebulizer
Ion mode:	negative
Ion spray voltage:	3000V
Temp.:	400°C
Orifice:	-40V
Ring:	-100V
Auxiliary gas:	6000cc/min.



Antidepressants
Ultra IBD

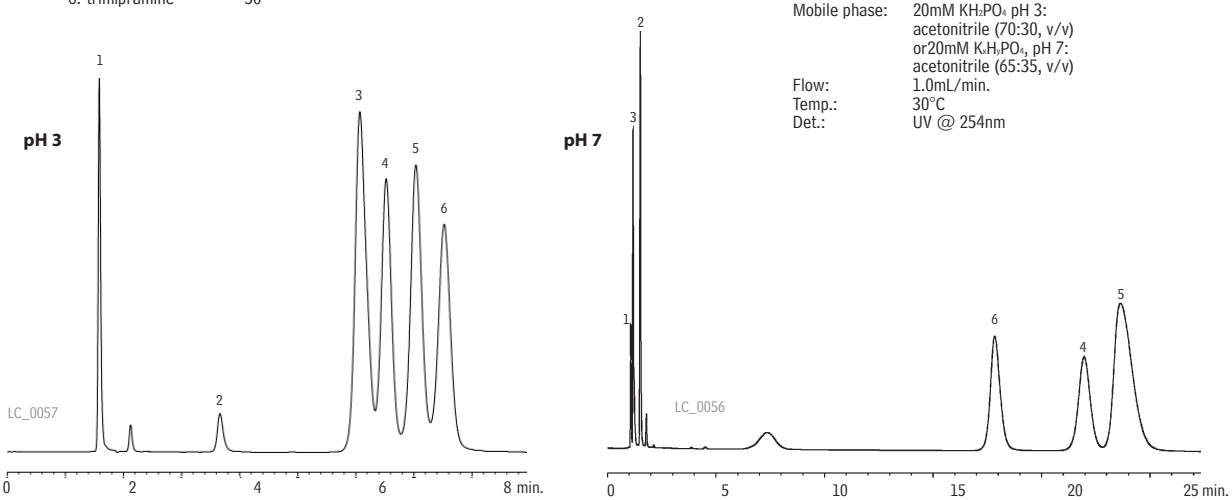
HPLC

Peak List:	Conc. (µg/mL)
1. uracil	5
2. maleate	5
3. benzoic acid	50
4. nortriptyline	50
5. amitriptyline	50
6. trimipramine	50

Sample:
Inj.: 10µL
Solvent: mobile phase

Column: Ultra IBD
Cat. #: 9175565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:
Mobile phase: 20mM KH_2PO_4 pH 3: acetonitrile (70:30, v/v) or20mM $K_2H_2PO_4$, pH 7: acetonitrile (65:35, v/v)
Flow: 1.0mL/min.
Temp.: 30°C
Det.: UV @ 254nm



Anithistamines

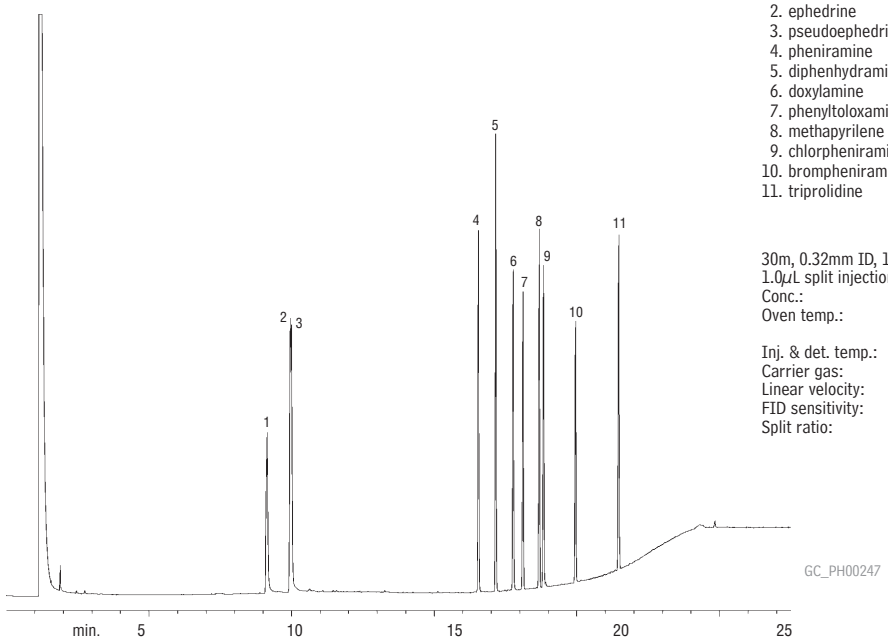
Antihistamines and decongestants are common ingredients in over-the-counter cold medications. Many include amine functional groups and are classified as basic compounds. Our Rtx®-5 Amine column is specially designed for basic compounds: antihistamines and other compounds that tail or are adsorbed on poorly deactivated columns exhibit excellent peak shape and better response on an Rtx®-5 Amine column.

Antihistamines (Underivatized)
Rtx®-5 Amine

GC

1. phenylpropanolamine
2. ephedrine
3. pseudoephedrine
4. pheniramine
5. diphenhydramine
6. doxylamine
7. phenyltoloxamine
8. methapyrilene
9. chlorpheniramine
10. brompheniramine
11. triprolidine

30m, 0.32mm ID, 1.0µm Rtx®-5 Amine (cat.# 12354)
1.0µL split injection of antihistamines.
Conc.: 1000ng/µL
Oven temp.: 130°C (hold 5 min.) to 305°C @ 10°C/min. (hold 5 min.)
Inj. & det. temp.: 305°C
Carrier gas: hydrogen
Linear velocity: 43cm/sec. set @ 130°C
FID sensitivity: 6.4 x 10^{-11} AFS
Split ratio: 50:1



Antiepileptics

Laboratories that perform therapeutic drug monitoring tests analyze antiepilileptic (anti-convulsant) drugs using either GC or HPLC because immunoassays do not exhibit linearity and show cross reactivities in the toxic range. Common antiepileptics are resolved using an Rtx®-20 or Rtx®-1701 GC column.

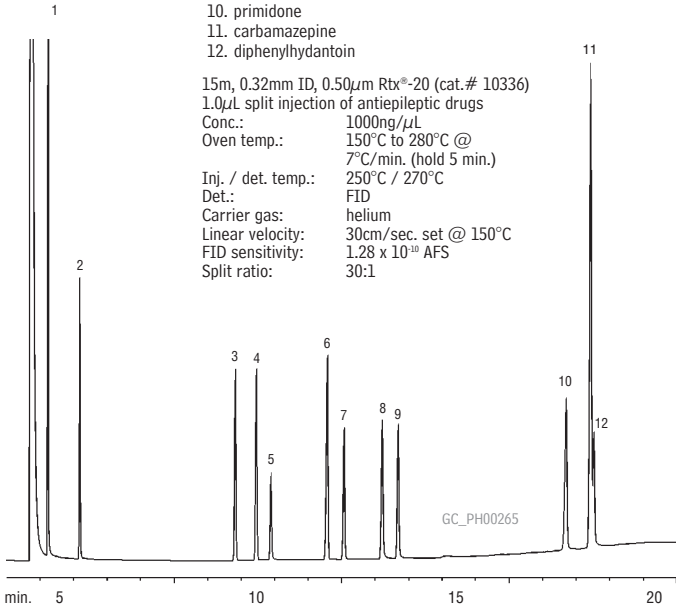
Antiepileptics (Underivatized) Rtx®-20

GC

1. valproic acid
2. ethosuximide
3. methsuximide
4. phensuximide
5. desmethyl methsuximide
6. mephenytoin
7. ethotoin
8. PEMA
9. phenobarbital
10. primidone
11. carbamazepine
12. diphenylhydantoin

15m, 0.32mm ID, 0.50µm Rtx®-20 (cat.# 10336)
1.0µL split injection of antiepileptic drugs

Conc.:	1000ng/µL
Oven temp.:	150°C to 280°C @ 7°C/min. (hold 5 min.)
Inj. / det. temp.:	250°C / 270°C
Det.:	FID
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 150°C
FID sensitivity:	1.28 x 10^{-10} AFS
Split ratio:	30:1



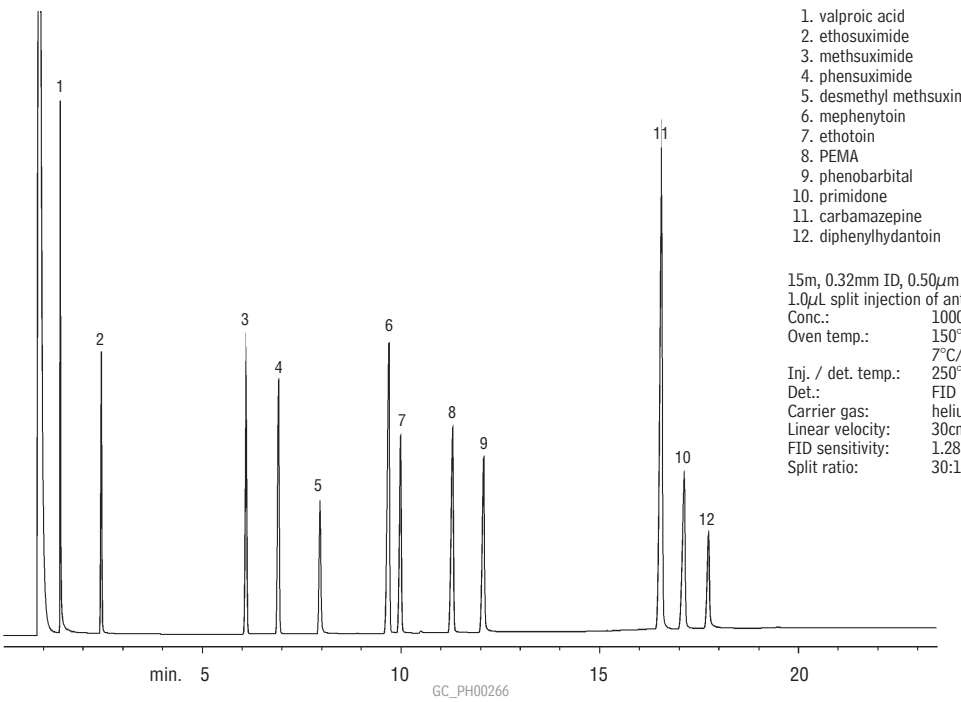
Antiepileptics (Underivatized) Rtx®-1701

GC

1. valproic acid
2. ethosuximide
3. methsuximide
4. phensuximide
5. desmethyl methsuximide
6. mephenytoin
7. ethotoin
8. PEMA
9. phenobarbital
10. primidone
11. carbamazepine
12. diphenylhydantoin

15m, 0.32mm ID, 0.50µm Rtx®-1701 (cat.# 12036)
1.0µL split injection of antiepileptic drugs

Conc.:	1000ng/µL
Oven temp.:	150°C to 280°C @ 7°C/min. (hold 5 min.)
Inj. / det. temp.:	250°C / 270°C
Det.:	FID
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 150°C
FID sensitivity:	1.28 x 10^{-10} AFS
Split ratio:	30:1



Atenolol (Antiarrhythmic) Allure™ Basix

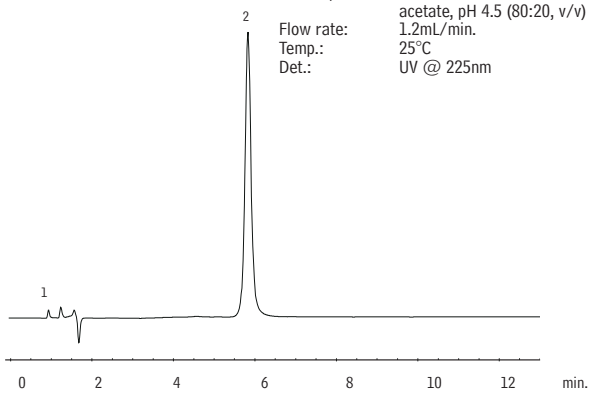
HPLC

Peak List:
1. uracil (marker)
2. atenolol

Sample:
Inj.: 1µL
Conc.: 1mg/mL
Solvent: water:methanol (7:3, v/v)

Column: Allure™ Basix
Cat. #: 9161565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 60Å

Conditions:
Mobile phase: acetonitrile:20mM ammonium acetate, pH 4.5 (80:20, v/v)
Flow rate: 1.2mL/min.
Temp.: 25°C
Det.: UV @ 225nm



LC_0072

Triamterene and Hydrochlorothiazide (Antiarrhythmics) Allure™ Basix

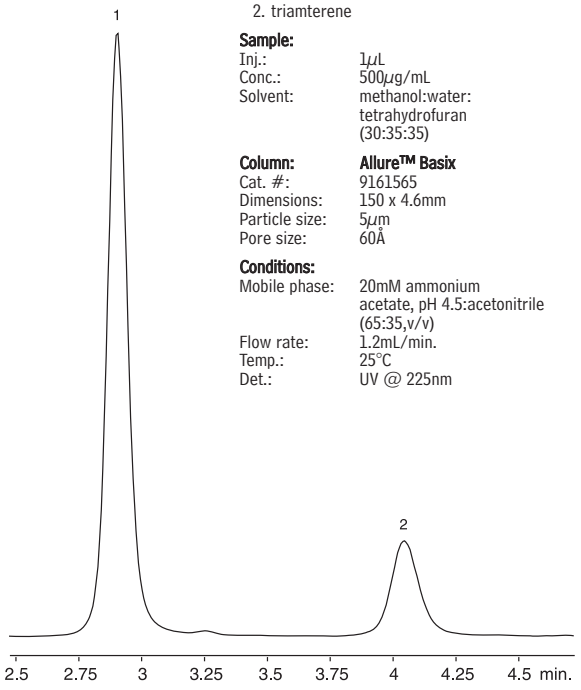
HPLC

Peak List:
1. hydrochlorothiazide
2. triamterene

Sample:
Inj.: 1µL
Conc.: 500µg/mL
Solvent: methanol:water: tetrahydrofuran (30:35:35)

Column: Allure™ Basix
Cat. #: 9161565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 60Å

Conditions:
Mobile phase: 20mM ammonium acetate, pH 4.5:acetonitrile (65:35,v/v)
Flow rate: 1.2mL/min.
Temp.: 25°C
Det.: UV @ 225nm



LC_0086

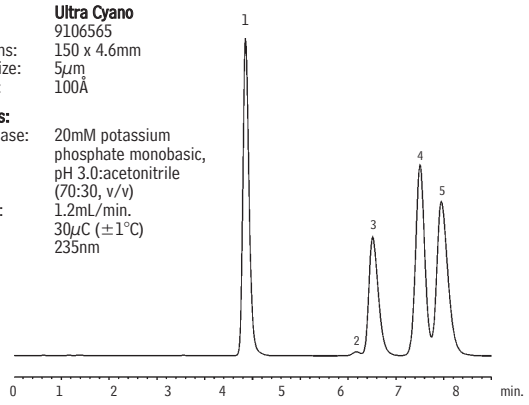
Calcium Channel Blockers (Antiarrhythmics) Ultra Cyano

Peak List:
1. diltiazem
2. nifedipine impurity
3. verapamil
4. nifedipine
5. nicardipine

Sample:
Inj.: 5µL
Conc.: 100mg/mL
Solvent: acetonitrile:water (1:1)

Column: Ultra Cyano
Cat. #: 9106565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:
Mobile phase: 20mM potassium phosphate monobasic, pH 3.0:acetonitrile (70:30, v/v)
Flow rate: 1.2mL/min.
Temp.: 30µC (±1°C)
Det.: 235nm



LC_0062

Digitalis Extracts/Derivatives Ultra PFP Propyl Cartridge Column (Fast LC)

HPLC

Peak List:	Conc.	Ret. Time (min.)
1. digoxigenin	100µg/mL	0.40
2. gitoxigenin	100µg/mL	0.80
3. digoxin	100µg/mL	1.10
4. gitoxin	~10µg/mL	2.20
5. digitoxin	100µg/mL	2.60

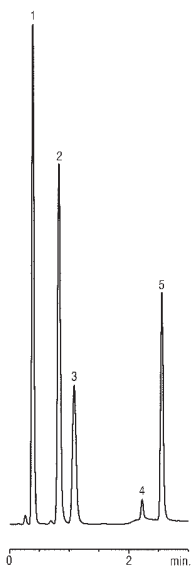
Sample:
Inj.: 10µL
Solvent: water:acetonitrile (80:20 v/v)

Column: Ultra PFP Propyl (Custom)
Dimensions: 30 x 4.0mm
Particle size: 3µm
Pore size: 100Å

Conditions:
Mobile phase: A: water
B: acetonitrile

Time (min.)	%B
0.0	20
1.5	20
1.51	35
3.0	35
3.1	20

Flow: 2.0mL/min.
Temp.: 27°C
Det.: UV @ 230nm



LC_0250

Cardiac Medications; CNS Depressants (Sedatives)

Cardiac Medications

Several classes of medications are used to decrease high blood pressure, control arrhythmias (abnormal heart rhythms), and treat congestive heart failure. These medications include beta antagonists, ACE inhibitors, diuretics, and calcium channel blockers. HPLC is the preferred technique for analyzing many of these compounds. Selecting the appropriate analytical column is critical, because many of the basic compounds tail badly on poorly deactivated HPLC phases. Restek's fully end-capped Allure™ Basix, Allure™ PFP Propyl, Ultra PFP, and Ultra Cyano phases can use the basic nature of these compounds to achieve separation without peak tailing.

free literature

Analyzing Cardiac Medications by HPLC

lit. cat.# 59151

Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

HPLC

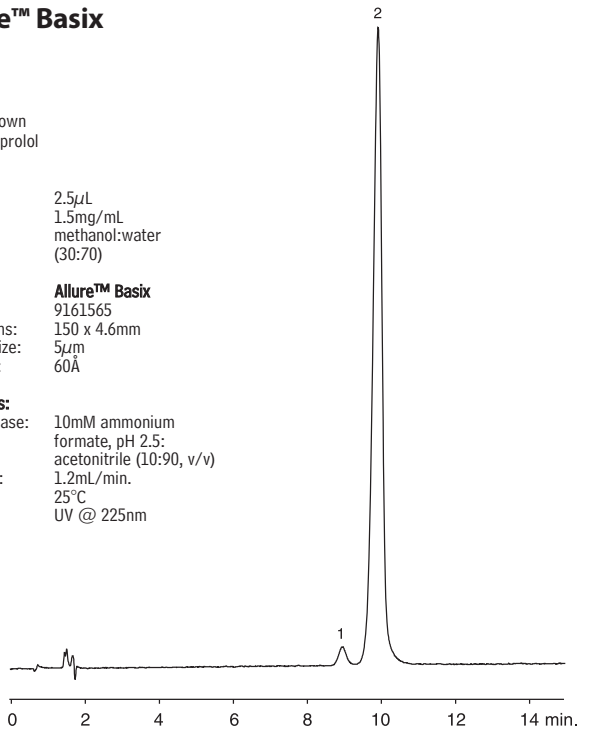
Metoprolol (Antiarrhythmic)
Allure™ Basix

Peak List:
1. unknown
2. metoprolol

Sample:
Inj.: 2.5µL
Conc.: 1.5mg/mL
Solvent: methanol:water (30:70)

Column: **Allure™ Basix**
Cat. #: 9161565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 60Å

Conditions:
Mobile phase: 10mM ammonium formate, pH 2.5: acetonitrile (10:90, v/v)
Flow rate: 1.2mL/min.
Temp.: 25°C
Det.: UV @ 225nm



LC_0090

Sedatives

Sedatives are CNS (central nervous system) depressants. They have been identified as contributors in impaired driving cases, and have been used in greyhound racing and horseracing to decrease an animal's speed. CNS depressants can be analyzed by HPLC on an Allure™ Basix column.

free literature

HPLC Analysis of Basic Pharmaceutical Compounds on an Ultra Cyano Phase

lit. cat.# 59545

Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

HPLC

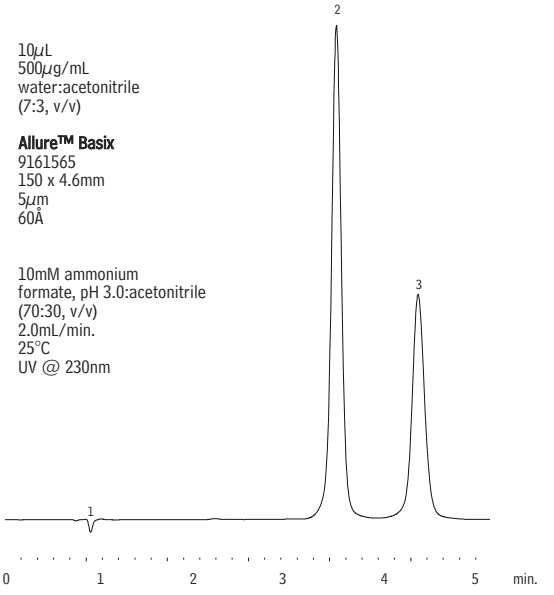
Alprazolam and Lorazepam (Sedatives)
Allure™ Basix

Peak List:
1. toluene (marker)
2. lorazepam
3. alprazolam

Sample:
Inj.: 10µL
Conc.: 500µg/mL
Solvent: water:acetonitrile (7:3, v/v)

Column: **Allure™ Basix**
Cat. #: 9161565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 60Å

Conditions:
Mobile phase: 10mM ammonium formate, pH 3.0:acetonitrile (70:30, v/v)
Flow rate: 2.0mL/min.
Temp.: 25°C
Det.: UV @ 230nm

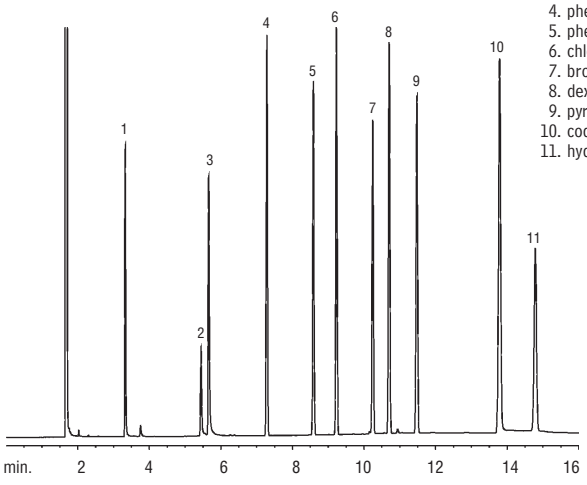


LC_0078

Cold Medications (Underivatized) Rtx®-5 Amine

GC

1. phenylpropanolamine
2. phenylephrine
3. guaifenesin
4. pheniramine
5. phenyltoloxamine
6. chlorpheniramine
7. brompheniramine
8. dextromethorphan
9. pyrilamine
10. codeine
11. hydrocodone



GC_PH00469

30m, 0.53mm ID, 1.0μm Rtx®-5 Amine (cat.# 12355)

Oven temp.:	175°C to 280°C @ 10°/min. (hold 5.5 min.)
Inj./det. temp.:	250°C/280°C
Carrier gas:	helium
Linear velocity:	40cm/sec.
Sample size:	1μL
Split vent flow:	88mL/min.
Split ratio:	20:1

Cold & Sinus Medications

Cold and sinus medications can be analyzed for phenylpropanolamine using a simple extraction procedure followed by GC analysis. An Rtx®-5 Amine or Rtx®-35 Amine column provides excellent resolution of all the compounds commonly found in most cold medications. Phenylpropanolamine is separated easily from the other compounds. All target analytes exhibit good peak shape, even when in the free base form. Additionally, the analysis is complete in less than 15 minutes, which allows quick turn-around of multiple samples.

free literature

GC Analysis of Phenylpropanolamine in Cold Medications, Using an Rtx®-5Amine Column

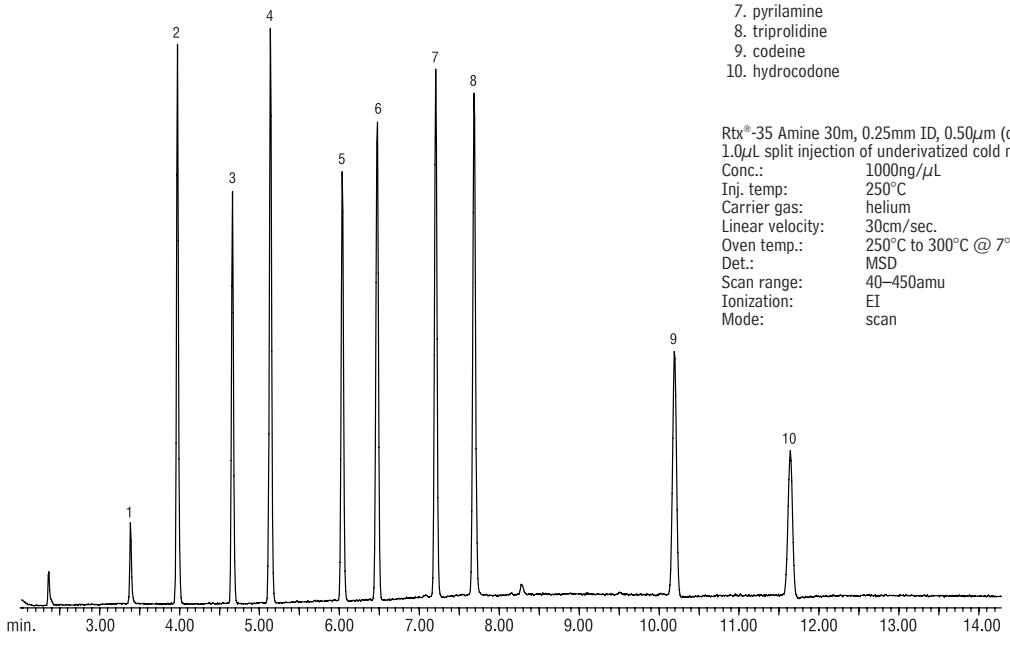
lit. cat.# 59339

Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

Cold Medications (Underivatized) Rtx®-35 Amine

GC

1. guaifenesin
2. pheniramine
3. phenyltoloxamine
4. chlorpheniramine
5. brompheniramine
6. dextromethorphan
7. pyrilamine
8. triprolidine
9. codeine
10. hydrocodone



GC_PH00575

Rtx®-35 Amine 30m, 0.25mm ID, 0.50μm (cat.# 11338)
1.0μL split injection of underivatized cold medicines

Conc.:	1000ng/μL
Inj. temp:	250°C
Carrier gas:	helium
Linear velocity:	30cm/sec.
Oven temp.:	250°C to 300°C @ 7°C/min. (hold 7 min.)
Det.:	MSD
Scan range:	40–450amu
Ionization:	EI
Mode:	scan

Steroids

Anabolic steroids can be analyzed as TMS-derivatized or as underivatized compounds. In either case, this analysis is usually done at high temperature by GC. Analysis time can be reduced through the use of thin film GC columns. Lower polarity GC columns will help to reduce the effective elution temperature. Rtx®-5 columns provide sufficient selectivity to resolve many anabolic steroids, and have the stability necessary to withstand the high temperatures needed.

Allure™ Biphenyl columns are an excellent choice for steroid analyses. Through π-π interactions with double bonds in the steroid ring structure, the biphenyl stationary phase greatly improves selectivity, relative to alkyl phases (e.g., C18). For example analyses, request the free Applications Note listed below.

Steroids also can be analyzed by HPLC on an octadecylsilyl (C18) stationary phase. Specially developed for HPLC/MS. Allure™ C18 columns increase the sensitivity of the analysis by allowing higher concentrations of organic content in the mobile phase.

free literature

Improved HPLC Analysis of Steroids, Using Restek's Unique Allure™ Biphenyl Column

lit. cat.# 580020

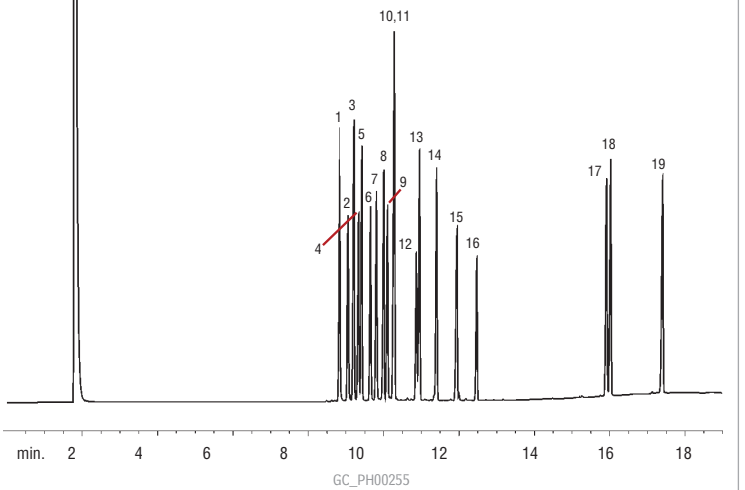
Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

Steroids, Anabolic (Underivatized)
Rtx®-5

GC

30m, 0.25mm ID, 0.10µm Rtx®-5 (cat.# 10208)
0.5µL split injection of anabolic steroids

Conc.:	1000ng/µL
Oven temp.:	180°C to 340°C @ 10°C/min. (hold 3 min.)
Inj. / det. temp.:	280°C / 340°C
Carrier gas:	helium
Linear velocity:	35cm/sec. set @ 180°C
FID sensitivity:	1.28 x 10^{-10} AFS
Split ratio:	50:1



GC_PH00255

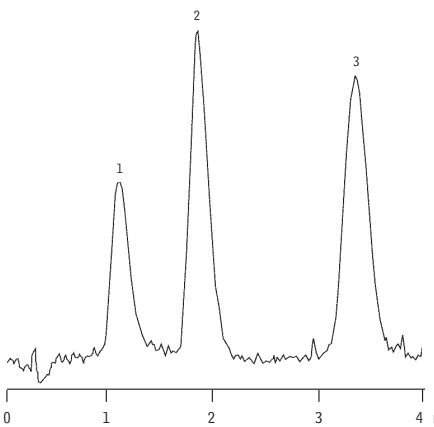
1. 5-androstene-3β,17β-diol
2. 17α-methyl-5-androstene-3β,17β-diol
3. 5α-androstan-17β-ol-3-one
4. 19-nortestosterone
5. 17α-methylandrostan-17β-ol-3-one
6. mesterolone
7. testosterone
8. 17α-methyltestosterone
9. 1-dehydrotestosterone
10. 1-dehydro-17α-methyltestosterone
11. bolasterone
12. oxymethalone
13. 19-nortestosterone-17-propionate
14. testosterone propionate
15. fluoxymesterone
16. 4-chlorotestosterone-17-acetate
17. testosterone-17β-cypionate
18. 1-dehydrotestosterone benzoate
19. 1-dehydrotestosterone undecylenate

Corticosteroids
Allure™ C18 vs. Conventional C18 (LC/MS)

HPLC

Conventional C18

water:methanol (40:60, v/v)
3324.0 cps



Allure™ C18

water:methanol (33:67, v/v)
4191.1 cps

12% increase in organic = 26% increase in LC/MS sensitivity

Peak List:
1. deoxycorticosterone (DCC) acetate
2. DCC glucoside
3. DCC

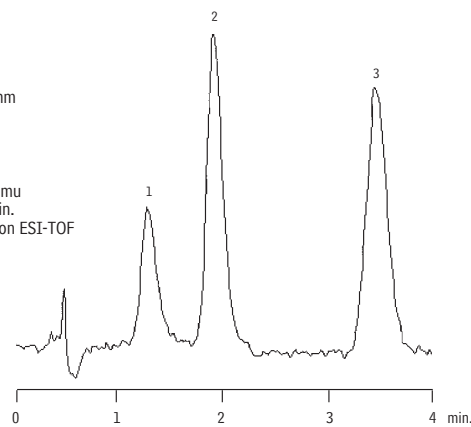
Column:

Cat. #:	9164552
Dimensions:	50 x 2.1mm
Particle size:	5µm
Pore size:	60Å

Conditions:

Scan range:	320-520amu
Flow rate:	0.4mL/min.
LC/MS Interface:	positive ion ESI-TOF
Voltage:	3800V
Nozzle temp.:	160V
Nozzle volt.:	190V

LC_0111



Data Courtesy of Pfizer Inc

Corticosteroids Ultra C18

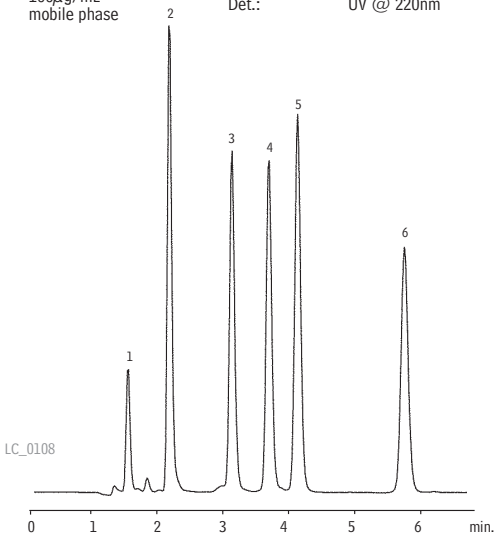
HPLC

Peak List:
1. uracil
2. triamcinolone
3. hydrocortisone
4. dexamethasone
5. corticosterone
6. deoxycorticosterone

Sample:
Inj.: 5µL
Conc.: 100µg/mL
Solvent: mobile phase

Column: Ultra C18
Cat. #: 9174565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 100Å

Conditions:
Mobile phase: water:methanol (30:70, v/v)
Flow: 1.0mL/min.
Temp.: 30°C
Det.: UV @ 220nm



Corticosteroids Pinnacle™ II Phenyl

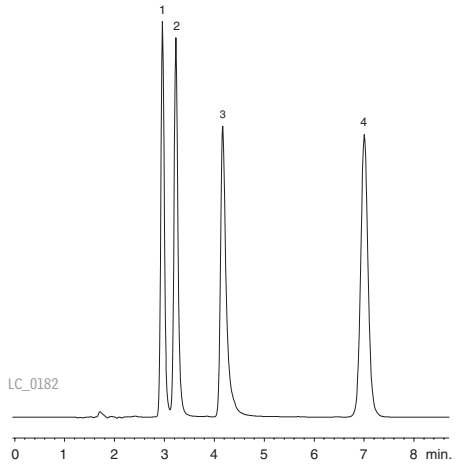
HPLC

Peak List:
1. hydrocortisone
2. cortisone
3. corticosterone
4. cortisone acetate

Sample:
Inj.: 5µL
Conc.: 200µg/mL each
Solvent: methanol

Column: Pinnacle™ II Phenyl
Cat. #: 9215565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 110Å

Conditions:
Mobile phase: water:methanol (60:40 v/v)
Flow: 1.0mL/min.
Temp.: ambient
Det.: UV @ 254nm



Corticosteroids Allure™ Biphenyl

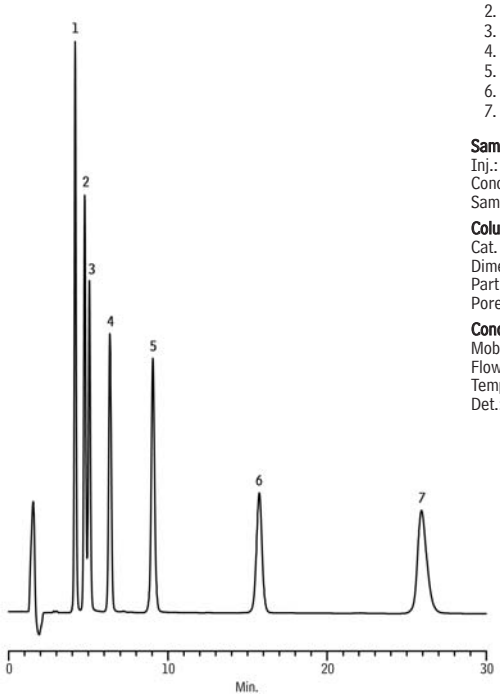
HPLC

Peak List	Ret. Time (min.)
1. hydrocortisone	4.19
2. prednisone	4.79
3. cortisone	5.08
4. dexamethasone	6.37
5. corticosterone	9.01
6. cortisone acetate	15.75
7. desoxycorticosterone	25.94

Sample:
Inj.: 5µL
Conc.: 100µg/mL each component
Sample diluent: methanol

Column: Allure™ Biphenyl
Cat. #: 9166565
Dimensions: 150 x 4.6 mm
Particle size: 5µm
Pore size: 60Å

Conditions:
Mobile phase: water:acetonitrile, 60:40
Flow: 1mL/min.
Temp.: ambient
Det.: UV @ 254 nm



Chiral Drugs

In many instances stereochemical properties of chiral drugs are the controlling factor concerning activity. One enantiomer might provide a biological function, the other might be inactive or might exhibit another functionality, which could result in side effects. In some cases, one optical isomer might be harmful. Enantiomeric separation of these compounds for accurate interpretation of drug tests, is easily achieved on Rt-βDEXcst™ and Rt-βDEXsm™ chiral capillary GC columns.

free literature

A Guide to the Analysis of Chiral Compounds by GC

lit. cat.# 59889

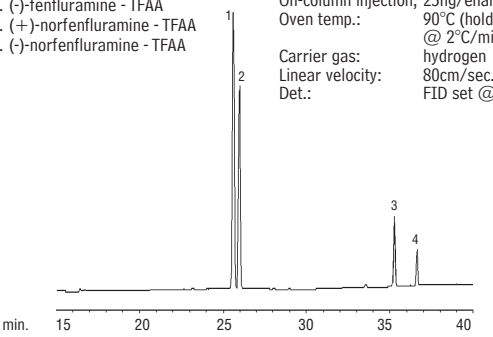
Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

Fenafluramine (TFAA Derivative)
Rt-βDEXcst™

GC

1. (+)-fenfluramine - TFAA
2. (-)-fenfluramine - TFAA
3. (+)-norfenfluramine - TFAA
4. (-)-norfenfluramine - TFAA

30m, 0.32mm ID, 0.25µm
Rt-βDEXcst™ (cat.# 13102)
On-column injection, 25ng/enantiomer
Oven temp.: 90°C (hold 1 min.) to 200°C @ 2°C/min. (hold 3 min.)
Carrier gas: hydrogen
Linear velocity: 80cm/sec. set @ 60°C
Det.: FID set @ 220°C



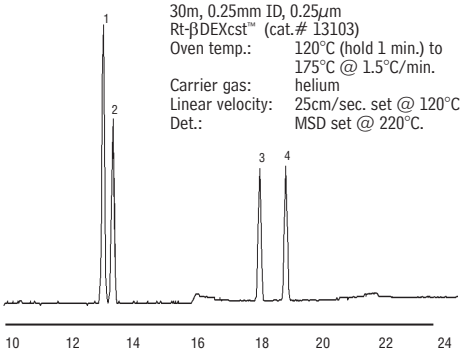
GC_PH00244

Amphetamine & Methamphetamine (TFAA Derivatives)
Rt-βDEXcst™

GC

1. (+)methamphetamine - TFAA
2. (-)methamphetamine - TFAA
3. (+)amphetamine - TFAA
4. (-)amphetamine - TFAA

30m, 0.25mm ID, 0.25µm
Rt-βDEXcst™ (cat.# 13103)
Oven temp.: 120°C (hold 1 min.) to 175°C @ 1.5°C/min.
Carrier gas: helium
Linear velocity: 25cm/sec. set @ 120°C
Det.: MSD set @ 220°C.



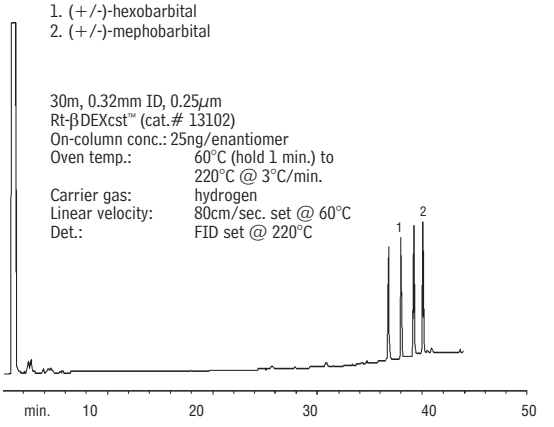
GC_PH00246

Barbiturates (Underivatized)
Rt-βDEXcst™

GC

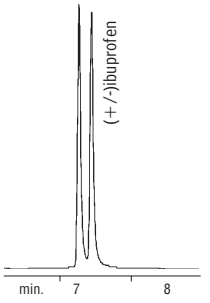
1. (+/-)-hexobarbital
2. (+/-)-mephobarbital

30m, 0.32mm ID, 0.25µm
Rt-βDEXcst™ (cat.# 13102)
On-column conc.: 25ng/enantiomer
Oven temp.: 60°C (hold 1 min.) to 220°C @ 3°C/min.
Carrier gas: hydrogen
Linear velocity: 80cm/sec. set @ 60°C
Det.: FID set @ 220°C



GC_PH00245

Ibuprofen (Underivatized)
Rt-βDEXsm™

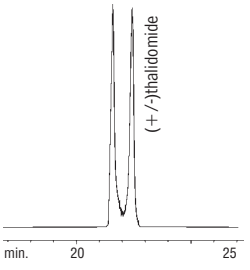


GC_PH00242

30m, 0.32mm ID, 0.25µm Rt-βDEXsm™ (cat.# 13104)
On-column injection
Conc.: 125ng each enantiomer
Oven temp.: 175°C to 200°C @ 2°C/min.
Inj./det. temp.: 200°C/230°C
Carrier gas: helium
Linear velocity: 60cm/sec.
Det.: GC-FID
Split ratio: 13:1 using cup splitter liner (cat.# 20709)

Thalidomide (Underivatized)
Rt-βDEXcst™

GC



GC_PH00243

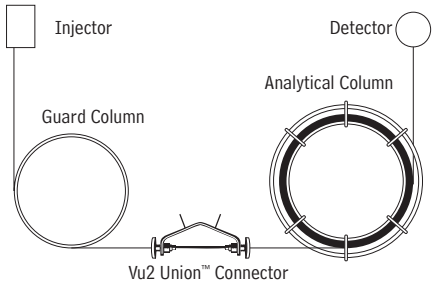
30m, 0.32mm ID, 0.25µm Rt-βDEXcst™ (cat.# 13102)
On-column injection
Conc.: approximately 15ng each enantiomer
Oven temp.: 200°C to 230°C @ 1°C/min.
Inj./det. temp.: 200°C/230°C
Carrier gas: hydrogen
Linear velocity: 80cm/sec.
Detector type: GC/FID
Split ratio: 13:1 using cup splitter liner (cat.# 20709)

What is a guard column?

A capillary GC guard column is a short length of deactivated, uncoated fused silica or MXT® stainless steel tubing that is connected to the inlet end of the analytical column (Figure 1). The guard column traps nonvolatile residues and prevents them from collecting at the inlet of the analytical column, with important benefits:

- Increased column lifetime (more injections).
- Prevents peak splitting during splitless analysis.
- Improved analyte focusing.

Figure 1 A guard column connected to an analytical column



Contaminants that accumulate within the analytical column can adsorb active compounds, reduce resolution, and cause poor peak symmetry. Packed inlet liners remove only a portion of the non-volatile contaminants in samples. When this contamination begins to affect sample analysis, a section (15-30cm) of the analytical column must be removed to restore performance. Each time a section of the analytical column is removed, retention times change and some resolution is lost, eventually resulting in a need to replace the column. By removing contaminated loops from the guard column instead of the analytical column, the inertness and length of the analytical column remain intact, so retention times and resolution do not change. The amount of time the sample spends in the guard column is minimal because there is no stationary phase.

did you **know**?

We test our guard columns/ transfer lines with the Grob test mix to ensure high inertness.

What type of guard column should be used?

It is important to match the polarity of the sample solvent and the polarity of the surface deactivation in the guard column. Intermediate Polarity (IP) guard columns allow most common solvents (methylene chloride, hexane, isooctane, toluene) to easily wet and create a uniform film on the tubing surface. A polar-deactivated guard column is necessary to allow more polar solvents such as methanol or water to wet the tubing surface. Polar-deactivated guard columns are not resistant to water vaporization, which occurs when liquid water is injected onto the tubing, and rapidly vaporizes (such as in steam cleaning). Hydroguard™ deactivation is an alternative for direct aqueous injections. However, a Hydroguard™-deactivated guard column will not allow polar solvents to wet the tubing surface, and may cause beading of the solvent if the oven temperature is 20°C below the solvent boiling point. Siltek® deactivation creates a highly inert surface for very active compounds such as chlorinated pesticides. Base-deactivated guard columns reduce adsorption and tailing for amines and other basic compounds.

please **note**

For superior inertness, try our Siltek® guard columns!

How is a guard column connected to the analytical column?

The most common connector is the Press-Tight® connector. In addition, Restek offers Vu-Union®, Vu2-Union™, and Gerstel GRAPHPACK® connectors for attaching guard columns. MXT® unions are available for connecting stainless steel MXT® columns and guard columns. See our general catalog, or website, for information about these connectors.

for **more** info

Having trouble making a leak-free connection? Try our "built in" Integra-Guard™ columns!

See **page 30** for details.

Intermediate-Polarity Deactivated Guard Columns/Transfer Lines

- Useful for a wide range of applications.
- Use with most common solvents.
- Maximum temperature: 325°C

Fused Silica

Nominal ID	Nominal OD	1-Meter	5-Meter	5-Meter/6-pk.
0.18mm	0.37 ± 0.04mm	10102	10046	
0.25mm	0.37 ± 0.04mm		10043	10043-600
0.32mm	0.45 ± 0.04mm		10044	10044-600
0.53mm	0.69 ± 0.05mm		10045	10045-600

Capillary GC Columns: Guard Columns

Siltek®-Deactivated Guard Columns/Transfer Lines

- Revolutionary deactivation process for superior inertness.
- Minimize bleed.
- Analyze active samples accurately; ideal for chlorinated pesticide analysis (reduces endrin breakdown to less than 1%).
- Maximum temperature: 380°C.

Fused Silica

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

Base-Deactivated Guard Columns

- Excellent inertness for basic compounds.
- Recommended for use with Rtx®-5 Amine, Rtx®-35Amine, and Stabilwax®-DB capillary columns.
- Tested with basic amine test mix.
- Batch test chromatogram included.
- Maximum temperature: 315°C.

Chemists using guard columns in analyses of basic compounds frequently observe peak tailing and low recovery, because conventionally deactivated tubing surfaces can be adsorptive to basic compounds. Restek offers both base-deactivated columns and base-deactivated guard columns for completely inert sample pathways.

Fused Silica

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.
0.25mm	0.37 ± 0.04mm	10000	10000-600
0.32mm	0.45 ± 0.04mm	10001	10001-600
0.53mm	0.69 ± 0.05mm	10002	10002-600

Innovative Integra-Guard™ Columns

For analysts who find it inconvenient to make a leak-free connection between the guard column and the analytical column, Restek offers Integra-Guard™ columns. These innovative columns incorporate both guard column and analytical column in a continuous length of tubing, eliminating the connection and all connection-associated problems! The guard column section is tied separately from the analytical column, using high-temperature string. The column is suspended in our unique "crush-free" cage, which protects the column from damage.

restek
innovation!
Integra-Guard™ Columns: guard columns WITHOUT connections—protecting your analytical column has never been this easy!

Restek offers a wide variety of Integra-Guard™ capillary columns, listed in the figure below. The Integra-Guard™ column is so economical that we challenge you to compare our price against that of a conventional connection, even if you assemble it yourself. If you are currently using a guard column, or are considering using one, call today and ask about Integra-Guard™ columns.

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®-5 column with a 5-meter Integra-Guard™ column is cat.# 10223-124.

ID	Length	Suffix #
0.25mm	5m	-124
	10m	-127
0.32mm	5m	-125
	10m	-128
0.53mm	5m	-126
	10m	-129

Phases currently available as Integra-Guard™ columns

Rtx®-1
Rtx®-1MS
Rtx®-5
Rtx®-5MS
Rtx®-5Sil MS
XTI®-5
Rtx®-1301
Rtx®-624
Rtx®-1701
Rtx®-Volatiles
Rtx®-20
Rtx®-35
Rtx®-35MS
Rtx®-BAC 1 & 2
Stabilwax®

Available for all phases listed, for columns with 0.25 to 0.53mm ID and lengths to 75 meters.

Rxi™-1ms (nonpolar phase, Crossbond® 100% dimethyl polysiloxane)

- General purpose columns for drugs of abuse, essential oils, hydrocarbons, pesticides, PCB congeners or (e.g.) Aroclor® mixes, sulfur compounds, amines, solvent impurities, simulated distillation, oxygenates, gasoline range organics (GRO), refinery gases.
- Temperature range: -60°C to 330/350°C (bleed tested temperature/maximum operating temperature).
- Low bleed - improved signal to noise ratio, for better sensitivity and mass spectral integrity.
- Equivalent to USP G2 phase.

new **column** technology!

Rxi™-1ms (fused silica)
(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.25mm	0.25	-60 to 330/350°C	13320	13323	13326
	0.50	-60 to 330/350°C	13335	13338	13341
	1.00	-60 to 330/350°C	13350	13353	13356
0.32mm	0.25	-60 to 330/350°C	13321	13324	13327
	0.50	-60 to 330/350°C	13336	13339	13342
	1.00	-60 to 330/350°C	13351	13354	13357
0.53mm	0.50	-60 to 330/350°C	13337	13340	
	1.00	-60 to 330/350°C	13352	13355	
	1.50	-60 to 330/350°C	13367	13370	

ID	df (µm)	temp. limits	12-Meter	20-Meter	25-Meter	50-Meter
0.18mm	0.18	-60 to 330/350°C		13302		
0.20mm	0.33	-60 to 330/350°C	13397		13398	13399

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 contact your Restek representative, and we will do everything we can to help you find a solution.

Similar **Phases**

DB-1, DB-1ms, HP-1, HP-1ms, Ultra-1, SPB-1, Equity-1

Rtx®-1MS (fused silica)
(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.10	-60 to 330/350°C	11605	11608
	0.25	-60 to 330/350°C	11620	11623
	0.50	-60 to 330/350°C	11635	11638
0.32mm	0.10	-60 to 330/350°C	11606	11609
	0.25	-60 to 330/350°C	11621	11624
	0.50	-60 to 330/350°C	11636	11639
0.53mm	1.00	-60 to 320/340°C	11652	11655
	1.50	-60 to 310/330°C	11667	11670

Similar **Phases**

DB-1, DB-1MS, HP-1, HP-1MS, Ultra-1, SPB-1, Equity-1, MDN-1

Rtx®-20 (low/mid-polarity phase; Crossbond® 80% dimethyl / 20% diphenyl polysiloxane)

- General purpose columns for volatile compounds, flavor compounds, alcoholic beverages.
- Temperature range: -20°C to 320°C.
- 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	0.25	-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	0.25	-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	0.50	-20 to 260/280°C	10337	10340	10343	
	1.00	-20 to 260/280°C	10352	10355	10358	

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

Similar **Phases**

SPB-20

Capillary GC Columns

Similar **Phases**

DB-5, HP-5, HP-5ms, Ultra-2, SPB-5, Equity-5

Rxi™-5ms (low-polarity phase, Crossbond® 5% diphenyl / 95% dimethyl polysiloxane)

- General purpose columns for semivolatiles, phenols, amines, residual solvents, drugs of abuse, pesticides, PCB congeners or (e.g.) Aroclor® mixes, solvent impurities.
- Temperature range: -60°C to 330/350°C (bleed tested temperature/maximum operating temperature).
- Low bleed - improved signal to noise ratio, for better sensitivity and mass spectral integrity.
- Equivalent to USP G27 phase.

new **column** technology**!**

Rxi™-5ms (fused silica)

(Crossbond® 5% diphenyl / 95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.25mm	0.25	-60 to 330/350°C	13420	13423	13426
	0.50	-60 to 330/350°C	13435	13438	
	1.00	-60 to 330/350°C	13450	13453	13456
0.32mm	0.25	-60 to 330/350°C	13421	13424	
	0.50	-60 to 330/350°C	13436	13439	
	1.00	-60 to 330/350°C	13451	13454	13457
0.53mm	0.25	-60 to 330/350°C	13422	13425	
	0.50	-60 to 330/350°C	13437	13440	
	1.00	-60 to 330/350°C	13452	13455	
	1.50	-60 to 330/350°C	13467	13470	

ID	df (µm)	temp. limits	12-Meter	20-Meter	25-Meter	50-Meter
0.18mm	0.18	-60 to 330/350°C		13402		
	0.36	-60 to 330/350°C		13411		
0.20mm	0.33	-60 to 330/350°C	13497		13498	13499

also **available**

Custom lengths and film thicknesses are available. Call technical service at **800-356-1688 (ext. 4)**, or contact your Restek representative.

Rtx®-5MS (fused silica)

(Crossbond® 5% diphenyl / 95% dimethyl polysiloxane)

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
0.32mm	0.25	-60 to 330/350°C	12621	12624	12627
	0.50	-60 to 330/350°C	12636	12639	12642
0.53mm	0.50	-60 to 320/340°C	12637	12640	
	1.00	-60 to 320/340°C	12652	12655	

Similar **Phases**

DB-5, HP-5, HP-5MS, Ultra-2, SPB-5, Equity-5, MDN-5
DB-5MS is equivalent to Rtx®-5Sil MS

Rtx®-5 Amine (low-polarity phase; Crossbond® 5% diphenyl / 95% dimethyl polysiloxane)

- Application-specific columns for amines and other basic compounds, including alkylamines, diamines, triamines, ethanolamines, and nitrogen-containing heterocyclics.
- Stable to 315°C.

Active basic compounds that previously required derivatization or another analytical technique can be analyzed on the Rtx®-5 Amine column. The tubing surface is chemically altered to reduce tailing of basic compounds, eliminating the need for column priming. Breakthrough technology also allows the analysis of neutral compounds, adsorptive compounds with oxygen groups susceptible to hydrogen bonding, and even mildly acidic compounds such as phenols.

Thorough testing of each Rtx®-5 Amine column ensures that every column exceeds the requirements for analyzing ppm levels of amines, without priming. The temperature program/bleed profile for each column is measured to ensure low bleed at maximum operating temperature. Rtx®-5 Amine columns are bonded and can be rejuvenated by solvent rinsing.

Rtx®-5 Amine (fused silica)

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.50	-60 to 300/315°C	12335	12338
	1.00	-60 to 300/315°C	12350	12353
0.32mm	1.00	-60 to 300/315°C	12351	12354
	1.50	-60 to 290/305°C	12366	12369
0.53mm	1.00	-60 to 290/305°C	12352	12355
	3.00	-60 to 280/295°C	12382	12385

Similar **Phases**

PTA-5

Rtx®-1701 (mid-polarity phase; Crossbond® 14% cyanopropylphenyl / 86% dimethyl polysiloxane)

- General purpose columns for alcohols, oxygenates, PCB congeners or (e.g.) Aroclor® mixes, pesticides.
- Temperature range: -20°C to 280°C.
- Equivalent to USP G46 phase.

Rtx®-1701 (fused silica)

(Crossbond® 14% cyanopropylphenyl/86% dimethyl polysiloxane)

ID	df (µm)	temp. limits*	15-Meter	30-Meter	60-Meter	105-Meter
0.25mm	0.25	-20 to 280°C	12020	12023	12026	12029
	0.50	-20 to 270/280°C	12035	12038	12041	12044
0.32mm	0.25	-20 to 280°C	12021	12024	12027	12030
	0.50	-20 to 270/280°C	12036	12039	12042	12045
0.53mm	0.50	-20 to 260/270°C	12037	12040	12043	
	1.00	-20 to 250/270°C	12052	12055	12058	

Similar **Phases**

DB-1701, HP-1701, SPB-1701

Rtx®-35/Rtx®-35MS (mid-polarity phase; Crossbond® 35% diphenyl / 65% dimethyl polysiloxane)

- General purpose columns for organochlorine pesticides, PCB congeners or (e.g.) Aroclor® mixes, herbicides, pharmaceuticals, sterols, rosin acids, phthalate esters.
- Temperature range: 0°C to 320°C.
- Equivalent to USP G42 phase.

Rtx®-35 (fused silica)

(Crossbond® 35% diphenyl/65% dimethyl polysiloxane)

ID	df (µm)	temp. limits*	15-Meter	30-Meter	60-Meter	105-Meter
0.25mm	0.25	0 to 320°C	10420	10423	10426	10429
	0.50	0 to 310°C	10435	10438	10441	10444
0.32mm	0.25	0 to 320°C	10421	10424	10427	10430
	0.50	0 to 310°C	10436	10439	10442	10445
0.53mm	0.50	0 to 300°C	10437	10440	10443	
	1.00	0 to 290°C	10452	10455	10458	

Similar **Phases**

DB-35, HP-35, SPB-35, SPB-608

Rtx®-35MS (fused silica)

(Crossbond® 35% diphenyl / 65% dimethyl polysiloxane)

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.10	-20 to 320°C	14605	14608
	0.25	-20 to 320/340°C	14620	14623
0.32mm	0.10	-20 to 320/340°C	14606	14609
	0.25	-20 to 320/340°C	14621	14624
0.53mm	0.50	-20 to 300/320°C	14637	14640
	1.00	-20 to 290°C	14652	14655

Rtx®-35 Amine (mid-polarity phase; Crossbond® 35% diphenyl / 65% dimethyl polysiloxane)

- Application-specific columns for amines and other basic compounds, including alkylamines, diamines, triamines, ethanolamines, and nitrogen-containing heterocyclics.
- Stable to 310°C.

Active basic compounds that otherwise require derivatization can be analyzed on an Rtx®-35 Amine column. The tubing surface is chemically altered to reduce tailing of basic compounds, eliminating the need for column priming. An Rtx-35® Amine column is ideal for a wide variety of basic compounds, but also is suitable for neutral compounds, adsorptive compounds with oxygen groups susceptible to hydrogen bonding, or even weakly acidic compounds such as phenols. Every Rtx®-5 Amine column is tested to ensure that it exceeds the requirements for analyzing ppm levels of amines, without priming, and to ensure low bleed at maximum operating temperature.

Rtx®-35 Amine (fused silica)

(Crossbond® 35% diphenyl/65% dimethyl polysiloxane)

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.50	0 to 290/310°C	11335	11338
	1.00	0 to 280/300°C	11350	11353
0.32mm	1.00	0 to 280/300°C	11351	11354
	1.50	0 to 270/290°C	11366	11369
0.53mm	1.00	0 to 260/280°C	11352	11355
	3.00	0 to 240/260°C	11382	11385

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

Capillary GC Columns

Rtx®-200/Rtx®-200MS (mid-polarity phase; Crossbond® trifluoropropylmethyl polysiloxane)

- General purpose columns for solvents, Freon® fluorocarbons, alcohols, ketones, silanes, glycols. Excellent confirmation column, with an Rtx®-5 column, for phenols, nitrosamines, organochlorine pesticides, chlorinated hydrocarbons, chlorophenoxy herbicides.
- Temperature range: -20°C to 340°C.
- Equivalent to USP G6 phase.

Similar Phases

DB-200, DB-210

Rtx®-200 (fused silica)

(Crossbond® trifluoropropylmethyl polysiloxane)

ID	df (µm)	temp. limits*	15-Meter	30-Meter	60-Meter	105-Meter
0.25mm	0.25	-20 to 320/340°C	15020	15023	15026	15029
	0.50	-20 to 310/330°C	15035	15038	15041	15044
0.32mm	0.25	-20 to 320/340°C	15021	15024	15027	15030
	0.50	-20 to 310/330°C	15036	15039	15042	15045
0.53mm	0.50	-20 to 300/320°C	15037	15040	15043	
	1.00	-20 to 290/310°C	15052	15055	15058	

also available

Custom lengths and film thicknesses are available. Call technical service at **800-356-1688 (ext. 4)**, or contact your Restek representative.

Rtx®-200MS (fused silica)

(Crossbond® trifluoropropylmethyl polysiloxane)

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.10	-20 to 320/340°C	15605	15608
	0.25	-20 to 320/340°C	15620	15623
0.32mm	0.10	-20 to 320/340°C	15606	15609
	0.25	-20 to 320/340°C	15621	15624
0.53mm	0.50	-20 to 300/320°C	15637	15640

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

Stabilwax® (polar phase; Crossbond® Carbowax® polyethylene glycol)

- General purpose columns for FAMEs, flavor compounds, essential oils, amines, solvents, xylene isomers, US EPA Method 603 (acrolein/acrylonitrile).
- Resistant to oxidative damage.
- Temperature range: 40°C to 250°C.
- Equivalent to USP G14, G15, G16, G20, G39 phases.

Similar Phases

DB-WAX, DB-WAXetr, HP-Wax, HP-Innowax, Supelcowax 10

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	0.25	40 to 250°C	10620	10623		10626
	0.50	40 to 250°C	10635	10638		10641
0.32mm	0.25	40 to 250°C	10621	10624		10627
	0.50	40 to 250°C	10636	10639		10642
0.53mm	0.50	40 to 250°C	10637	10640		10643
	1.00	40 to 240/250°C	10652	10655	10655-600	10658

Similar Phases

DB-WAX, HP-Wax

Rtx®-Wax (polar phase; Crossbond® Carbowax® polyethylene glycol)

- General purpose columns for FAMEs, solvents, BTEX aromatics, flavor compounds.
- Temperature range: 20°C to 250°C.
- 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	0.25	20 to 250°C	12420	12423	12426
	0.50	20 to 250°C	12435	12438	12441
0.32mm	0.25	20 to 250°C	12421	12424	12427
	0.50	20 to 250°C	12436	12439	12442
	1.00	20 to 240/250°C	12451	12454	12457
0.53mm	0.50	20 to 250°C	12437	12440	12443
	1.00	20 to 240/250°C	12452	12455	12458

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

Rtx®-G27 & Rtx®-G43

- Application-specific columns, designed for analysis of residual solvents in pharmaceutical products.
- Meet all requirements of USP 467.
- Integra-Guard™ guard+analytical column eliminates connecting problems and leaks.
- Rtx®-G27 thermally stable to 290°C; Rtx®-G43 thermally stable to 240°C.

Some USP 467 methods require the use of a guard column. Our Integra-Guard™ integrated guard column system makes this an easy task.

Rtx®-G27 (fused silica with 5-meter Integra-Guard™)

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	30-Meter with 5-Meter, 0.53mm ID Integra-Guard™ Column
0.53mm	5.00	-60 to 270/290°C	10279-126

Rtx®-G43 (fused silica with 5-meter Integra-Guard™)

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

ID	df (µm)	temp. limits	30-Meter with 5-Meter, 0.53mm ID Integra-Guard™ Column
0.53mm	3.00	-20 to 240°C	16085-126

Rtx®-BAC1/Rtx®-BAC2 (proprietary Crossbond® phase)

- Application-specific columns for blood alcohol analysis, achieving baseline resolution in less than 3 minutes. Also excellent for abused inhalant anesthetics, γ-hydroxybutyrate (GHB) / γ-butyrolactone (GBL), glycols, and common industrial solvents.
- Rtx®-BAC2 confirmation column provides four elution order changes under the same conditions.
- Rtx®-BAC1 stable to 260°C, Rtx®-BAC2 stable to 240°C.

These columns achieve baseline separation of all blood alcohol compounds in blood, breath, or urine in less than 3 minutes, under isothermal conditions. Isothermal analysis increases productivity by eliminating the need for oven cycling. Confirmation is easily achieved with this tandem set because there are four elution order changes between the two columns.

Rtx®-BAC1 (fused silica)

ID	df (µm)	temp. limits	30-Meter
0.32mm	1.80	-20 to 240/260°C	18003
0.53mm	3.00	-20 to 240/260°C	18001

Rtx®-BAC2 (fused silica)

ID	df (µm)	temp. limits	30-Meter
0.32mm	1.20	-20 to 240/260°C	18002
0.53mm	2.00	-20 to 240/260°C	18000

Cyclodextrin Columns for Analyzing Many Chiral Compounds

By adding β or γ cyclodextrin to our bonded Rtx®-1701 stationary phase, we greatly enhance overall utility and column lifetime for our chiral columns, compared to columns that have pure cyclodextrin stationary phases. Separations of more than one hundred chiral compounds have been achieved using our unique DEX columns, and our columns continue to demonstrate stability after hundreds of temperature program cycles. Refer to the applications section of this catalog for example applications, or call our Technical Service chemists or your Restek representative for assistance in matching a column to your chiral analysis.

Rt-βDEXm™ (fused silica)

(permethylated beta cyclodextrin doped into 14% cyanopropylphenyl/86% dimethyl polysiloxane)

ID	df (µm)	temp. limits	30-Meter
0.25mm	0.25	40 to 230°C	13100
0.32mm	0.25	40 to 230°C	13101

Uses: General purpose chiral phase with many published 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 chiral compounds found in essential oils.

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, and styrene oxides.

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: Proprietary stationary phase, developed specifically for the fragrance industry. Also used for pharmaceutical applications.

Rt-γDEXsa™ (fused silica)

(2,3-di-acetoxy-6-O-*tert*-butyl dimethylsilyl gamma cyclodextrin doped into 14% cyanopropylphenyl/86% dimethyl polysiloxane)

ID	df (µm)	temp. limits	30-Meter
0.25mm	0.25	40 to 230°C	13113
0.32mm	0.25	40 to 230°C	13112

Uses: Larger organic molecules. Also useful for flavor compounds in fruit juices.

CarboBlack™ Solid Supports

Graphitized carbon black offers unique selectivity for alcohols, with very little adsorption. 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 Carbopack™ and Carbograph™ supports.

for **more** info

Blood alcohol analysis on a CarboBlack™ B packed column is shown on **page 11**.

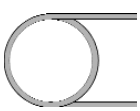
		Stainless Steel Tubing				SilcoSmooth™ Tubing**			
On CarboBlack™ B	Mesh	L (ft.)	OD (in.)	ID (mm)	cat.#*	L (m)	OD (in.)	ID (mm)	cat.#*
5% Carbowax® 20M	80/120	—	—	—	—	2	1/8	2	80105-
5% Carbowax® 20M	60/80	6	1/8	2.1	88012-	1.8	1/8	2	80106-
6.6% Carbowax® 20M	80/120	6	1/8	2.1	80451-	2	1/8	2	80107-
4% Carbowax® 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_3PO_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-

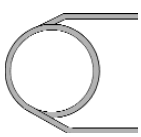
		Stainless Steel Tubing				SilcoSmooth™ Tubing**			
On CarboBlack™ C	Mesh	L (ft.)	OD (in.)	ID (mm)	cat.#*	L (m)	OD (in.)	ID (mm)	cat.#*
0.2% Carbowax® 1500	60/80	6	1/8	2.1	80456-	2	1/8	2	80121-
0.2% Carbowax® 1500	80/100	6	1/8	2.1	80457-	2	1/8	2	80122-
0.1% Rt-1000	80/100	6	1/8	2.1	80458-	1.8	1/8	2	80205-
0.19% picric acid	80/100	6	1/8	2.1	80459-	2	1/8	2	80311-
0.3% Carbowax® 20M/0.1% H_3PO_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.

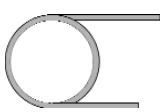
Column Configurations



General Configuration
Suffix -800

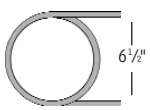


Agilent 5880, 5890, 5987, 6890:
Suffix -810



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

Note: Initial 2" of column will be empty, to accommodate a needle. For a completely filled column add suffix -901.

See our general catalog for custom configurations

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 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 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**			
On 100/120 Silcoport™ W***	L (ft.)	OD (in.)	ID (mm)	cat.#*	L (m)	OD (in.)	ID (mm)	cat.#*
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**			
On Chromosorb® PAW	Mesh	L (ft.)	OD (in.)	ID (mm)	cat.#*	L (m)	OD (in.)	ID (mm)	cat.#*
10% TCEP		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. See page 37.
**Silcosteel®-deactivated stainless steel.
***Modified version of Chromosorb® W; highest inertness, most consistent performance.

COLUMN INSTALLS THIS END

Splitless Liners for Agilent/Finnigan GCs	Benefits/Uses	ID*/OD & Length (mm)	Similar to Agilent part #	cat.# ea.	5-pk.	25-pk.
4mm Splitless	trace samples >2µL	4.0 ID 6.5 OD x 78.5	210-3003 (ea.) 210-3003-5 (5-pk.)	20772	20773	20774
Siltek® 4mm Splitless	trace samples >2µL	4.0 ID 6.5 OD x 78.5	—	20772-214.1	20773-214.5	20774-214.25
4mm Splitless w/ Wool	trace samples >2µL	4.0 ID 6.5 OD x 78.5	19251-60540 (ea.) 5183-4691 (5-pk.) 5183-4692 (25-pk.)	22400	22401	22402
Gooseneck Splitless (4mm)†	trace samples >2µL	4.0 ID 6.5 OD x 78.5	5181-3316 (ea.) 5183-4695 (5-pk.) 5183-4696 (25-pk.)	20798	20799	20800
Siltek® Gooseneck Splitless (4mm)†	trace samples >2µL	4.0 ID 6.5 OD x 78.5	—	20798-214.1	20799-214.5	20800-214.25
Gooseneck Splitless (4mm) w/ Wool†	trace samples >2µL	4.0 ID 6.5 OD x 78.5	5062-3587 (ea.) 5183-4693 (5-pk.) 5183-4694 (25-pk.)	22405	22406	22407
Siltek® Gooseneck Splitless (4mm) w/ Wool†	trace samples >2µL	4.0 ID 6.5 OD x 78.5	—	22405-213.1	22406-213.5	22407-213.25

Splitless Liners for Shimadzu GCs	Benefits/Uses:	ID*/OD & Length (mm)	Similar to Shimadzu part #	cat.# ea.	5-pk.	25-pk.
17A & 2010 Double Gooseneck	reduces backflash and catalytic decomposition	3.5 ID 5.0 OD x 95	—	20958	20959	20960
17A & 2010 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 #	cat.# ea.	5-pk.	25-pk.
17A & 2010 Split/Splitless w/ Wool	universal, for most common analyses	3.5 ID 5.0 OD x 95	221-41444-00	20955	20956	20957
Siltek® 17A & 2010 Split/Splitless w/ Wool	universal, for most common analyses	3.5 ID 5.0 OD x 95	—	20955-213.1	20956-213.5	20957-213.25

Liners for Varian 1177 GCs	Benefits/Uses:	ID*/OD & Length (mm)	Similar to Varian part #	cat.# ea.	5-pk.	25-pk.
Gooseneck Splitless (4mm)	trace samples <2µL	4.0 ID 6.5 OD x 78.5	39-26119-27	21896	21897	—
Siltek® Gooseneck Splitless (4mm)	trace samples <2µL	4.0 ID 6.5 OD x 78.5	—	21896-214.1	21897-214.5	—
Gooseneck Splitless (4mm) w/ Wool	trace samples <2µL	4.0 ID 6.5 OD x 78.5	39-26119-36	21896-200.1	21897-200.5	—
Siltek® Gooseneck Splitless (4mm) w/ Wool	trace samples <2µL	4.0 ID 6.5 OD x 78.5	—	21896-213.1	21897-213.5	—

Splitless Liners for PerkinElmer GCs	Benefits/Uses:	ID*/OD & Length (mm)	Similar to PE part #	cat.# ea.	5-pk.	25-pk.
Auto SYS™ Splitless w/ Wool (2mm ID)	trace samples	2.0 ID 6.2 OD x 92.1	N6101372	20829	20830	20831
Siltek® Auto SYS™ Splitless w/ Wool (2mm ID)	trace samples	2.0 ID 6.2 OD x 92.1	—	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	—

Splitless Liners for Thermo Finnigan 8000 & TRACE™ Series GCs	Benefits/Uses:	ID*/OD & Length (mm)	Similar to TF part #	cat.# ea.	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	3.0 ID 8.0 OD x 105	—	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

*Nominal ID at syringe needle expulsion point.
†Use this liner for increased sensitivity.

All liners are **100%** deactivated

All liners are shipped intermediate polarity (IP) deactivated unless otherwise requested.

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
GCQ9.5	
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	
54011.5	
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

for more info
To see our complete selections of septa, ferrules, and other consumables for GC, please request our current catalog, or visit our website.

save money!
Buy ferrules in 50-packs!

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 (3/16")	20351	20352	20353
6mm (1/4")	20355	20356	20357
7mm	20381	20382	20383
8mm	20370	20371	—
9mm	20354	20358	20362
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 Plug	20372	20373	20374

Measure your old septum here (size in mm)

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	≤ 0.20µµ	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	24912
1.2mm	0.75mm	10-pk.	22218	—	—
1.6mm	1.00mm*	10-pk.	—	21060	—

*For micropacked columns.
**For 0.28mm MXT® columns.

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.#
5mm Graphite Liner Seals for Varian 1078/1079 GCs	450°C	392611919 392534201	10-pk.	22683

Viton® O-Rings for PerkinElmer Auto SYS™ GCs

Graphite O-Rings for PerkinElmer Auto SYS™ XL PSS

Description	Max. temp.	Similar to PE part #	qty.	cat.#
Graphite O-Rings for PerkinElmer Auto SYS™ XL PSS	450°C	N610-1751	10-pk.	21475
Graphite O-Rings for PerkinElmer Auto SYS™ 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

- Keep several on hand in your laboratory—can be used in many different ways.
- Use hooked end for removing septa and O-rings; pointed end works well for removing stuck ferrules or fragments.

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

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

GC Replacement Parts for Agilent GCs, Lit. Cat. #59627E

2006 General Catalog Lit. Cat. #580021

Leak Detector

- Reliable thermal conductivity leak detector—every analyst should have one.
- Compact, portable, ergonomic design—easy to hold and operate.
- Sensitive—detects helium or hydrogen at $1x10^{-4}$ cc/sec*.
- Fast results—responds to leaks in less than 2 seconds, zeros with the touch of a button.
- 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 retains 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 detecting helium, hydrogen, or nitrogen leaks in your GC system. 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 drawn into the system through the leaks will contaminate the system.

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 possibly hazardous conditions.

*Sensitivity measured using helium.

Leak Detector Accessory Kit

The kit includes an adaptor fitting that fits over the probe assembly to detect leaks in hard-to-reach locations, and a mounting bracket that can be affixed to the wall or GC.

Verify pinpoint leaks with the adaptor fitting.

Leak Detector is easily accessed when stored in the mounting bracket.

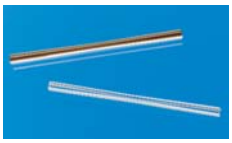
Description	qty.	cat.#
Leak Detector Accessory Kit (adaptor fitting for probe, mounting bracket)	kit	22453

Press-Tight® Connectors

- Fit column ODs from 0.33–0.74mm (Restek 0.1mm–0.53mm ID).
- Made from inert fused silica.
- Deactivated Press-Tight® connectors are ideal for better recovery of polar and non-polar compounds.

Universal Press-Tight® Connectors

- Connect a guard column to an analytical column.
- Repair a broken column.
- Connect a column outlet to a transfer line.



Description	5-pk.	25-pk.	100-pk.
Universal Press-Tight® Connectors	20400	20401	20402
Deactivated, Universal Press-Tight® Connectors	20429	20430	20431

Universal Angled Press-Tight® Connectors

- Angle approximates the curvature of a capillary column, reduces strain on column-end connections.

Description	5-pk.	25-pk.	100-pk.
Universal Angled Press-Tight® Connectors	20446	20447	20448
Deactivated Universal Angled Press-Tight® Connectors	20446-261	20447-261	20448-261

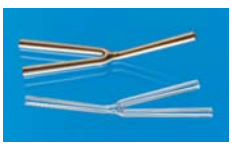
Universal "Y" Press-Tight® Connectors

- Split sample flow onto two columns.
- Split a single column flow to two detectors—perform confirmation analysis with a single injection.

Description	ea.	3-pk.
Universal "Y" Press-Tight® Connector	20405	20406
Deactivated Universal "Y" Press-Tight® Connector	20405-261	20406-261

Universal Angled "Y" Press-Tight® Connectors

- Angle approximates the curvature of a capillary column, reduces strain on column-end connections.



Description	ea.	3-pk.
Universal Angled "Y" Press-Tight® Connector	20403	20404
Deactivated Universal Angled "Y" Press-Tight® Connector	20403-261	20404-261

MXT®-Union Connector Kits for Fused Silica Columns

- Low-dead-volume, leak-tight connection.
- Reusable; use to oven temperatures of 350°C.
- Siltek® treatment ensures maximum inertness.
- Ideal for connecting a guard column or transfer line to an analytical column.

These MXT® connectors can be used with fused silica tubing, as well as with metal tubing, because a Valcon polyimide $^{1}/_{32}$-inch one-piece fused silica adaptor allows a capillary column to slide into the adaptor and be locked in place simply by loosening and tightening the fitting.

MXT®-Union Connector Kits for Fused Silica Columns

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

MXT® "Y"-Union Connector Kits for Fused Silica Columns

Each kit contains the MXT® union, three $^{1}/_{32}$-inch 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

$^{1}/_{32}$-Inch Replacement Nut

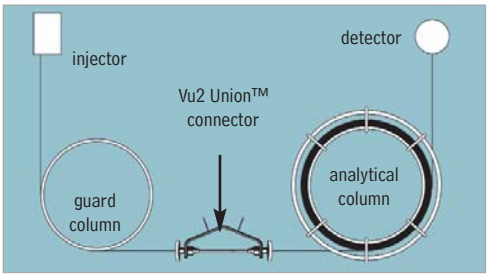
Description	qty.	cat.#
$^{1}/_{32}$" Replacement Nut	5-pk	20389

Vu2 Union™ Connectors

- Connect a guard column to an analytical column.
- Connect a column to a transfer line.
- Connect two columns in series.
- Repair a broken column.

Our Vu2 Union™ connector combines the simplicity of a Press-Tight® union with the strength of a metal union. The columns cannot unexpectedly disconnect, even at temperatures as high as 400°C.

A guard column connected to an analytical column by a Vu2 Union™ connector.



Secure, reliable column-to-column connections!

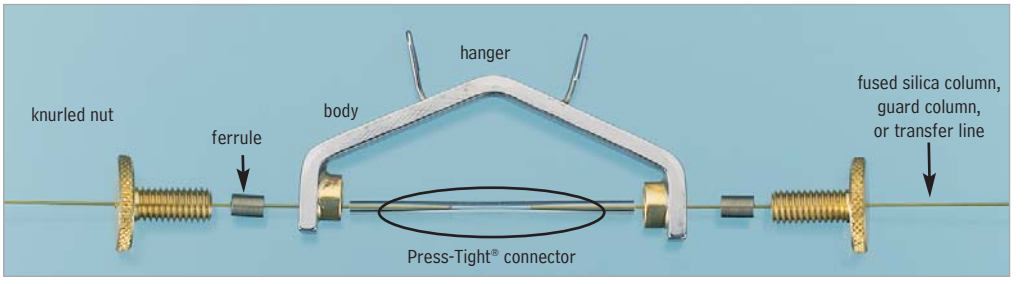
How does a Vu2 Union™ connector work?

A Press-Tight® union in the Vu2 Union™ connector joins the fused silica ends together; the ferrule and knurled nut at each end of the connector hold the tubing in place via a secondary seal between the ferrule and the Press-Tight® union. Each knurled nut applies independent pressure to each ferrule, to make a leak-tight seal with the column end. These ultra-strong connections will not unexpectedly disconnect under temperature changes, vibrations, or other stresses normally encountered in GC analyses. The open design allows visual confirmation of the seal between the column and the Press-Tight® union, to ensure confidence in the connection. Hang the connector from the column cage, to minimize stress on the connections.

Who will benefit from using Vu2 Union™ connectors?

Any analyst using guard columns, transfer lines, or restrictor tubing, performing a dual-column analysis with columns connected in series, or seeking to repair a broken column will find Vu2 Union™ connectors the simple, reliable, easy-to-use solution to their connection needs.

The Vu2 Union™ connector's open design allows visual confirmation of the seal; secondary seals ensure a leak-tight connection.



for **more** info
See **page 43** for Universal Press-Tight® Connectors.

Kits include: Vu2 Union™ body, 2 knurled nuts, 2 Press-Tight® unions, and 4 ferrules

Description	Ferrules Fit Column ID	qty.	cat.#
Vu2 Union™ Connector Kit	0.10/0.15mm	kit	22220
Vu2 Union™ Connector Kit	0.18/0.28mm	kit	21105
Vu2 Union™ Connector Kit	0.32mm	kit	21106
Vu2 Union™ Connector Kit	0.45/0.53mm	kit	21107
Knurled nut		2-pk.	21108

NOTE: Not recommended for GC column-to-MS connections—use the Vacuum Vu-Union® (see our general catalog, or website).

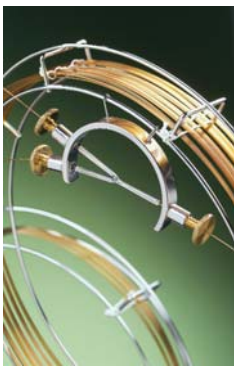
Graphite Ferrules for Vu2 Union™ Connectors

- High-purity, high-density graphite.
- Stable to 450°C.
- No binders that can off-gas or adsorb analytes.
- Smooth surface and clean edges.

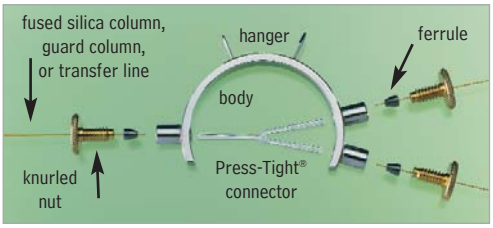
Ferrule ID	Fits Column ID	Graphite 2-pk.	Graphite 10-pk.
0.3mm	0.10/0.15mm	22221	22222
0.4mm	0.18/0.28mm	20280	20281
0.5mm	0.32mm	20282	20283
0.8mm	0.45/0.53mm	20284	20285

SeCure™ "Y" Connector Kits

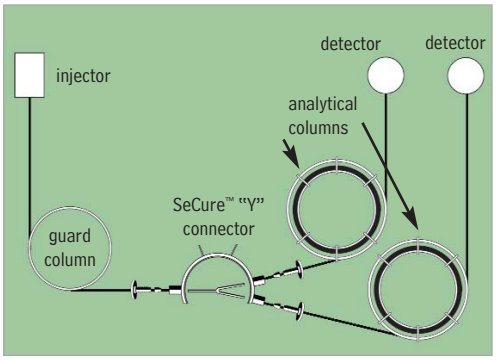
- Connect two analytical columns to a transfer line or guard column.
- Use standard "Y" Press-Tight® connectors and 1/16" graphite ferrules.
- Reliable seal integrity, will not unexpectedly disconnect during temperature-programmed analyses.
- Open design allows visual confirmation of the seal for added confidence in the connection.



Combine the simplicity of a "Y" Press-Tight® connector with the strength of a metal union. The ferrules and knurled nuts hold the fused silica tubing in place, which prevents the tubing from unexpectedly disconnecting, even at temperatures as high as 400°C.



The SeCure™ "Y" Connector's open design allows visual confirmation of the seal; secondary seals ensure a leak-tight connection.



The SeCure™ "Y" connector allows dual-column confirmational analysis with a single injection—one of the connector's many uses.

Kits include: SeCure™ "Y" connector body, 3 knurled nuts, "Y" Universal Press-Tight® union, 3 ferrules.

Description	Ferrules Fit Column ID	qty.	cat.#
SeCure™ "Y" Connector Kit	0.25/0.28mm	kit	20276
SeCure™ "Y" Connector Kit	0.28/0.32mm	kit	20277
SeCure™ "Y" Connector Kit	0.45/0.53mm	kit	20278
Knurled nut		3-pk.	20279

for **more** info
See **page 43** for Universal "Y" Press-Tight® Connectors.

Graphite Ferrules for SeCure™ "Y" Connectors

- High-purity, high-density graphite.
- Stable to 450°C.
- No binders that can off-gas or adsorb analytes.
- Smooth surface and clean edges.

Ferrule ID	Fits Column ID	Graphite 10-pk.	Graphite 50-pk.
0.4mm	0.25/0.28mm	20200	20227
0.5mm	0.28/0.32mm	20201	20228
0.8mm	0.45/0.53mm	20202	20224

Kit installs easily, without special tools or plumbing.

restek
innovation!

Change columns in minutes—without venting!

EZ No-Vent™ GC Column-Mass Spectrometer Connector

- Change GC/MS columns in minutes without venting—100µm transfer line throttles vacuum and prevents MS venting.
- Easy to install and maintain—no special tools or plumbing required.
- Gold-plated body for inertness.
- Deactivated transfer line keeps analytes focused; high-temperature polyimide ferrules eliminate leaks at the problematic transfer line fitting.
- Lower cost than other "no-vent" fittings.
- Available for Agilent GCs with 5971/5972 or 5973 GC/MS and Varian Saturn 2000 Series Mass Spectrometers.

We designed the EZ No-Vent™ GC column-mass spectrometer connector to be simple and easy to use. After studying user feedback concerning our EZ-Vent™ 2000 connector, we re-engineered the connector fitting for even better performance. A critical orifice in the EZ No-Vent™ connector minimizes the amount of oxygen allowed into the MS source, eliminating the need for purge gas and enabling you to skip the lengthy vent and pump-down cycle otherwise required when you make a column change. This can save nearly a day of downtime with each column change. The EZ No-Vent™ connector easily attaches to the MS source without special tools or extra plumbing. Figure 1 shows the internal structure of the connector.

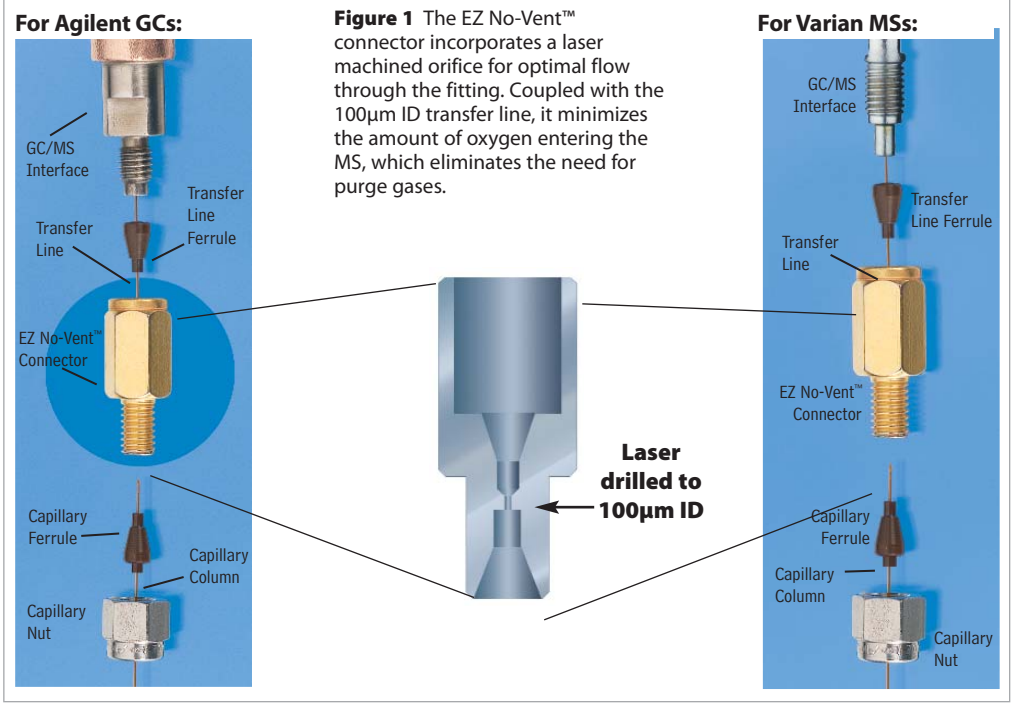


Figure 1 The EZ No-Vent™ connector incorporates a laser machined orifice for optimal flow through the fitting. Coupled with the 100µm ID transfer line, it minimizes the amount of oxygen entering the MS, which eliminates the need for purge gases.

Description	qty.	cat.#
EZ No-Vent™ Connector Kit for Agilent 5971/5972 and 5973 GC/MS Kit includes: EZ No-Vent™ Connector, two 0.4mm ID ferrules for capillary column, two 0.4mm ID ferrules for transfer line, 100µm deactivated transfer line (3 ft.), column plug, column nut.	kit	21323
EZ No-Vent™ Connector Kit for Varian Saturn 2000 Series MSs Kit includes: EZ No-Vent™ Connector, two 0.4mm ID ferrules for capillary column, two 0.4mm ID ferrules for transfer line, 100µm deactivated transfer line (3 ft.), column plug, column nut.	kit	22423
Replacement ferrules for connecting capillary column to EZ No-Vent™:		
0.4mm ID	2-pk.	21015
0.5mm ID	2-pk.	21016
Replacement ferrules for connecting transfer line to EZ No-Vent™: 0.4mm ID	2-pk.	21043
Replacement 100µm deactivated transfer line	3 ft.	21018
Replacement EZ No-Vent™ Column Nut	5-pk.	21900
Replacement EZ No-Vent™ Plug	2-pk.	21915
Open-End Wrenches (1/4" x 5/16")	2-pk.	20110

Choosing a Stationary Phase

Identifying the appropriate stationary phase for your separation is the most critical step of column selection. The choice of stationary phase should be based on sample solubility and on chemical differences among the sample compounds that can be exploited to separate the analytes of interest (Figure 1).

Sample hydrophobicity also is a major determinant of the separation mode (reversed phase or normal phase). In reversed phase separations, the mobile phase is more polar than the stationary phase, which is traditionally a straight alkyl chain, most often octadecylsilyl, C18 (ODS). The majority of HPLC analyses are performed in reversed phase mode because most analytes of interest can be dissolved in mixtures of water and/or a polar organic solvent such as methanol or acetonitrile.

In normal phase separations, the mobile phase is less polar than the stationary phase (e.g., when bare silica is the stationary phase and hexane with a modifier is the mobile phase). The normal phase mode is used primarily when the reversed phase mode cannot be used, because reversed phase separations generally are more robust, reproducible, and versatile. Note that a stationary phase incorporating both polar and nonpolar functionality can be used in either reversed phase or normal phase mode. Cyano phases (e.g., Ultra, Pinnacle™ DB, or Pinnacle™ II cyano columns) commonly are used in either reversed phase or normal phase mode. Ultra IBD and Allure™ Basix columns also can be used for either reversed phase or normal phase separations. Allure™ PFP Propyl and Ultra PFP columns also display dual functionality.

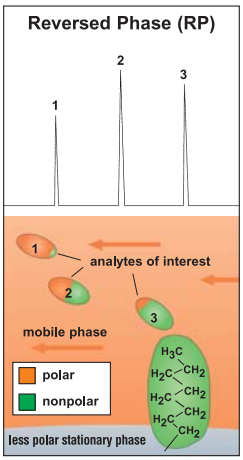
Choose the best stationary phase for your application based on analyte functionality.

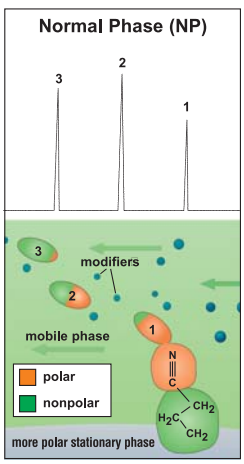
Analyte Functional Group

Acid	Base	Neutral		Mixed Acids, Bases, & Neutrals
		Hydrophobic	Hydrophilic	
Ultra IBD Ultra Aqueous C18 Allure™ Aqueous C18 Allure™ Organic Acids	Allure™ Basix Allure™ PFP Propyl Ultra IBD Ultra PFP Pinnacle™ DB Cyano Ultra Cyano Ultra Amino Pinnacle™ II Amino	Pinnacle™ DB C18 Ultra C18 Pinnacle™ II C18 Allure™ C18 Ultra C8 Pinnacle™ II C8 Pinnacle™ II Phenyl Viva C18 Allure™ Biphenyl	Ultra IBD Ultra Aqueous C18 Allure™ Aqueous C18 Allure™ Organic Acids	Ultra IBD Allure™ Basix

Figure 1
Choice of stationary phase is dependent upon polarity of the sample.

Non-polar analytes are more attracted to the less polar stationary phase. Polar analytes are more strongly retained by the more polar stationary phase.





Pinnacle™ II Phenyl (USP L11)

Physical Characteristics:

particle size: 3µm or 5µm, spherical
pore size: 110Å
carbon load: 6%
endcap: fully endcapped
pH range: 2.5 to 10
temperature limit: 80°C

Chromatographic Properties:

The Pinnacle™ II Phenyl phase offers unique selectivity versus traditional alkyl chain phases, especially for aromatic compounds.

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 52.

Allure™ C18 (USP L1)
Excellent Columns for LC/MS and ELSD

Physical Characteristics:

particle size: 3µm or 5µm, spherical
pore size: 60Å
carbon load: 27%
endcap: fully endcapped
pH range: 2.5 to 7.5
temperature limit: 80°C

Chromatographic Properties:

Most retentive phase for hydrophobic and slightly polar analytes due to large surface area of the base silica and high-density bondings. High-purity packings exhibit excellent peak shapes for a wide range of compounds.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
3µm Columns				
30mm	9164331	9164332	9164333	9164335
50mm	9164351	9164352	9164353	9164355
100mm	9164311	9164312	9164313	9164315
5µm Columns				
30mm	9164531	9164532	9164533	9164535
50mm	9164551	9164552	9164553	9164555
100mm	9164511	9164512	9164513	9164515
150mm	9164561	9164562	9164563	9164565

Allure™ Basix (USP L10)
Excellent Columns for LC/MS and ELSD

Physical Characteristics:

particle size: 3µm or 5µm, spherical
pore size: 60Å
carbon load: 12%
endcap: fully endcapped
pH range: 2.5 to 7.5
temperature limit: 80°C

Chromatographic Properties:

Highly retentive propyl cyano phase. Excellent choice for analytes containing amine group functionality.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
3µm Columns				
30mm	9161331	9161332	9161333	9161335
50mm	9161351	9161352	9161353	9161355
100mm	9161311	9161312	9161313	9161315
5µm Columns				
30mm	9161531	9161532	9161533	9161535
50mm	9161551	9161552	9161553	9161555
100mm	9161511	9161512	9161513	9161515
150mm	9161561	9161562	9161563	9161565

Allure™ Biphenyl

Physical Characteristics:

particle size: 3µm or 5µm, spherical
pore size: 60Å
carbon load: 23%
endcap: yes
pH range: 2.5 to 7.5
temperature limit: 80°C

Chromatographic Properties:

Highly retentive and selective phase for aromatic compounds. Increased retention over phenyl phases; uses high-purity, Type B silica.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
5µm Columns				
30mm	9166531	9166532	9166533	9166535
50mm	9166551	9166552	9166553	9166555
100mm	9166511	9166512	9166513	9166515
150mm	9166561	9166562	9166563	9166565
200mm	9166521	9166522	9166523	9166525
250mm	9166571	9166572	9166573	9166575

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.

Allure™ PFP Propyl
Excellent Columns for LC/MS and ELSD

Physical Characteristics:

particle size: 3µm or 5µm, spherical
pore size: 60Å
carbon load: 17%
endcap: fully endcapped
pH range: 2.5 to 7.5
temperature limit: 80°C

Chromatographic Properties:

A pentafluorophenyl phase with a propyl spacer. Highly retentive for basic analytes. An excellent phase for separating nucleosides, nucleotides, purines, pyrimidines, halogenated compounds, β-blockers, and tricyclic antidepressants.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
3µm Columns				
30mm	9169331	9169332	9169333	9169335
50mm	9169351	9169352	9169353	9169355
100mm	9169311	9169312	9169313	9169315
5µm Columns				
30mm	9169531	9169532	9169533	9169535
50mm	9169551	9169552	9169553	9169555
100mm	9169511	9169512	9169513	9169515
150mm	9169561	9169562	9169563	9169565

Ultra C18 (USP L1)

Physical Characteristics:

particle size: 3µm or 5µm, spherical
pore size: 100Å
carbon load: 20%
endcap: fully endcapped
pH range: 2.5 to 7.5
temperature limit: 80°C

Chromatographic Properties:

A 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
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 52.

for **more** info

Restek offers an extensive line of GC and HPLC columns, accessories, and replacement parts. Call to request our general catalog, or our HPLC products catalog, for a full listing of products, or visit us on the web at www.restek.com

CLINICAL/FORENSICS

Ultra Aqueous C18 (USP L1)

Physical Characteristics:

particle size: 3µm or 5µm, spherical
pore size: 100Å
endcap: no
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
250mm	9178571	9178572	9178573	9178575

free literature

HPLC Analysis of Vitamins

lit. cat.# 59181

Analyze Polar Compounds by Reversed Phase HPLC Using Ultra Aqueous C18 Columns

lit. cat.# 59177

Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

Ultra IBD
Specialized Columns for Mixed Polar and Nonpolar Compounds

Physical Characteristics:

particle size: 3µm or 5µm, spherical
pore size: 100Å
carbon load: 12%
endcap: no
pH range: 2.5 to 7.5
temperature limit: 80°C

Chromatographic Properties:

An intrinsically base-deactivated (IBD) phase, containing a polar group within, or intrinsic to, the hydrocarbon bonded phase. Unique selectivity and a high level of base deactivation, while reducing or eliminating the need for mobile phase additives.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
3µm Columns				
30mm	9175331	9175332	9175333	9175335
50mm	9175351	9175352	9175353	9175355
100mm	9175311	9175312	9175313	9175315
5µm Columns				
30mm	9175531	9175532	9175533	9175535
50mm	9175551	9175552	9175553	9175555
100mm	9175511	9175512	9175513	9175515
150mm	9175561	9175562	9175563	9175565

free literature

Excellent LC/MS Separation of Penicillins and Cephalosporins Using Ultra IBD Columns

lit. cat.# 59133

Analyze Nucleotides, Nucleosides, Purines, and Pyrimidines Simultaneously with the Ultra IBD Column

lit. cat.# 59141

The Ultra IBD Column Allows HPLC Separation of Polar and Non-Polar Analytes from the Same Sample

lit. cat.# 59512

Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

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

Also order the appropriate XG-XF fitting - see page 52.

For guard cartridges for these columns, see page 52.

Ultra Cyano (USP L10)

Physical Characteristics:

particle size: 3µm or 5µm, spherical
pore size: 100Å
carbon load: 8%
endcap: fully endcapped
pH range: 2.5 to 7.5
temperature limit: 80°C

Chromatographic Properties:

High-purity cyano phase with few silanol sites. Often a better choice than C18 phases for basic pharmaceuticals, especially regarding peak shape and selectivity. Cyano phases are more rugged than bare silica for normal phase analyses because they are less sensitive to small amounts of water present in the mobile phase.

Length	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
3µm Columns				
30mm	9106331	9106332	9106333	9106335
50mm	9106351	9106352	9106353	9106355
100mm	9106311	9106312	9106313	9106315
5µm Columns				
30mm	9106531	9106532	9106533	9106535
50mm	9106551	9106552	9106553	9106555
100mm	9106511	9106512	9106513	9106515
150mm	9106561	9106562	9106563	9106565

Ultra Phenyl (USP L11)

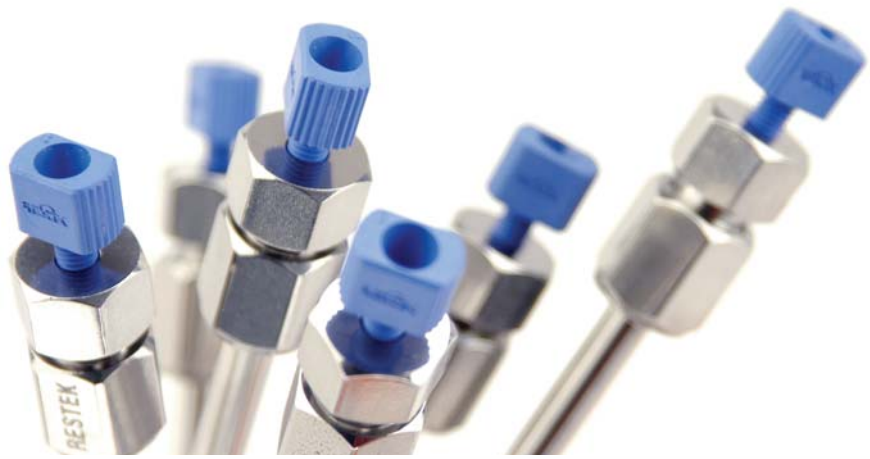
Physical Characteristics:

particle size: 3µm or 5µm, spherical
pore size: 100Å
carbon load: 10%
endcap: fully endcapped
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



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 gainst irreversibly adsorbed compounds.

Trident™ Direct Guard Column System

Easy to Use, Low Dead Volume—The Ultimate Combination of Convenience and Column Protection

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

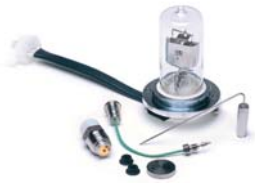
*The standard PEEK® tip in Trident™ Direct systems is compatible with Parker®, Upchurch®, Valco®, and other CPI-style fittings. To use Trident™ Direct systems with Waters®-style end fittings, replace the tip with cat.# 25088.

10 & 20mm Guard Cartridges

Guard 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™ Basix	916150212	916150210	916150222	916150220
Allure™ C18	916450212	916450210	916450222	916450220
Allure™ PFP Propyl	916950212	916950210	916950222	916950220
Pinnacle™ II Phenyl	921550212	921550210	921550222	921550220
Ultra Aqueous C18	917850212	917850210	917850222	917850220
Ultra C18	917450212	917450210	917450222	917450220
Ultra Cyano	910650212	910650210	910650222	910650220
Ultra IBD	917550212	917550210	917550222	917550220
Ultra Phenyl	910550212	910550210	910550222	910550220

Genuine Restek Replacement Parts for Agilent HPLC Systems

Description	Model #	Similar to Agilent part #	qty.	cat.#
Preventive Maintenance Kit (Includes: rotor seal, needle seat, needle assembly, seat cap)	1050	01078-68721	kit	25259
Autosampler Preventive Maintenance Kit (Includes: rotor seal, needle assembly, needle seat)	1100	G1313-68709	kit	25271
Pump Maintenance Kit (Includes: PTFE frit, outlet cap, active inlet cartridge, gold disk seal, 2 piston seals, glass solvent filter)	1050 & 1100	G1311-68710	kit	25270
Outlet Ball Valve, Binary Pump	1100	G1312-60012	ea.	25267
Outlet Ball Valve	1050 & 1100	G1311-60012	ea.	25276
Sieves for Outlet Valve	1050 & 1100	5063-6505	10-pk.	25266
Check Valve Cartridge Assembly	1090	79835-67101	ea.	25344
Piston Seals, Teflon® w/Graphite	1050 & 1100	5063-6589	2-pk.	22482
Piston Seals, Teflon® w/Graphite	1050 & 1100	5063-6589	10-pk.	22483
Piston Seals (Black)	1090	5062-2494	4-pk.	25347
Seal Wash Kit, Binary Pump (4 seals, 4 gaskets)	1100	—	kit	25268
Seal Wash Kit (2 seals, 2 gaskets)	1100	—	kit	25269
Wash Seal	1050 & 1100	0905-1175	ea.	25277
Sapphire Piston	1050 & 1100	5063-6586	ea.	25273
Sapphire Piston	1090	6980-0672	ea.	25345
Needle Seat	1050	79846-67101	ea.	25258
Needle Seat	1090	79846-67101	ea.	25348
Needle Seat Assembly	1100	G1313-87101	ea.	25265
Needle Assembly	1100	G1313-87201	ea.	25278
Rotor Seal (not for use with 7125 injection valve)	1050	0101-0626	ea.	25272
Rotor Seal	1100	0100-1853	ea.	25275
Rotor Seal (Rheodyne®-style)	1090	0101-0623	ea.	25349
Frits, PTFE	1050 & 1100	01018-22707	5-pk.	25466
Seal, Gold Disk (outlet)	1050 & 1100	5001-3707	ea.	25467
Detector Lamp, 1090 DA, 1050 VW/DA/MWD	1090, 1050	79883-60002	ea.	25260
Lamp, DAD G1315A, G1365A	1100	2140-0590	ea.	25261
Lamp, VWD G1314A	1100	G1314-60100	ea.	25262
8453 Deuterium Lamp	—	2140-0605	ea.	25263
G1321 Fluorescence Detector Flash Lamp	—	2140-0600	ea.	25264
Lamp, DAD Long Life Deuterium (2000 hours)	1100	5181-1530	ea.	25399



for **more** info

Genuine Restek Replacement Parts for HPLC Systems

lit. cat.# 59012A

Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your Restek representative, to request your free copy!

Genuine Restek Replacement Parts for Thermo Separation HPLC Systems

Description	Model #	Similar to TSP/SP part #	qty.	cat.#
Inlet Check Valve Assembly	SP8800 & P-Series Pumps	A3495-010	ea.	25474
Outlet Check Valve Assembly	SP8800 Series Pumps	A3490-010	ea.	25475
Piston	SP8800 & P-Series Pumps	A3102-010	ea.	25476
Back-up Seal	SP8800 & P-Series Pumps	A2963-010	ea.	25477
Plunger Seal, Gold Superseal	SP8800 & P-Series Pumps	A2962-010	ea.	25478
Check Valve and Transducer Assembly	P-Series Pumps	A3990-010	ea.	25479
Kel-F® Washer	P-Series Pumps	A2973-010	ea.	25480
Rotor Seal Assembly, Rheodyne® 7010	TSP AS100, 300, 1000, 3000, 3500, 8875, and 8880 Autosamplers	7010-039	ea.	25481
Syringe Assembly, 250µL	TSP AS100, 300, 1000, 3000, 3500, 8875, and 8880 Autosamplers	A3588-020	ea.	25482
Syringe, 500µL	TSP AS100, 300, 1000, 3000, 3500, 8875, and 8880 Autosamplers	A3588-010	ea.	25483
Lamp, UV	Linear UV-200, 203, 204, 205, 206, and UV 100, 150, 1000, and 2000 Detectors	9551-0023	ea.	25484

Description	Model #	Similar to TSP part #	qty.	cat.#
Check Valve Cartridge	LDC Constametric Pumps	900946	ea.	25485
Sapphire Plunger	LDC Constametric Pumps	801306	ea.	25486
Plunger Seal Kit, Gold	LDC Constametric Pumps	31-36-00754	ea.	25487
Plunger Seal, Black	LDC Constametric Pumps	206129001	ea.	25488
Plunger Seal, Gold	LDC Constametric Pumps	206156001	ea.	25489
Lamp, Deuterium	LDC SM-I, II, III, 3000, 3100, 3100X, and 4000 Detectors	108035	ea.	25490
Lamp, Deuterium Pre-aligned	LDC 3200 and 4100 Detectors	900918001	ea.	25491

did you **know**?

We also offer Genuine Restek Replacement Parts for Beckman, Hitachi, PerkinElmer, and Shimadzu systems. Please refer to our current catalog - or visit our website.

Genuine Restek Replacement Parts for Waters HPLC Systems

Description	Model #	Similar to Waters part #	qty.	cat.#
Preventative Maintenance Kit Includes: sparge diffuser, filter insert, compression screws, SS ferrule, battery for 2690/717, 250µL WISP™ syringe, seal wash plunger seal kits (2), wash tube seal kits (4), 2690 seal pack rebuild kit (steel bodies not included), 2690 head plunger seal kits (2), solvent reservoir 20µm filters (4), Alliance® check valve cartridges (2), Alliance® plunger assemblies (2), 2690 face seals (4)	Alliance® 2690, 2695	WAT270944	kit	25143
Preventative Maintenance Kit Includes: PerformancePLUS™ cartridges (2), sparge diffusers (4), Super Seals™ (2), solvent reservoir 20µm filters (2), sapphire plungers (2), reference valve rebuild kit, inlet manifold kit	600 Pump	WAT052675	kit	25144
Preventative Maintenance Kit Includes: 717 seal pack with needle, filter insert, 250µL WISP™ syringe	717 Autosampler	WAT052669	kit	25145
Preventative Maintenance Kit Includes: 616/326/625 plunger assemblies (2), pump seal kit, sparge diffusers (4), solvent reservoir 20µm filters (4), 616 cartridge assemblies (4)	616 Pump	WAT052672	kit	25146
Inlet Check Valve Assembly	M6KA, 501, 510, 515, 590, 600E	33679, 25214	ea.	25360
Inlet Check Valve Housing	M6KA, 501, 510, 515, 590, 600E	25203	ea.	25361
Inlet Check Valve Rebuild Kit	M6KA, 501, 510, 515, 590, 600E	60495	2-pk.	25362
Outlet Check Valve Assembly (Actuator Style)	M6KA, 501, 510, 515, 590, 600E	25030	ea.	25363
Outlet Check Valve Housing (Actuator Style)	M6KA, 501, 510, 515, 590, 600E	25212	ea.	25364
Outlet Check Valve Rebuild Kit (Actuator Style)	M6KA, 501, 510, 515, 590, 600E	26016	2-pk.	25365
Outlet Check Valve Assembly (Ball & Seat Style)	M6KA, 501, 510, 515, 590, 600E	25216	ea.	25366
Outlet Check Valve Housing (Ball & Seat Style)	M6KA, 501, 510, 515, 590, 600E	25207	ea.	25367
Outlet Check Valve Rebuild Kit (Ball & Seat Style)	M6KA, 501, 510, 515, 590, 600E	26014	2-pk.	25368
Inlet Check Valve Assembly, 225µL (Extended Flow)	M6KA, 501, 510, 515, 590, 600E	60307	ea.	25369
PerformancePLUS™ Check Valve Cartridge	M6KA, 501, 510, 515, 590, 600E	700000254	2-pk.	25370
Check Valve Rebuild Kit (Extended Flow)	M6KA, 501, 510, 515, 590, 600E	88223	2-pk.	25371
PerformancePLUS™ Check Valve Housing	M6KA, 501, 510, 515, 590, 600E	—	ea.	25372
Check Valve Cartridges	Alliance®	WAT270941	2-pk.	25373
Super Seal™ for Analytical Heads	M6KA, 501, 510, 515, 590, 600E	22946, 22934	ea.	25374
Plunger Seal, Gold for Analytical Heads*	M6KA, 501, 510, 515, 590, 600E	22934	ea.	25375
Plunger Seal, Black for graphite-filled Teflon®	M6KA, 501, 510, 515, 590, 600E	26613	ea.	25378
Plunger Seal, Black for EF Heads	510, 590, 600E	26644	ea.	25379
Plunger Seal, Gold for EF Heads	510, 590, 600E	26644	ea.	25380
Seal Wash Plunger Seal	Alliance®	WAT271018	2-pk.	25386
Head Plunger Seal Kit	Alliance®	WAT270938	2-pk.	25387
Head Plunger Seal Kit (Black)	Alliance®	WAT271066	2-pk.	25388
Insert Seal Parts Kit	M6KA, 501, 510, 515, 590, 600E	60012	kit	25389
Sapphire Plunger	M6KA, 510, 590, 600	25656	ea.	25381
Sapphire Plunger (Extended Flow)	510, 590, 600E	60304	ea.	25382
Sapphire Plunger	M45, M501	26524	ea.	25383
Sapphire Plunger	M515	WAT207069	ea.	25384
Sapphire Plunger	616, 625, 626	31788	ea.	25420
Sapphire Plunger	Alliance®	WAT270959	ea.	25385
Single Solvent Inlet Manifold	600E	60034, 60042	ea.	25390
Pressure Transducer	M6KA, 501, 510, 515, 590, 600E	60328	ea.	25391
Draw-Off Tube Assembly	M6KA, 501, 510, 515, 590, 600E	25470	ea.	25392
1/16" Stainless Steel TEE	M6KA, 501, 510, 515, 590, 600E	75215	ea.	25411
Inlet Manifold Kit	M45, 501, 510, 590, 600E	60448	kit	25412
Ferrule, Stainless Steel	515	22330	ea.	25417
Gradient Proportioning Valve, 9Volt	600E	34423	ea.	25418
Gradient Proportioning Valve, 12Volt	600E	62037	ea.	25419
Wash Face Seal	Alliance® 2690	WAT271017	ea.	25428
Wash Tube Seal Kit	Alliance® 2690	WAT270940	4-pk.	25429
Proportioning Valve	Alliance® 2690	WAT270927	ea.	25430

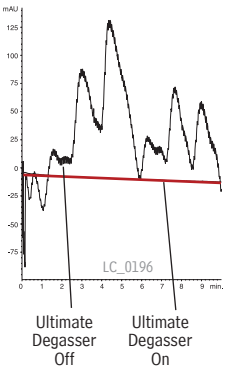
*Ultra-high molecular weight polyethylene (UHMWPE).

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. Most in-line degassers withdraw gas across a gas-permeable PTFE tubing membrane, 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.

Degasys Ultimate Degasser provides highly stable baselines



Mobile Phase: water:methanol 50:50
Flow: 1.0 mL/min.
Det.: UV @ 210nm

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

Not recommended for use with fluorinated mobile phases, including mobile phases containing TFA.

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. 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

Sidewinder™ Column Heater

- Easy to set up!
- Operation range: 5°C above ambient to 85°C, ±1°C.
- Lightweight, compact design fits in small spaces.
- Column holder can be placed in any orientation.

This unique design completely encloses any HPLC analytical column up to 25cm in length. Two lengths of heater jackets are available: the short column holder accommodates columns up to 10cm in length, while the long column holder 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. The new Sidewinder™ controller has fast 10Hz sampling for improved responsiveness. Power requirements: 24V control unit for maximum stability; RS232 control allows external programming.

Description	qty.	cat.#
Temperature Control Module and Long Column Holder, 25cm Holder	ea.	26516
Temperature Control Module and Short Column Holder, 10cm Holder	ea.	26517

Sidewinder™ Heater/Cooler Temperature Control Module

- Operation range: 5 - 55°C, ±0.2°C.
- Ability to program multiple temperature points.
- Accommodates columns up to 30cm in length and 7.8mm ID.
- Compact design.

The Sidewinder™ heater/cooler unit has a doubly insulated cover to maintain the programmed temperature to within 0.2°C. The 24V control unit provides maximum stability and rapid equilibration times; RS232 control allows external programming.

Description	qty.	cat.#
Sidewinder™ Heater/Cooler Temperature Control Module	ea.	26518

All Sidewinder™ temperature control products carry the value recognized CE mark. Each unit meets the demanding electromagnetic emission standards of the new European Union Directives, United States standards, and Canadian standards.

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

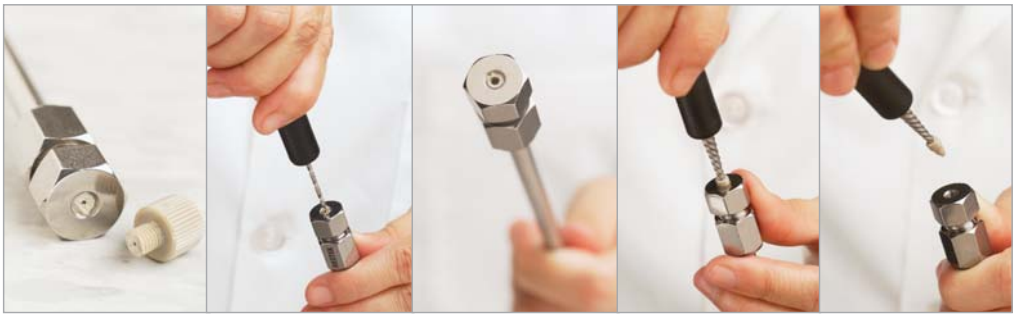
pinnacle™ II **hplc** columns

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)

PEEK® Fitting Extractor

Drill into the broken fitting, then screw the extractor into the fitting and remove it easily.



Description	qty.	cat.#
PEEK® Fitting Extractor	ea.	25325

PEEK® Union Connector

Allows you to quickly and reliably connect two pieces of 1/16-inch tubing. 0.3mm union bore. End fittings included.

Description	qty.	cat.#
PEEK® Union Connector 1/16"	2-pk.	25323

Zero-Dead-Volume Internal Union

Restek is pleased to offer a selected line of Valco® fittings for connecting fused silica or 1/16- or 1/8-inch metal tubing. For over 30 years, Valco® Instrument Co. has been the leading designer and manufacturer of valves and fittings for precision analytical instrumentation. Valco® fittings have been designed for the specific demands of instrumentation and manufactured with the tight tolerances and premium quality that analysts require. If you don't see the Valco® fitting you need, please request special ordering information.

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

Universal 10-32 PEEK® Column Connectors and Plugs

Universal PEEK® Connectors allow easy installation of all 1/16-inch tubing, including stainless steel.

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

Crimp-Top Vials, 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

11mm Aluminum Crimp Seals with Septa

Description	100-pk.	500-pk.	1000-pk.
Silver Seal, PTFE/Natural Rubber Septum	21174	—	21175
Blue Seal, PTFE/Natural Rubber Septum	24351	—	24352
Green Seal, PTFE/Natural Rubber Septum	24353	—	24354
Red Seal, PTFE/Natural Rubber Septum	24355	—	24356
Yellow Seal, PTFE/Natural Rubber Septum	24357	—	24358
Mixed Colors, PTFE/Natural Rubber Septum	—	21724	—
Silver Seal, PTFE/Silicone Septum	24359	—	24360
Blue Seal, PTFE/Silicone Septum	24361	—	24362
Green Seal, PTFE/Silicone Septum	24363	—	24364
Red Seal, PTFE/Silicone Septum	24365	—	24366
Yellow Seal, PTFE/Silicone Septum	24367	—	24368
Mixed Colors, PTFE/Silicone Septum	—	21725	—
Silver Seal, PTFE/Silicone/PTFE Septum**	24369	—	24370

Convenience Kits: Vials, Caps, & Septa

Vials packaged in a clear-lid tray. Caps with septa packaged in a plastic bag.

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, Flat Bottom Insert w/ID Ring

Glass, Limited Volume Insert with Bottom Spring

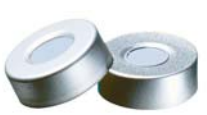
Limited Volume Inserts for 2mL Crimp-Top & Short-Cap, Screw-Thread Vials

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	—

6.0mL Headspace Vial with PTFE/Silicone Seal

Headspace Autosampler Vials*

Description	100-pk.	1000-pk.
a) 6mL Clear Vial	21166	21167
b) 10mL Clear Vial, Flat Bottom	24683	24684
c) 10mL Clear Vial, Rounded Bottom	21164	21165
d) 20mL Clear Vial, Flat Bottom	24685	24686
e) 20mL Clear Vial, Rounded Bottom	21162	21163
f) 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

*Colored marking spots available on request in blue, green, rust, or yellow (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	Hamilton cat.#	Hamilton 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	SGE cat.#	SGE 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

- Most popular gauge for Agilent 7673.
- Best for Merlin Microseal® septum and standard septum-equipped GCs.
- For packed column injection ports.
- For split/splitless injection ports.

26s—Single Gauge Needle

- For on-column injection ports.
- For 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.

Guide to Needle Termination Codes

Hamilton:
(ASN) Autosampler Cemented Needle

SGE:
(F) Fixed Needle

Syringes for Agilent 1090 & 1100 LC Autosamplers

25µL, 1/4-32 UNEF Thread

SGE Syringes

Volume	SGE Model	SGE cat.#	SGE qty.	Restek cat.#
25µL	25D-HP1090-GT	003670	ea.	22290
250µL	250D-HP1090-GT	006670	ea.	22291

ordering **note**

Hamilton and SGE syringes are in stock and available for same-day shipment.

Syringes for Waters WISP® LC Autosamplers

250µL, 1/4-28 UNF Thread

SGE Syringes

Volume	SGE Model	SGE cat.#	SGE qty.	Restek cat.#
25µL	25D-WISP	003990	ea.	22293
250µL	250D-WISP	006690	ea.	22294

All cartridges are polypropylene and have polyethylene frits unless otherwise noted.

Resprep™ SPE Cartridges: Normal Phase

Hydrophilic (polar) adsorbents used to extract hydrophilic analytes from nonpolar matrices, such as organic solvents (e.g., polar contaminants from sample extracts).

	3mL/200mg (50-pk.)	3mL/500mg (50-pk.)	6mL/500mg (30-pk.)	6mL/1000mg (30-pk.)
Florisil®	—	24031	—	24034
(EPA SW 846 methods and CLP protocols)	—	24032*	26086**	26085**
Silica (EPA SW 846 methods)	—	24035	—	24038
	—	24036*	—	—
Cyanopropyl (endcapped)	26048	26049	—	—

*Teflon® frits

**Glass tubes with Teflon® frits

Strong Anion Exchange (SAX)
Strong Cation Exchange (SCX)
Weak Anion Exchange (WAX)
Weak Cation Exchange (WCX)

Resprep™ SPE Cartridges: Ion Exchange Phases

Ionized adsorbents used to extract positively- or negatively-charged analytes from aqueous matrices (e.g., tricyclic antidepressants from plasma).

	1mL/100mg (100-pk.)	3mL/200mg (50-pk.)	3mL/500mg (50-pk.)	6mL/500mg (30-pk.)	6mL/1000mg (30-pk.)
SAX, quaternary amine	26054	—	26055	—	—
SCX, propyl	26056	—	26057	—	—
SCX, benzene	—	26058	—	26059	26060
WAX, amino, primary amine	26050	26051	26052	26053	—
WCX, carboxylic acid	26061	—	26062	—	—

Syringe Filters

- Nylon - PTFE - PVDF membranes.
- 13mm or 25mm diameter.
- 0.22µm or 0.45µm porosity.
- Color coded for easy identification.
- 100 filters, reusable storage container.

Excellent syringe filters—great prices!

	Size	Porosity	qty.	cat.#
Nylon	13mm	0.22µm	100-pk.	26146
	13mm	0.45µm	100-pk.	26147
	25mm	0.22µm	100-pk.	26148
	25mm	0.45µm	100-pk.	26149
PTFE (polytetrafluoroethylene)	13mm	0.22µm	100-pk.	26142
	13mm	0.45µm	100-pk.	26143
	25mm	0.22µm	100-pk.	26144
	25mm	0.45µm	100-pk.	26145
PVDF (polyvinyldifluoride)	13mm	0.22µm	100-pk.	26150
	13mm	0.45µm	100-pk.	26151
	25mm	0.22µm	100-pk.	26152
	25mm	0.45µm	100-pk.	26153

Resprep™ SPE Cartridges: Bonded Reversed Phases

Hydrophobic (nonpolar) adsorbents, used to extract hydrophobic analytes from polar matrices (e.g., pesticides from water).

	1mL 100mg (100-pk)	3mL 200mg (50-pk.)	3mL 500mg (50-pk.)	6mL 500mg (30-pk.)	6mL 1000mg (30-pk.)	20mL 5g (20-pk.)	60mL 10g (16-pk.)
C18 (high load, endcapped)	26030	26031	24050	24052	24051	26034	26035
C8 (high load, endcapped)	26036	26037	26038	26039	26040	—	—
C2 (endcapped)	26041	26042	—	—	—	—	—
Cyclohexyl (endcapped)	—	—	—	—	26043	—	—

Specialty SPE Cartridges

These cartridges have been specifically designed to provide consistent and reproducible results for the method stated.

Description	Applications	Tube Volume, Bed Weight	qty.	cat.#
Drug Prep I	Acidic, basic, and/or neutral drugs. Mixed-mode hydrophobic and ion exchange sites provide selective, reproducible extraction of biological samples containing therapeutic or illegal drugs.	3mL, 200mg	50-pk.	26044
		10mL, 200mg	50-pk.	26045
Drug Prep II	Extraction of THCA from biological samples. Copolymeric anion exchange sites provide selective, reproducible extraction of THCA from urine.	3mL, 200mg	50-pk.	26046
		10mL, 200mg	50-pk.	26047

Exempted Drug of Abuse Reference Materials

1,000µg/mL in P&T methanol (*except where noted), 1mL/ampul

Compound	Individual cat.#
Benzodiazepines	
alprazolam	34042
bromazepam	34043
chlordiazepoxide	34044
clobazam	34045
clonazepam	34046
diazepam	34047
flunitrazepam	34049
flurazepam	34050
lorazepam	34051
nitrazepam	34053
oxazepam	34054
prazepam	34055
temazepam	34056
triazolam	34057
Cocaine & Metabolites	
cocaine	34015
benzoylecgonine	34016
ecgonine	34017
ecgonine methyl ester	34018
Methadone & Metabolites	
methadone	34005
Amphetamines & Metabolites	
d-amphetamine	34020
(+)methamphetamine	34021
Opiates & Metabolites	
codeine	34000
hydrocodone	34002
hydromorphone	34063
morphine	34006
oxycodone	34007
oxymorphone	34065
Cannabinoid & Metabolites	
cannabidiol	34011
cannabinol	34010
Barbiturates	
amobarbital	34028
aprobarbital	34029
barbital	34030
butabarbital	34031
butalbital	34032
DL-glutethimide	34058
hexobarbital	34033
mephobarbital	34034
methohexital	34035
pentobarbital	34036
phenobarbital	34037
secobarbital	34038
talbutal	34039
thiamylal	34040
thiopental	34041
Other	
benzphetamine	34022
cocaethylene*	34066
fenfluramine	34023
levorphanol	34003
meperidine	34004
meprobamate	34059
methaqualone	34064
methyprylon	34060
pentazocine	34062
phencyclidine	34027
phendimetrazine	34025
phenmetrazine	34026
phentermine	34024
dextro-propoxyphene	34008
thebaine	34009

*1,000µg/mL in acetonitrile.
No datapacks available.

Single-Component Explosives Solutions

These materials support nitroaromatic, nitramine, and nitroester analyses by GC-ECD (Method 8095[1,2]. Compounds listed are explosives, manufacturing intermediates or degradation products. Method 8095 includes Method 8330 target compounds, plus 3,5-dinitroaniline, nitroglycerin, and pentaerythritol tetranitrate (PETN). Method 8095 mixtures contain the additional components at concentration ratios appropriate for ECD.

Compound Packaged 1mL/ampul	CAS#	Solvent Code	Solvent Concentration	Individual cat.#
2-amino-4,6-dinitrotoluene	35572-78-2	ACN	1,000	31670
4-amino-2,6-dinitrotoluene	19406-51-0	ACN	1,000	31671
3,5-dinitroaniline	618-87-1	ACN	1,000	31661
1,3-dinitrobenzene	99-65-0	ACN	1,000	31662
2,4-dinitrotoluene	121-14-2	ACN	1,000	31663
2,6-dinitrotoluene	606-20-2	ACN	1,000	31664
EGDN	628-96-6	M	1,000	31601
HMX	2691-41-0	ACN	1,000	31665
nitrobenzene	99-95-3	ACN	1,000	31657
nitroglycerin	55-63-0	M	1,000	31498
nitroguanidine	556-88-7	M	1,000	31602
2-nitrotoluene	88-72-2	ACN	1,000	31659
3-nitrotoluene	99-08-1	ACN	1,000	31660
4-nitrotoluene	99-99-0	ACN	1,000	31658
PETN	78-11-5	M	1,000	31600
picric acid	88-89-1	M	1,000	31499
propylene glycol dinitrate (PGDN)	6423-43-4	M	1,000	31821
RDX	121-84-4	ACN	1,000	31666
tetryl	479-45-8	ACN	1,000	31667
1,3,5-trinitrobenzene	99-35-4	ACN	1,000	31668
2,4,6-trinitrotoluene	118-96-7	ACN	1,000	31669

ACN=acetonitrile
M = methanol

References (Not available from Restek.)

[1]US Environmental Protection Agency. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods.* SW-846, Proposed Draft Update IVB, Office of Solid Waste, Washington, DC, 1999.
[2]M. E. Walsh, T. Ranney, J. Chromatogr. Sci., Vol. 36, pp. 406-416, August 1998.

free data packs

Restek offers free downloadable data packs for analytical reference material products.
Just visit our website at **www.restek.com/datapacks**. Enter the catalog number and lot number for the product you ordered and obtain a printable pdf file.

please note

We can custom prepare weathered accelerants for fire debris analysis.

Please complete the custom reference material request form on **page 64, or online.**

We'll be glad to work with you!

Single Source Weathered Petrochemical Standards

These solutions are prepared from a single source (one refinery) product. The weathered materials indicate the percent weight loss from the original material. Samples of regular and premium grade unleaded gasoline were collected, then blended in equal volumes.

There are four general types of mineral spirits, classified according to boiling point range (BPR):

- Type I (Stoddard solvent) BPR 149–182°C
- Type II (high flash point) BPR 177–196°C
- Type III (odorless) BPR 149–196°C
- Type IV (low dry point) BPR 149–174°C

The mineral spirit solutions listed below were prepared from an equal volume blend of Type I, II, and III mineral spirits.

Compound	cat.# (ea.)
5,000µg/mL in P&T methanol, 1mL/ampul	
unleaded gasoline: unweathered	30096
unleaded gasoline: 25% weathered	30097
unleaded gasoline: 50% weathered	30098
unleaded gasoline: 75% weathered	30099
unleaded gasoline: 99% weathered	30436
5,000µg/mL in methylene chloride, 1mL/ampul	
kerosene: unweathered	31229
kerosene: 25% weathered	31230
kerosene: 50% weathered	31231
kerosene: 75% weathered	31232
5,000µg/mL in methylene chloride, 1mL/ampul	
diesel fuel #2: unweathered	31233
diesel fuel #2: 25% weathered	31234
diesel fuel #2: 50% weathered	31235
diesel fuel #2: 75% weathered	31236
5,000µg/mL in methylene chloride, 1mL/ampul	
mineral spirits: unweathered	31225
mineral spirits: 25% weathered	31226
mineral spirits: 50% weathered	31227
mineral spirits: 75% weathered	31228
50,000µg/mL in methylene chloride, 1mL/ampul	
mineral spirits: unweathered	31260
mineral spirits: unweathered (5mL/ampul)	31261

Stoddard Solvent Standard

10,000µg/mL in P&T methanol, 1mL/ampul
cat. # 30487 (ea.)

Weathered Gasoline Kit

kit

30096: Unleaded Gasoline Standard
30097: Unleaded Gas Standard: 25% Weathered
30098: Unleaded Gas Standard: 50% Weathered
30099: Unleaded Gas Standard: 75% Weathered

Contains 1mL each of these mixtures.
cat. # 30100 (kit)

Weathered Gasoline Kit #2

kit

30096: Unleaded Gasoline Standard
30097: Unleaded Gas Standard: 25% Weathered
30098: Unleaded Gas Standard: 50% Weathered
30099: Unleaded Gas Standard: 75% Weathered
30436: Unleaded Gas Standard: 99% Weathered

Contains 1mL each of these mixtures.
cat. # 30437 (kit)

Weathered Kerosene Kit

kit

31229: Kerosene Standard
31230: Kerosene Standard: 25% Weathered
31231: Kerosene Standard: 50% Weathered
31232: Kerosene Standard: 75% Weathered

Contains 1mL each of these mixtures.
cat. # 31238 (kit)

Weathered Diesel Fuel #2 Kit

kit

31233: Diesel Fuel #2 Standard
31234: Diesel Fuel #2 Standard: 25% Weathered
31235: Diesel Fuel #2 Standard: 50% Weathered
31236: Diesel Fuel #2 Standard: 75% Weathered

Contains 1mL each of these mixtures.
cat. # 31239 (kit)

Weathered Mineral Spirits Kit

kit

31225: Mineral Spirits Standard
31226: Mineral Spirits Standard: 25% Weathered
31227: Mineral Spirits Standard: 50% Weathered
31228: Mineral Spirits Standard: 75% Weathered

Contains 1mL each of these mixtures.
cat. # 31237 (kit)

ASTM E1387-95 and E1618-97 Fire Debris Analysis

These materials also can be used for underground storage tank monitoring.

E1387-95 Column Resolution Check Mix

(13 components)

n-hexane (C6)
n-octane (C8)
n-decane (C10)
n-dodecane (C12)
n-tetradecane (C14)
n-hexadecane (C16)
n-octadecane (C18)
n-eicosane (C20)
2-ethyltoluene
3-ethyltoluene
toluene
1,2,4-trimethylbenzene
p-xylene

2,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31224 (ea.)

E1618-97 Test Mix (13 components)

Components in this mix (0.5µL/mL or 0.05% volume/volume each) are at 10X the concentration of the final test solution specified in ASTM 1618 and ASTM 1387.

n-hexane (C6)
n-octane (C8)
n-decane (C10)
n-dodecane (C12)
n-tetradecane (C14)
n-hexadecane (C16)
n-octadecane (C18)
n-eicosane (C20)
2-ethyltoluene
3-ethyltoluene
toluene
1,2,4-trimethylbenzene
p-xylene

0.05% volume/volume each in methylene chloride, 1mL/ampul
cat. # 31613 (ea.)

Glycols Standard

ethylene glycol
propylene glycol

50,000µg/mL each in DI water, 1mL/ampul
cat. # 30471 (ea.)

Blood Alcohol Standards

We have developed eleven calibration mixtures for performing multi-point instrument calibrations so that laboratories can construct calibration curves. The data pack includes a Certificate of Analysis, raw material testing results, statistical QA results, analytical balance printout, and gravimetric weight of each analyte. Ethanol in these mixes is National Institute of Standards and Technology (NIST)-traceable.

Compound	qty.	cat.#
0.015g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36232
1mL/ampul	10-pk.	36332
5mL/ampul	ea.	36240
20mL/ampul	ea.	36248
0.02g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36233
1mL/ampul	10-pk.	36333
5mL/ampul	ea.	36241
20mL/ampul	ea.	36249
0.025g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36234
1mL/ampul	10-pk.	36334
5mL/ampul	ea.	36242
20mL/ampul	ea.	36250
0.04g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36235
1mL/ampul	10-pk.	36335
5mL/ampul	ea.	36243
20mL/ampul	ea.	36251
0.05g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36257
1mL/ampul	10-pk.	36259
5mL/ampul	ea.	36258
20mL/ampul	ea.	36260
0.08g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36262
1mL/ampul	10-pk.	36264
5mL/ampul	ea.	36263
20mL/ampul	ea.	36265
0.1g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36236
1mL/ampul	10-pk.	36336
5mL/ampul	ea.	36244
20mL/ampul	ea.	36252
0.15g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36237
1mL/ampul	10-pk.	36337
5mL/ampul	ea.	36245
20mL/ampul	ea.	36253
0.2g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36238
1mL/ampul	10-pk.	36338
5mL/ampul	ea.	36246
20mL/ampul	ea.	36254
0.3g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36239
1mL/ampul	10-pk.	36339
5mL/ampul	ea.	36247
20mL/ampul	ea.	36255
0.4g/dL forensic ethanol solution		
1mL/ampul	5-pk.	36266
1mL/ampul	10-pk.	36268
5mL/ampul	ea.	36267
20mL/ampul	ea.	36269

Blood Alcohol Mix Resolution Control Standard

(8 components)

Use our Resolution Control Standard to verify the retention time for each compound normally included in a blood alcohol test, and to verify that the compounds are resolved from and do not interfere with one another. Concentration of ethanol is NIST-traceable.

acetaldehyde	ethyl acetate
acetone	isopropanol
acetonitrile	methanol
ethanol (NIST certified value)	methyl ethyl ketone

0.100g/dL each in water, 1mL/ampul

cat. # 36256 (ea.)

Bank Dye Standard (MAAQ)

Restek offers this qualitative standard to help investigators in municipal police stations and criminal laboratories fight crime.

1-N-(methylamino)anthraquinone (MAAQ)

100µg/mL in methylene chloride, 1mL/ampul

cat. # 31823 (ea.)

No data pack available.

Column Test Mixes

Routine analysis using these products can assist in determining the need to perform column and/or system maintenance.

Grob Test Mix (Capillary GC)

*n*C10-FAME	0.42mg/mL
*n*C11-FAME	0.42
*n*C12-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.)

No data pack available.

HPLC Reversed Phase Test Mix #1

benzene	0.50
3.00mg/mL	biphenyl
uracil 0.02	0.06
naphthalene	

In methanol:water (75:25), 1mL/ampul

cat. # 35005 (ea.)

No data pack available.

HPLC Normal Phase Test Mix #1

benzene	benzyl alcohol
1.00mg/mL	3.00
benzaldehyde	4-methoxybenzyl alcohol
0.04	2.00

In hexane, 1mL/ampul

cat. # 35004 (ea.)

No data pack available.

did you **know**?

Our Rtx®-BAC1 and Rtx®-BAC2 columns can resolve a blood alcohol sample in less than 3 minutes. We continually improve analysis methods and develop innovative products for clinical/forensic applications. If you have any questions about methods or products, please contact our Technical Service Team at: **support@restek.com** or contact your Restek representative.

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: _____

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.

Lit. Cat.# 59989A-INT

Technical Guide

Analyzing Cleaning and Personal Care Products by Gas and Liquid Chromatography

Inside:

RESTEK

www.restekcorp.com

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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 heath. 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.

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 cleaners, 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 (carboxylate 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.

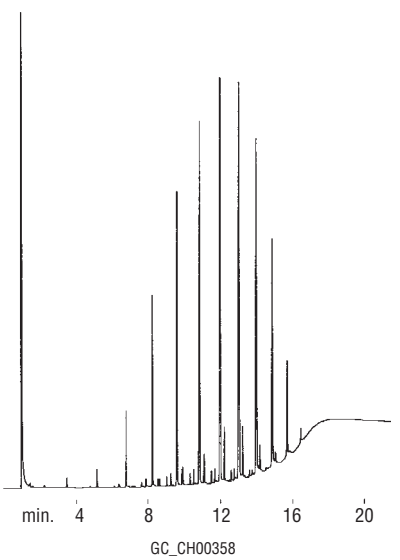
Fatty acids that make up a soap can be analyzed either in the free fatty acid form or after derivatization to the methyl esters (FAMEs). Figure 2 shows an analysis of free fatty acids by GC, using a Stabilwax®-DA capillary column. The acid-deactivated phase in the Stabilwax® DA column gives excellent 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

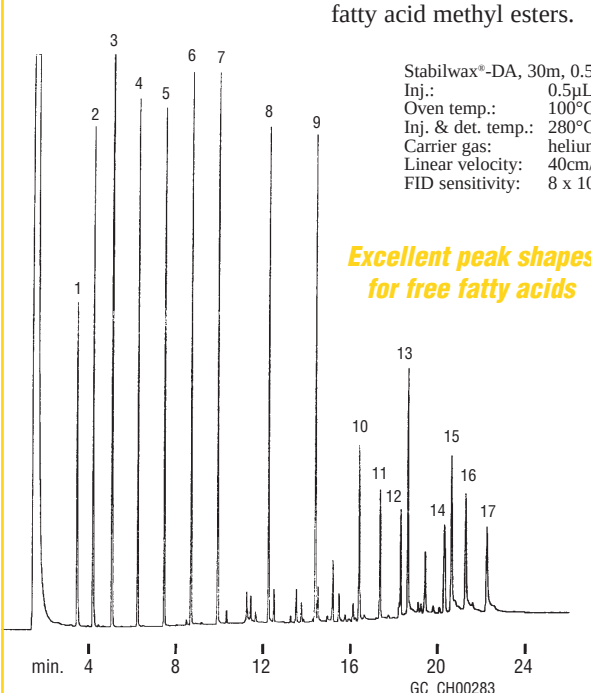
Triton® X-100 surfactant separated by number of ethylene oxide units on an MXT®-1 column.



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.



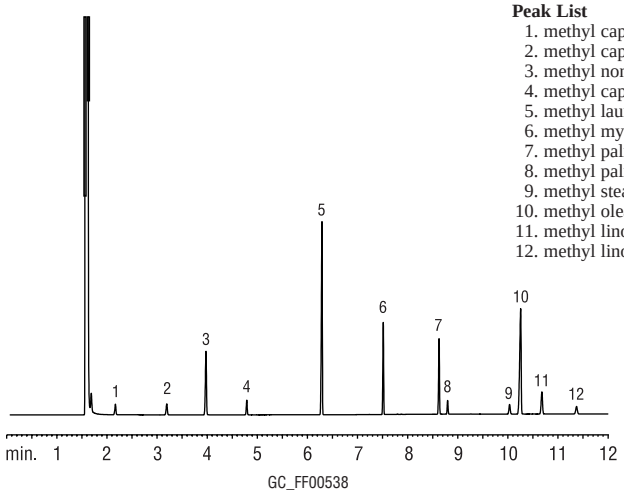
Stabilwax®-DA, 30m, 0.53mm ID, 0.25µm (cat.# 11025)
Inj.: 0.5µL direct injection of a 5mg/mL standard
Oven temp.: 100°C (hold 2 min.) to 250°C @ 8°C/min.
Inj. & det. temp.: 280°C
Carrier gas: helium
Linear velocity: 40cm/sec. (flow rate: 5.2cc/min.)
FID sensitivity: 8 x 10^{-11} AFS

Excellent peak shapes for free fatty acids

1. C2:0 - acetic acid
2. C3:0 - propionic acid
3. C4:0 - butyric acid
4. C5:0 - valeric acid
5. C6:0 - caproic acid
6. C7:0 - heptanoic acid
7. C8:0 - caprylic acid
8. C10:0 - capric acid
9. C12:0 - lauric acid
10. C14:0 - myristic acid
11. C15:0 - pentadecanoic acid
12. C16:0 - palmitic acid
13. C16:1 - palmitoleic acid
14. C18:0 - stearic acid
15. C18:1 - oleic acid
16. C18:2 - linoleic acid
17. C18:3 - linolenic acid

Figure 3

FAMEs analysis on an Rtx®-Wax column.

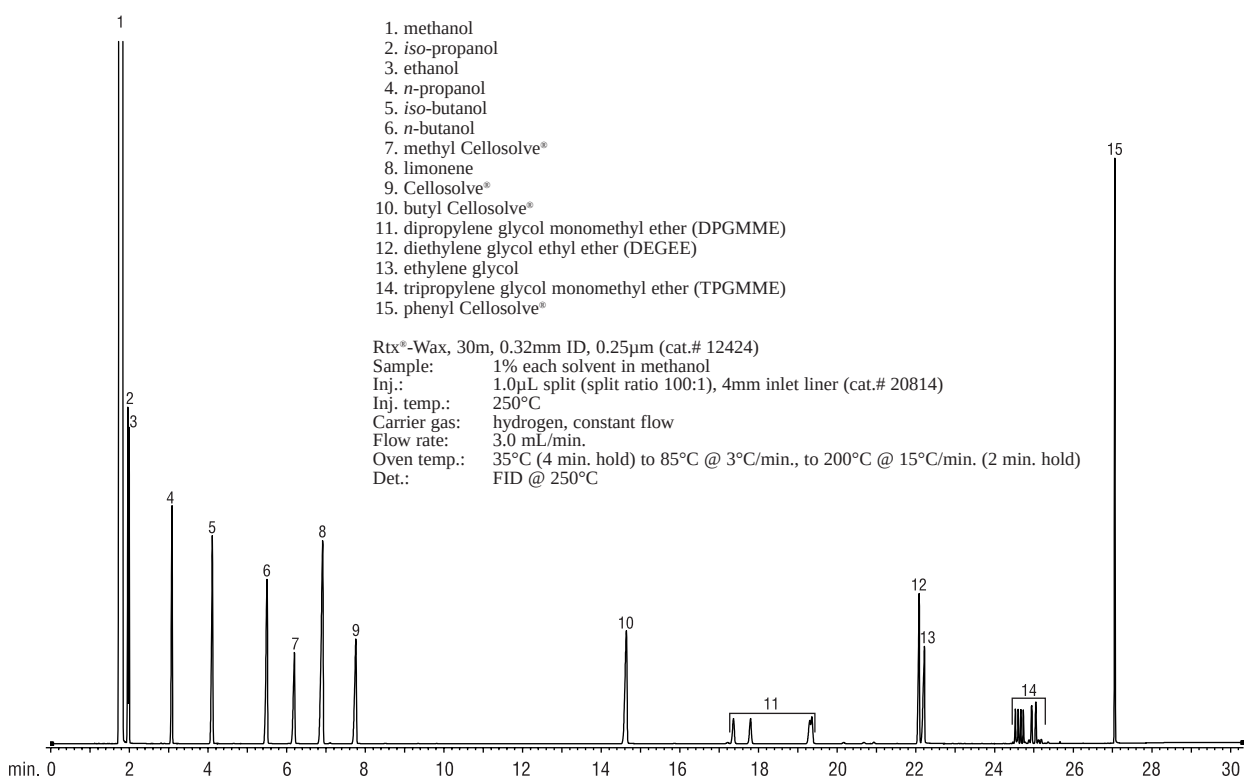


Peak List	Conc. (mg/mL)
1. methyl caproate (C6:0)	0.4
2. methyl caprylate (C8:0)	0.4
3. methyl nonanoate (C9:0)	2.0
4. methyl caprate (C10:0)	0.4
5. methyl laurate (C12:0)	5.0
6. methyl myristate (C14:0)	2.0
7. methyl palmitate (C16:0)	2.0
8. methyl palmitoleate (C16:1)	0.4
9. methyl stearate (C18:0)	0.4
10. methyl oleate (C18:1)	5.0
11. methyl linoleate (C18:2)	1.0
12. methyl linolenate (C18:3)	0.4

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.)

Figure 4

Alcohols, glycols, and other cleaning solvents can be quantified, using an Rtx®-Wax colum.



1. methanol
2. *iso*-propanol
3. ethanol
4. *n*-propanol
5. *iso*-butanol
6. *n*-butanol
7. methyl Cellosolve®
8. limonene
9. Cellosolve®
10. butyl Cellosolve®
11. dipropylene glycol monomethyl ether (DPGMME)
12. diethylene glycol ethyl ether (DEGEE)
13. ethylene glycol
14. tripropylene glycol monomethyl ether (TPGMME)
15. phenyl Cellosolve®

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.

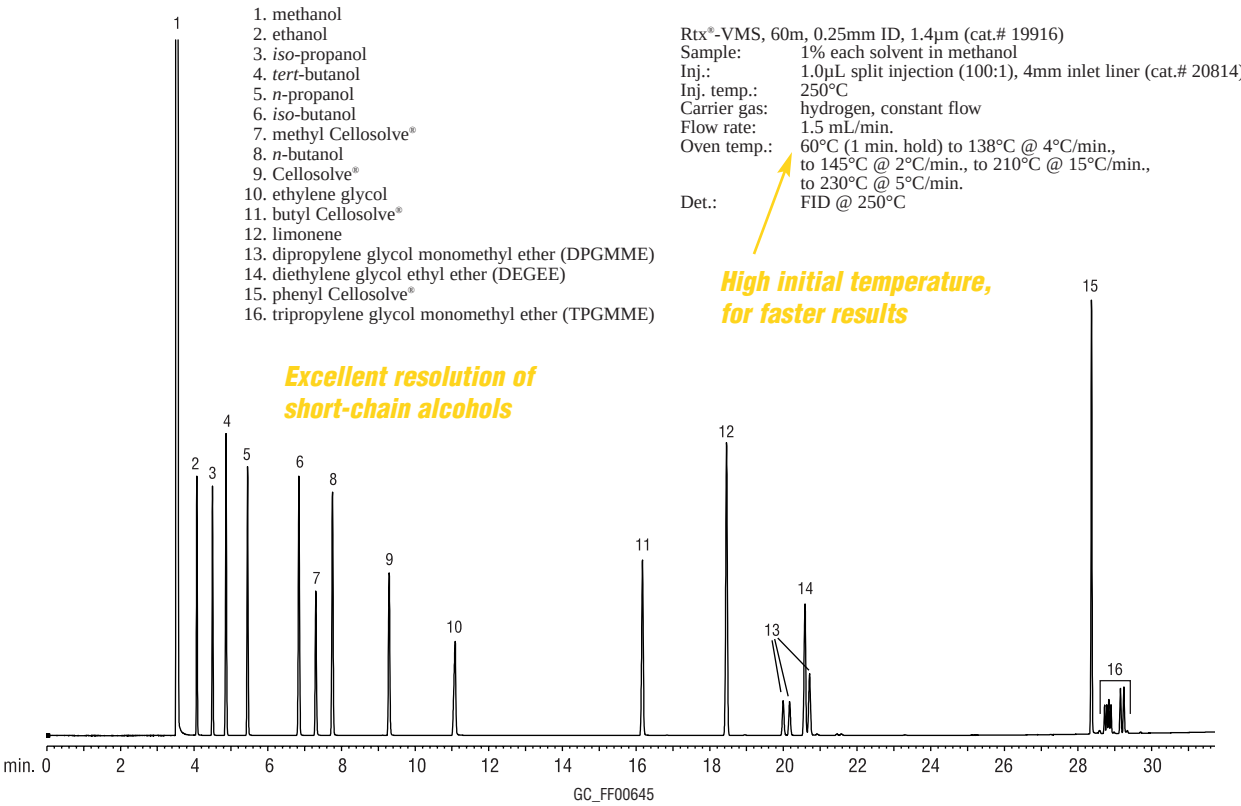
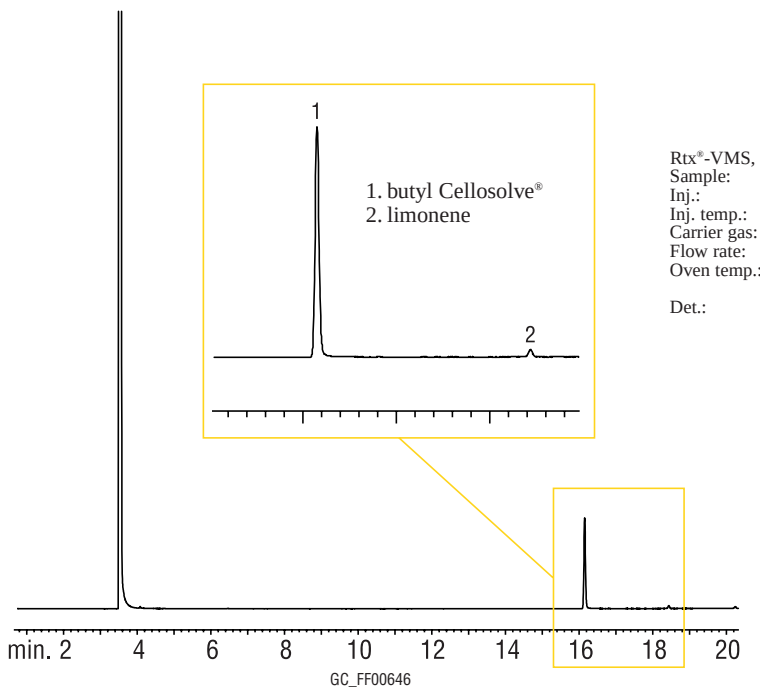


Figure 6

Quantify volatile ingredients in an all-purpose cleaner, using an Rtx®-VMS column.



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

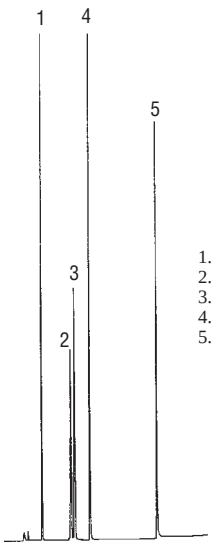
glycols are popular choices. Cleaning solvents can be resolved using an Rtx®-Wax column (Figure 4) or an Rtx®-VMS column (Figure 5). The latter column gives excellent selectivity and peak shape for a wide range of cleaning solvents. Analysis of an all-purpose cleaner is shown in Figure 6, and a glass 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 8

Glycols and alcohols on an ultra-low-bleed column.



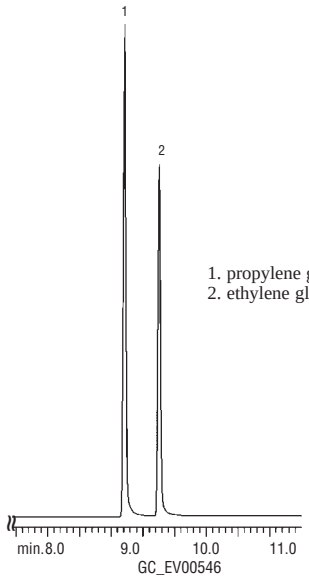
1. *n*-butyl alcohol
2. ethylene glycol
3. propylene glycol
4. butyl Cellosolve®
5. butyl carbitol

XTI®-5, 30m, 0.53mm ID, 1.0µm (cat.# 12255)

Inj.:	1.0µL direct injection of glycols and alcohols, 100ppm each
Oven temp.:	40°C to 185°C @ 15°C/min. (hold 5 min.)
Inj. / det. temp.:	150°C / 200°C
Carrier gas:	helium
Linear velocity:	40cm/sec. (flow rate: 5cc/min.)
FID sensitivity:	8 x 10^{-11} AFS

Figure 9

Glycols on a Stabilwax® column.



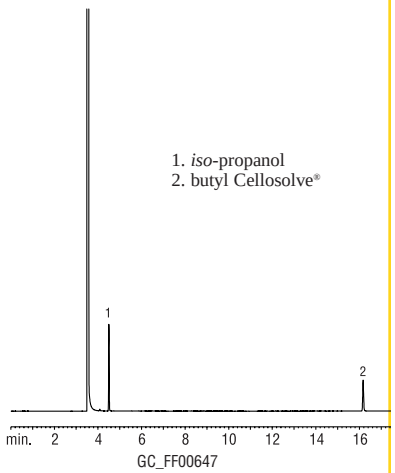
1. propylene glycol
2. ethylene glycol

Stabilwax®, 30m, 0.53mm ID, 1.0µm (cat.# 10655)

Inj.:	1.0µL direct injectionof 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 7

Glass cleaner on an Rtx®-VMS column.



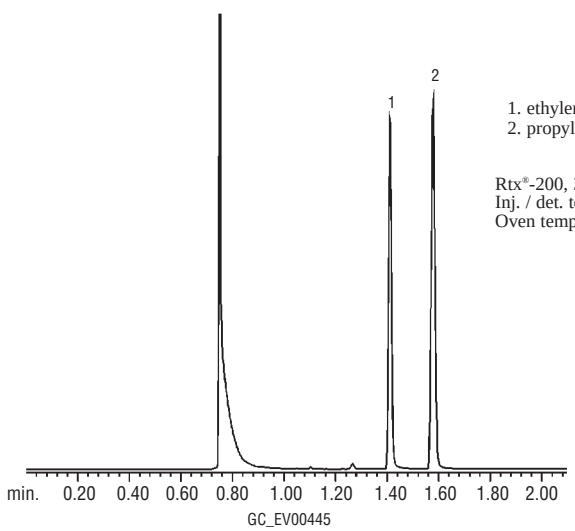
1. *iso*-propanol
2. butyl Cellosolve®

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 10

Glycols on a uniquely selective trifluoropropyl phase column.



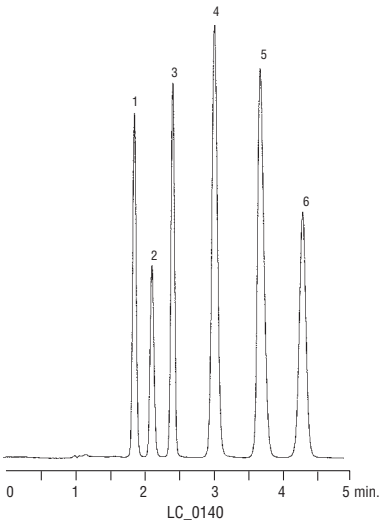
1. ethylene glycol
2. propylene glycol

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

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

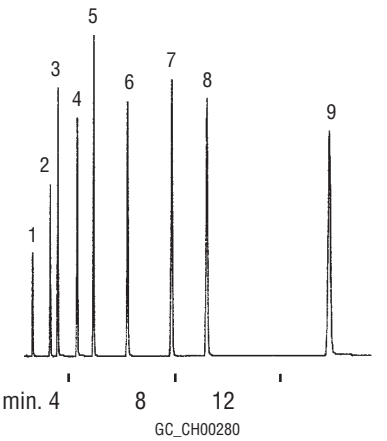
Sample:
Solvent: HPLC-grade water
Inj.: 10µL

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

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/µLeach 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

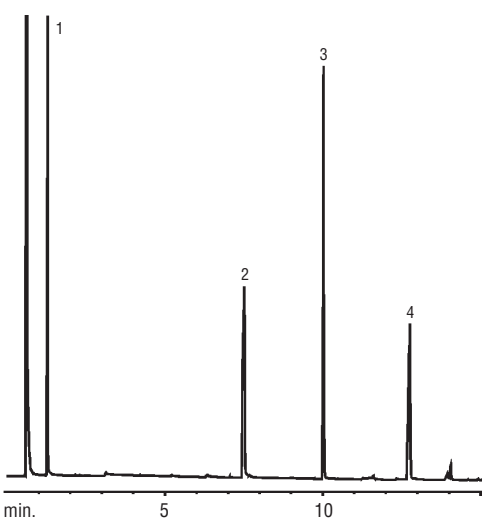
Products with higher pH are useful for dissolving fatty or oily soils. Alkalis, or bases, are used to neutralize acidic ingredients, or to raise the pH. Suitable alkalis include ethanolamines, ammonium hydroxide, and sodium silicate. The more basic compounds, such as ethanolamines, can be analyzed by 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

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.

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*-xylanol/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.

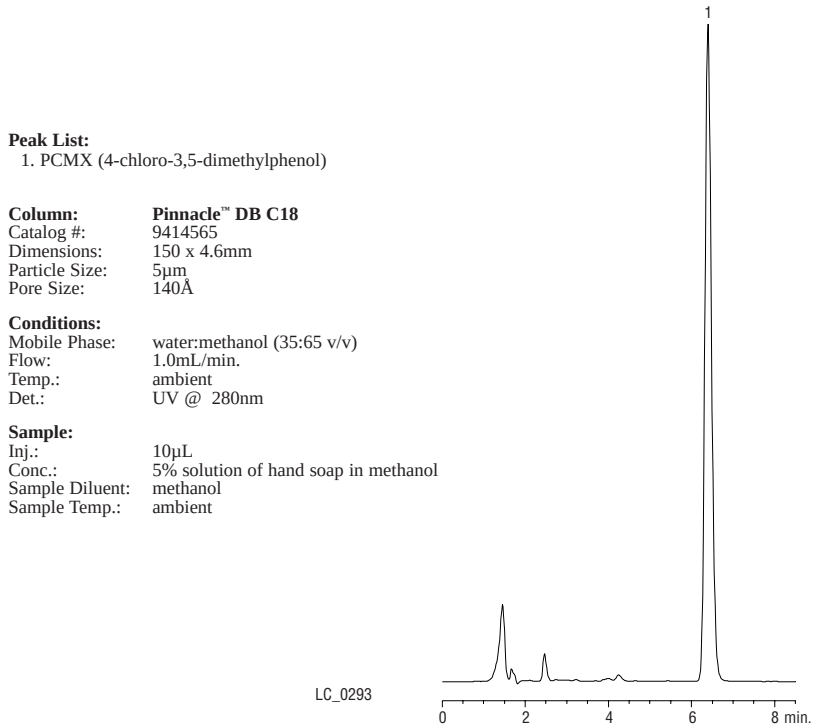


Figure 15

Resolution and symmetric peaks for sorbic and benzoic acids on an Ultra Phenyl HPLC column.

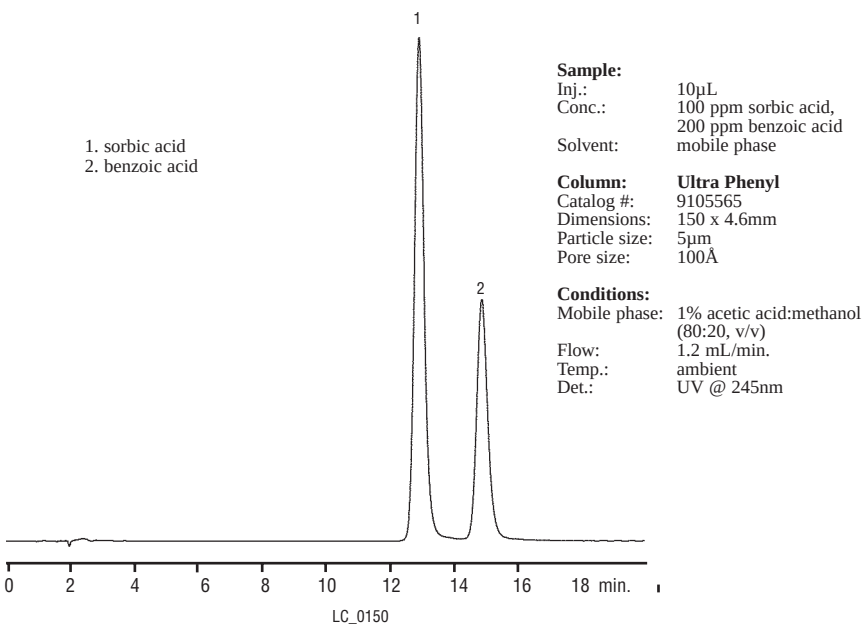
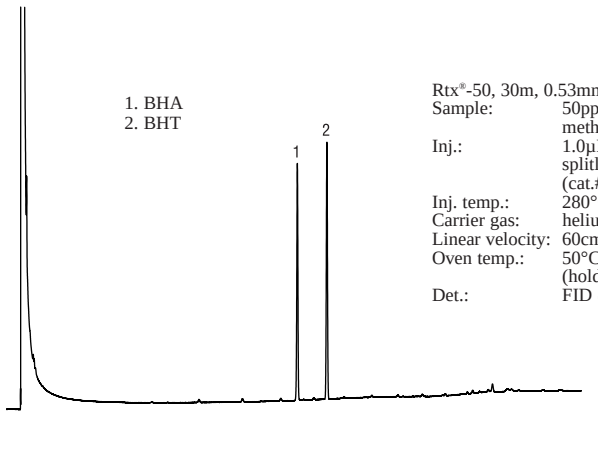


Figure 16

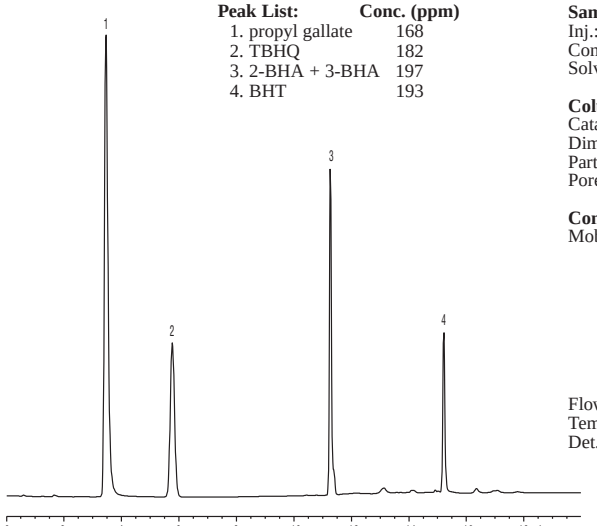
Preservatives BHA and BHT on an Rtx®-50 intermediate polarity column.



Rtx®-50, 30m, 0.53mm ID, 0.50µm (cat.# 10540)	
Sample:	50ppm each analyte in methanol
Inj.:	1.0µL direct, 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

Figure 17

Phenolic preservatives, including BHA and BHT, on a Pinnacle II™ C18 HPLC column.



Peak List:	Conc. (ppm)
1. propyl gallate	168
2. TBHQ	182
3. 2-BHA + 3-BHA	197
4. BHT	193

Sample:	
Inj.:	10µL
Conc.:	see peak list
Solvent:	methanol
Column:	**Pinnacle II™ C18**
Catalog #:	9214565
Dimensions:	150 x 4.6mm
Particle size:	5µm
Pore size:	110Å
Conditions:	
Mobile phase:	A = 1% acetic acid B = methanol

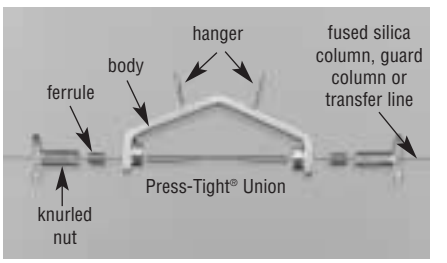
Time (min.)	B (%)
0	50
4	50
10	90
25	90
26	50

Flow:	1.0mL/min
Temp.:	30°C
Det.:	UV @ 280nm

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?

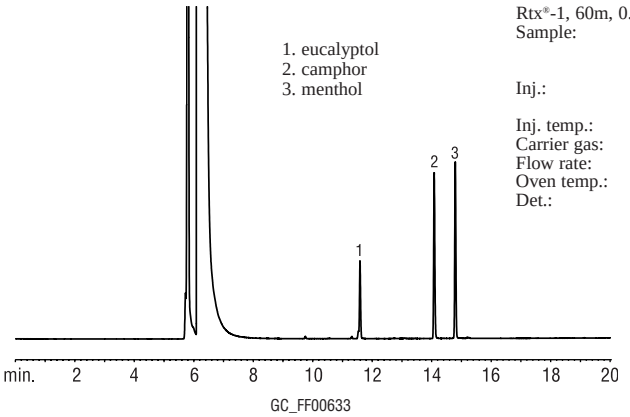
Contact Restek's Technical Service Team! We have answers to your toughest analytical questions. Call 800-356-1688 or 814-353-1300, ext. 4, email us at support@restekcorp.com or contact your local Restek representative.

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

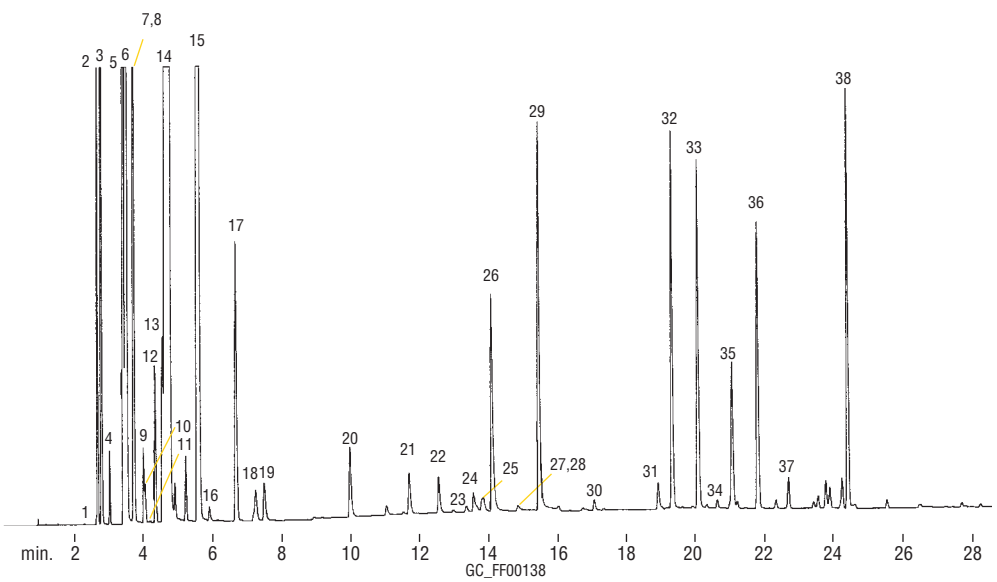
Personal care product fragrance compounds on an Rtx®-1 column.



Rtx®-1, 60m, 0.25mm ID, 0.25µm (cat.# 10126)
Sample: 130ppm eucalyptus oil
250ppm camphor
260ppm menthol in methanol
Inj.: 1.0µL split injection (20:1), 4mm inlet liner (cat.# 20814)
Inj. temp.: 275°C
Carrier gas: helium, constant flow
Flow rate: 0.6 mL/min.
Oven temp.: 80°C to 180°C @5°C/min.
Det.: FID @300°C

Figure 19

A complex fragrance, lemon oil, resolved on an Rtx®-5 column.

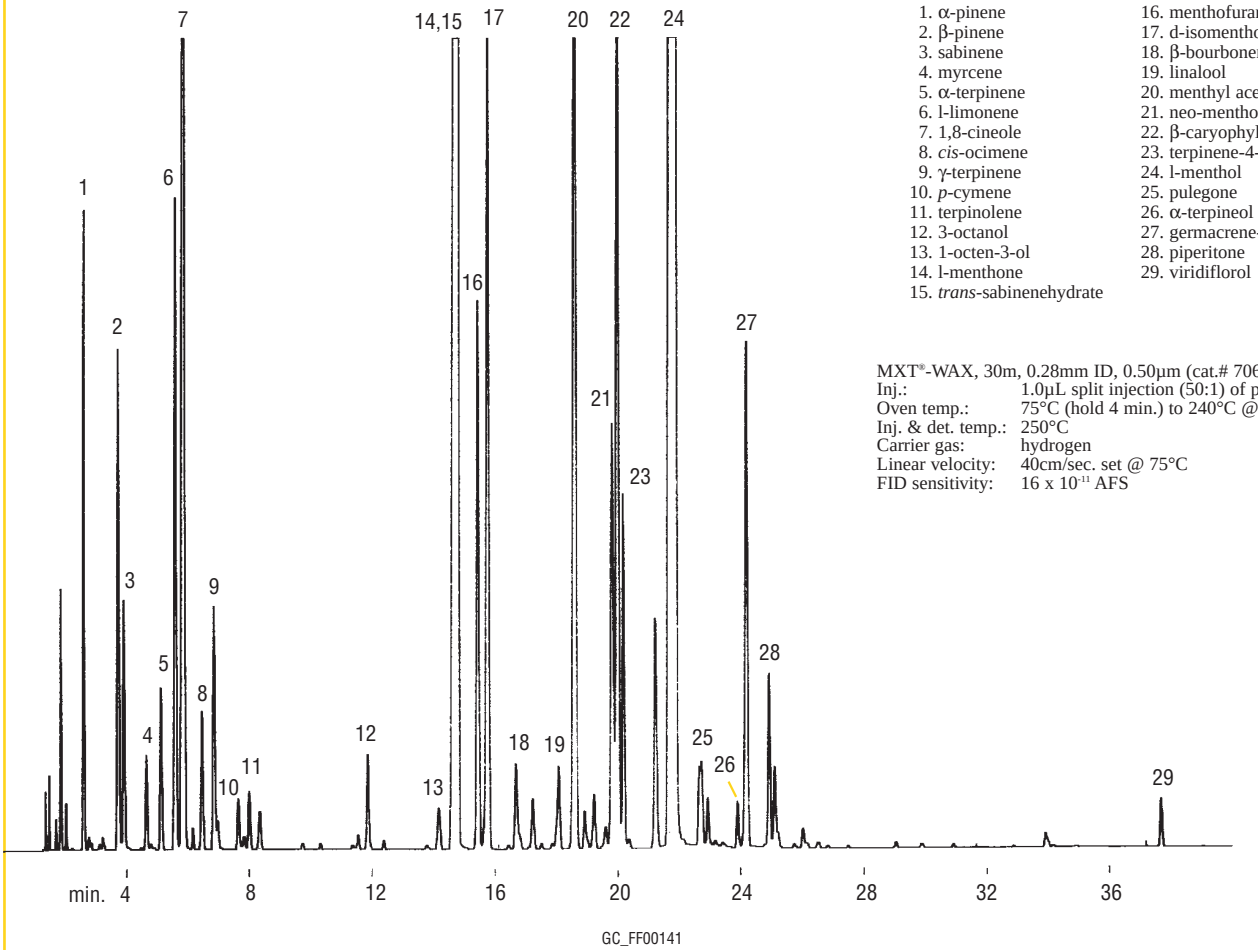


Rtx®-5, 30m, 0.32mm ID, 0.25µm (cat.# 10224)
Inj.: wet needle split injection (100:1) of neat lemon oil
Oven temp.: 75°C (hold 8 min.) to 250°C @ 4°C/min.
Inj. & det. temp.: 250°C
Carrier gas: hydrogen
Linear velocity: 40cm/sec. (flow rate: 3.2cc/min.)
FID sensitivity: 2 x 10^{-11} AFS

1. heptanol
2. α-thujene
3. α-pinene
4. camphene
5. sabinene
6. β-pinene
7. 6-methyl-5-hepten-2-one
8. myrcene
9. octanal
10. α-phellandrene
11. 3-carene
12. α-terpinene
13. *p*-cymene
14. limonene
15. γ-terpinene
16. octanol
17. terpinolene
18. linalool
19. nonanal
20. citronellal
21. terpinene-4-ol
22. α-terpineol
23. decanol
24. octyl acetate
25. nerol
26. neral
27. carvone
28. geraniol
29. geranial
30. nonyl acetate
31. citronellyl acetate
32. neryl acetate
33. geranyl acetate
34. dodecanal
35. β-caryophyllene
36. *trans*-α-bergamotene
37. α-humulene
38. β-bisabolene

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

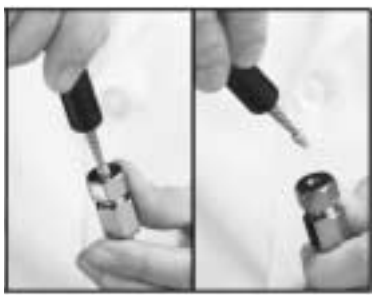
1. Branna, Tom *The I&I Market* in *Happi*, Nov. 2000.
2. The Soap and Detergent Association. www.sdahq.org
3. US Environmental Protection Agency. www.epa.gov/pesticides/citizens/antimic.htm

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

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

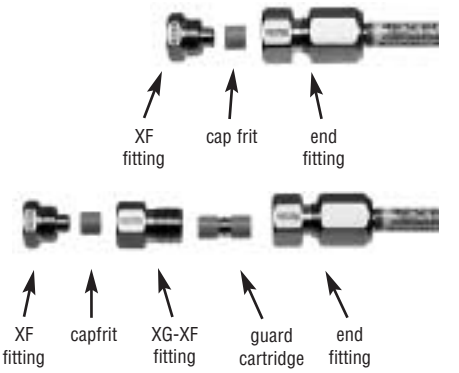
Trident™ HPLC In-Line Guard Cartridge Holders

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-styl*

Guard Column Systems



Trident™ Direct provides three levels of protection

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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

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% dimethylpolysiloxane)
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

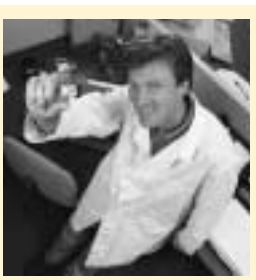
citric acid	2000µg/ml
fumaric acid	10
malic acid	2000
quinic acid	2000
tartaric acid	2000

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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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

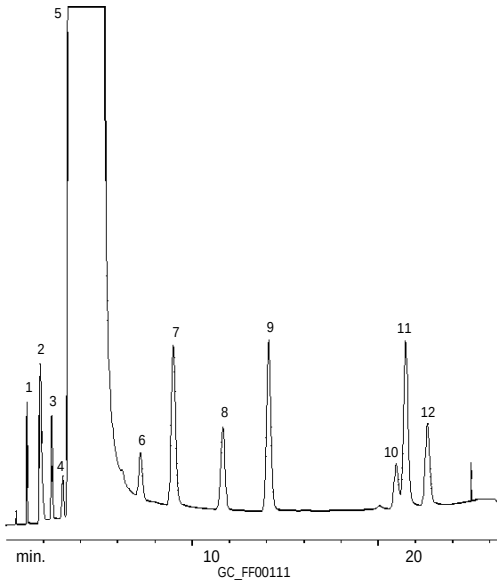
Alcoholic beverages contain a wide range of volatile compounds, including alcohols and short-chain aldehydes. Gas chromatography can be used to analyze these compounds without preliminary extractions. For example, AOAC International has published methods for the analysis of fusel oils, methanol, ethanol, 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.



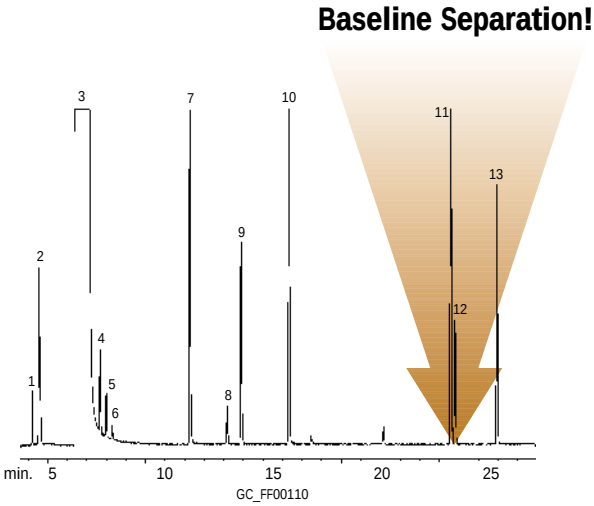
1. acetaldehyde
2. methanol
3. acetone
4. ethyl formate
5. ethanol
6. ethyl acetate
7. *n*-propanol
8. *sec*-butanol
9. isobutanol
10. active amyl alcohol
11. isoamyl alcohol
12. *n*-amyl alcohol

5% Carbowax® 20M 80/120 CarboBlack™ B (cat.# 80105)
2m, 1/8" OD x 2mm ID SilcoSmooth™ tubing
0.5µL on-column injection of fusel oils in rum,
Concentration: 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

Figure 2

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



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

60m, 0.25mm ID, 1.4µm Rtx®-1301 (cat.# 16016)
1.0µL split injection using a Cyclosplitter® sleeve (cat.# 20706).
Concentration: 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

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.

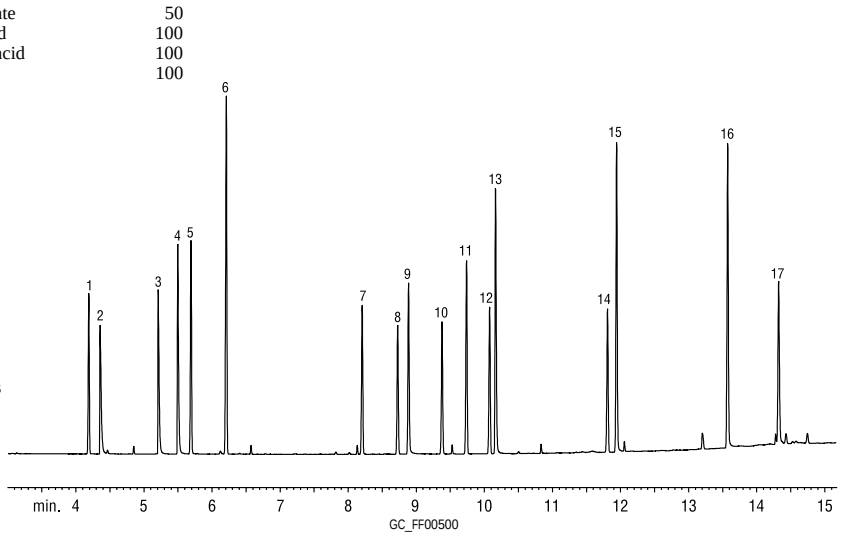
To test the applicability of this column in these dimensions, the critical pair of caproic acid and ethyl laurate was studied. These components can be very difficult to resolve on standard Carbowax®-type columns, especially if peak tailing or broadening occurs, or if one component is present at a significantly 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
2. acetic acid	100
3. propionic acid	100
4. isobutyric acid	100
5. decanol 3	50
6. ethyl decanoate	50
7. ethyl laurate	50
8. *cis*-lactone	100
9. 2-phenylethanol	50
10. *trans*-lactone	100
11. methyl myristate	50
12. ethyl myristate	50
13. octanoic acid	100
14. ethyl palmitate	50
15. decanoic acid	100
16. dodecanoic acid	100
17. vanillin	100

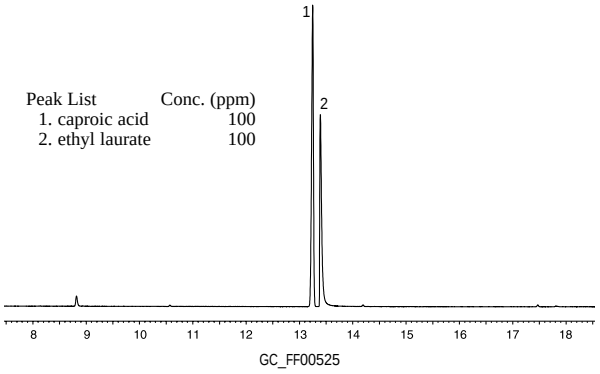
Stabilwax®-DA 30m, 0.18mm ID, 0.18µm (cat.# 550752)
Inj.: 1µL splitless (hold 0.5 min.) at conc. shown in peak list, in ethyl acetate, 4mm ID splitless liner w/wool (cat.# 20814-202.1)
Inj. temp.: 240°C
Carrier gas: hydrogen
Make-up gas: nitrogen
Linear velocity: 28psi @ 240°C
Oven temp.: 70°C to 240°C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



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.

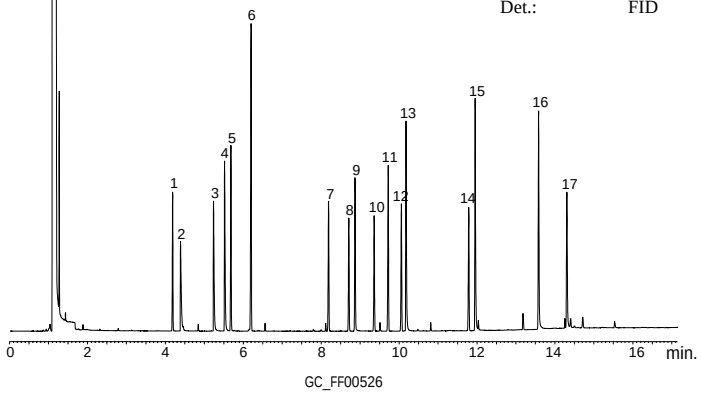


Stabilwax®-DA 30m, 0.18mm ID, 0.18µm (cat.# 550752)
Inj.: 1µL splitless (hold 0.5 min.) at conc. shown in peak list, in ethyl acetate, 4mm ID splitless liner w/wool (cat.# 20814-202.1)
Inj. temp.: 240°C
Carrier gas: hydrogen
Make-up gas: nitrogen
Linear velocity: 28psi @ 240°C
Oven temp.: 80°C to 230°C at 5°C/min.
Det.: FID

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.

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

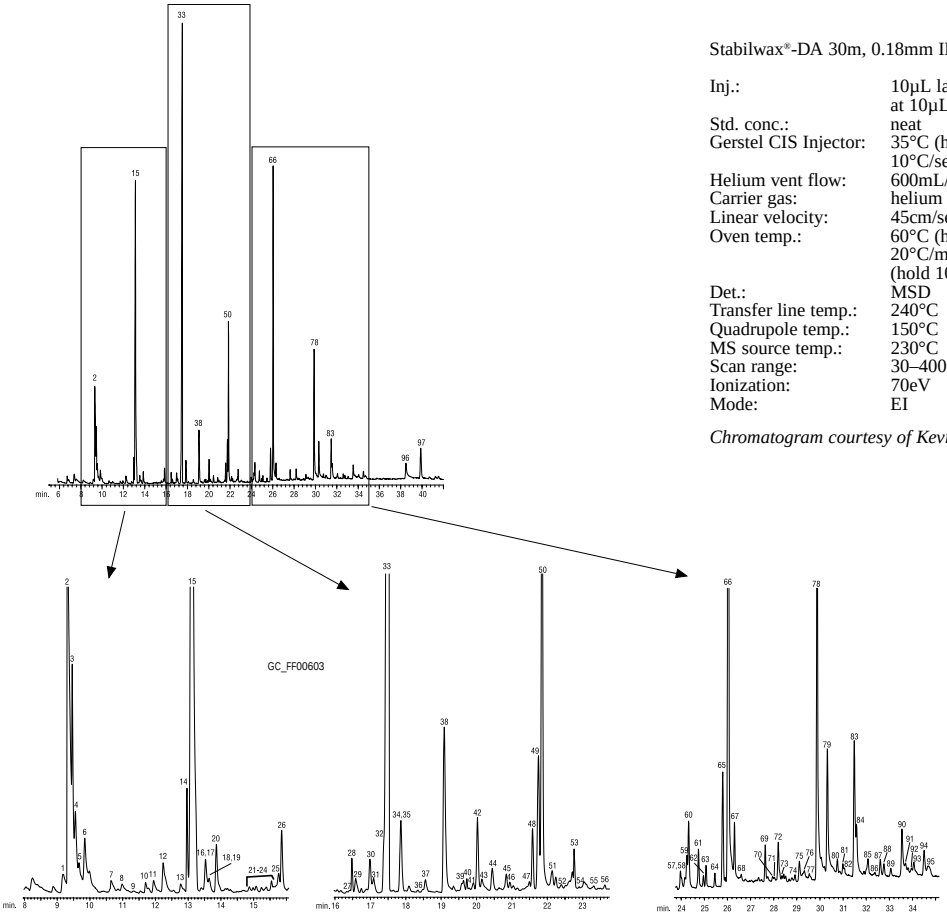


Peak List	Conc. (ppm)
1. ethyl octanoate	100
2. acetic acid	100
3. propionic acid	100
4. isobutyric acid	100
5. decanol 3	50
6. ethyl decanoate	50
7. ethyl laurate	50
8. *cis*-lactone	100
9. 2-phenylethanol	50
10. *trans*-lactone	100
11. methyl myristate	50
12. ethyl myristate	50
13. octanoic acid	100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

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.



Stabilwax®-DA 30m, 0.18mm ID, 0.18µm (cat.# 550752)

Inj.:	10µL large volume injection (splitless), at 10µL/min.
Std. conc.:	neat
Gerstel CIS Injector:	35°C (hold 2 min.), to 300°C @ 10°C/sec. (hold 5 min.)
Helium vent flow:	600mL/min with 1.8 min. vent end time
Carrier gas:	helium
Linear velocity:	45cm/sec.
Oven temp.:	60°C (hold 2 min.) to 100°C @ 20°C/min., to 240°C @ 5°C/min. (hold 10 min.)
Det.:	MSD
Transfer line temp.:	240°C
Quadrupole temp.:	150°C
MS source temp.:	230°C
Scan range:	30–400amu
Ionization:	70eV
Mode:	EI

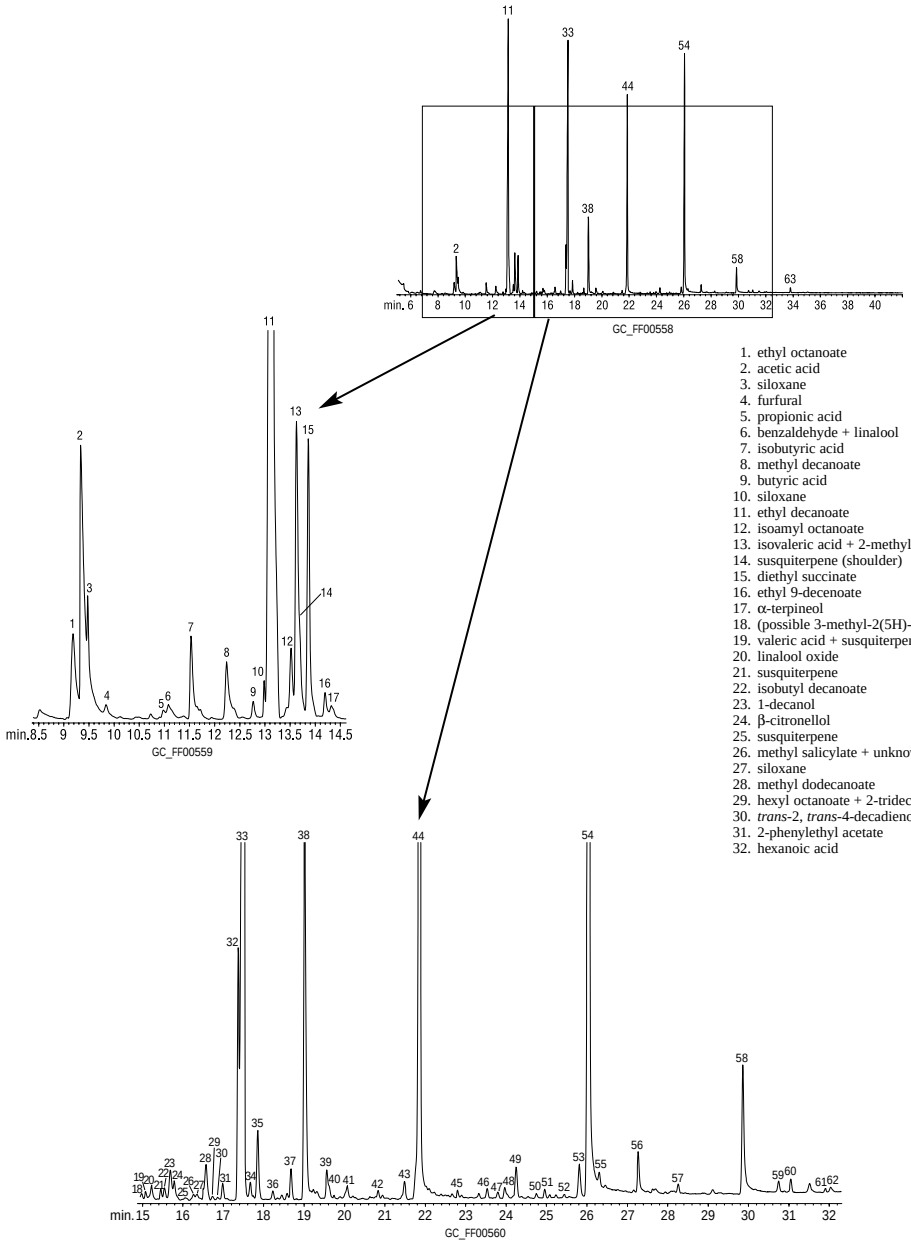
Chromatogram courtesy of Kevin MacNamara, Ph.D., Irish Distilleries, Ltd.

1. ethyl octanoate
2. acetic acid
3. siloxane
4. 1-hydroxy-2,3-butadione
5. 1-hydroxy-2-propanone acetate
6. furfural
7. formic acid
8. propionic acid
9. isobutyric acid
10. dimethyl sulfoxide
11. 5-methyl furfural
12. methyl decanoate + unknown
13. butyric acid
14. siloxane
15. ethyl decanoate
16. furfuryl alcohol
17. isoamyl octanoate
18. isovaleric acid
19. 2-methylbutyric acid
20. diethyl succinate
21. 3-methyl-2(5H)-furanone (possible)
22. valeric acid
23. ethyl undecanoate
24. isobutyl decanoate
25. 2(5H)-furanone
26. unknown
27. di(ethyleneglycol) butyl ether
28. siloxane
29. methyl dodecanoate
30. 2-phenylethyl acetate
31. methylcyclopentenolone
32. hexanoic acid
33. ethyl dodecanoate
34. isoamyl decanoate
35. guaiacol
36. dodecyl acetate (possible)
37. whiskey lactone (1)
38. 2-phenylethanol
39. heptanoic acid
40. siloxane
41. dimethoxybenzene or 4-methylguaiacol
42. whiskey lactone (2)
43. dodecanol
44. unknown
45. phenol
46. methyl tetradecanoate
47. nerolidol
48. diethyl malate
49. ethyl tetradecanoate
50. octanoic acid
51. unknown
52. *p*-cresol
53. siloxane
54. diethyl octanedioate
55. monomethyl succinate (possible)
56. 3,5-dimethyl-2,4(5H) furandione
57. nonanoic acid
58. diethyl 2-hydroxyglutarate
59. unknown
60. tetradecanol
61. 4-vinylguaiacol
62. diethyl nonanedioate
63. methyl hexadecanoate
64. ethyl γ-lactone 2-hydroxyglutarate
65. ethyl hexadecanoate
66. decanoic acid
67. ethyl 9-hexadecenoate
68. triacosan
69. unknown
70. phthalide
71. diethylphthalate
72. hexadecanol
73. 4-hydroxycinnamic acid (decomp.)
74. methyl stearate
75. benzoic acid
76. methyl 8-octadecenoate
77. ethyl stearate
78. dodecanoic acid
79. hydroxymethylfurfural
80. ethyl linoleate
81. 4-allyl-2,6-dimethoxyphenol
82. diisobutyl phthalate
83. vanillin
84. sinapic acid (decomp.)
85. 2-phenylethyl decanoate + 2 unknowns
86. 4-propenyl-2,6-dimethoxyphenol
87. ethyl vanillate
88. acetovanillone
89. vanillin methyl ketone
90. tetradecanoic acid
91. ethyl homovanillate
92. propiovanillone
93. fatty acid ester
94. (similar to 4-allyl-2,6-dimethoxyphenol)
95. unknown
96. hexadecanoic acid
97. syringealdehyde

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.



1. ethyl octanoate
2. acetic acid
3. siloxane
4. furfural
5. propionic acid
6. benzaldehyde + linalool
7. isobutyric acid
8. methyl decanoate
9. butyric acid
10. siloxane
11. ethyl decanoate
12. isoamyl octanoate
13. isovaleric acid + 2-methylbutyric acid
14. susquiterpene (shoulder)
15. diethyl succinate
16. ethyl 9-decenoate
17. α-terpineol
18. (possible 3-methyl-2(5H)- furanone)
19. valeric acid + susquiterpene
20. linalool oxide
21. susquiterpene
22. isobutyl decanoate
23. 1-decanol
24. β-citronellol
25. susquiterpene
26. methyl salicylate + unknown
27. siloxane
28. methyl dodecanoate
29. hexyl octanoate + 2-tridecanone
30. *trans*-2, *trans*-4-decadienol
31. 2-phenylethyl acetate
32. hexanoic acid
33. ethyl dodecanoate
34. unknown
35. isoamyl decanoate
36. benzyl alcohol
37. unknown
38. 2-phenylethanol
39. 2-ethylhexanoic acid
40. heptanoic acid
41. dodecanol
42. phenol
43. γ-nonalactone
44. octanoic acid
45. siloxane
46. ethyl cinnamate
47. γ-decalactone + unknown
48. nonanoic acid
49. phenylethyl hexanoate
50. diethyl nonanedioate
51. methyl hexadecanoate
52. dimethyl naphthalene
53. ethyl hexadecanoate
54. decanoic acid
55. ethyl 9-hexadecenoate
56. geranic acid
57. 2-phenylethyl octanoate
58. dodecanoic acid
59. ethyl linoleate
60. diisobutyl phthalate
61. ethyl linolenate
62. phenylethyl decanoate
63. dibutyl phthalate

Stabilwax®-DA 30m, 0.18mm ID, 0.18µm (cat.# 550752)

Inj.:	10µL large volume injection (split less), at 10µL/min.
Std. conc.:	neat
Gerstel CIS Injector:	35°C (hold 2 min.), to 300°C @ 10°C/sec. (hold 5 min.)
Helium vent flow:	600mL/min with 1.8 min. vent end time
Carrier gas:	helium
Linear velocity:	45cm/sec.
Oven temp.:	60°C (hold 2 min.) to 100°C @ 20°C/min., to 240°C @ 5°C/min. (hold 10 min.)
Det.:	MSD
Transfer line temp.:	240°C
Quadrupole temp.:	150°C
MS source temp.:	230°C
Scan range:	30–400amu
Ionization:	70eV
Mode:	EI

Chromatogram courtesy of Kevin MacNamara, Ph.D., Irish Distilleries, Ltd.

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.

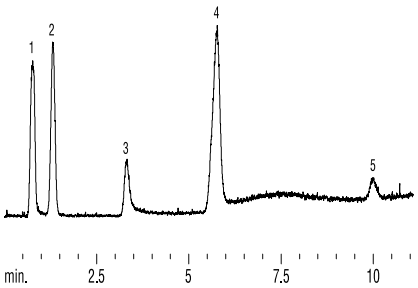
hydrogen sulfide	ethyl mercaptan	dimethyl disulfide
isopropyl mercaptan	t-butyl mercaptan	isobutyl mercaptan
carbonyl sulfide	sulfur dioxide	carbon disulfide
methyl ethyl sulfide	sec-butyl mercaptan	n-butyl mercaptan
methyl mercaptan	dimethyl sulfide	t-amyl mercaptan
n-propyl mercaptan	diethyl sulfide	

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 CO_2 (i.e., 20ppb) easily can be detected using an Rt-XLSulfur® micropacked column and a Sulfinert® treated sample pathway.



GC_FF00491

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

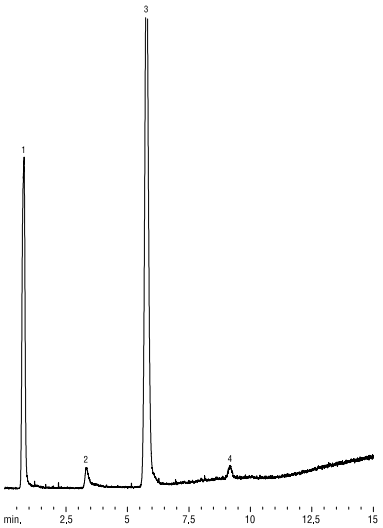
1m, 0.75mm ID Sulfinert™ tubing
Rt-XLSulfur™ 100/120 mesh (cat.# 19806)
Conc.: sulfur standard @ 20ppb each in CO_2
Inj.: 1cc sample loop, 6-port Valco® valve
Carrier gas: helium
Flow rate: 10mL/ min. @ ambient temp.
Oven temp.: 60°C to 260°C @ 15°C/min. (hold 5 min.)
Det. sensitivity: SCD, attn. x 1
Det. temp.: 800°C

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



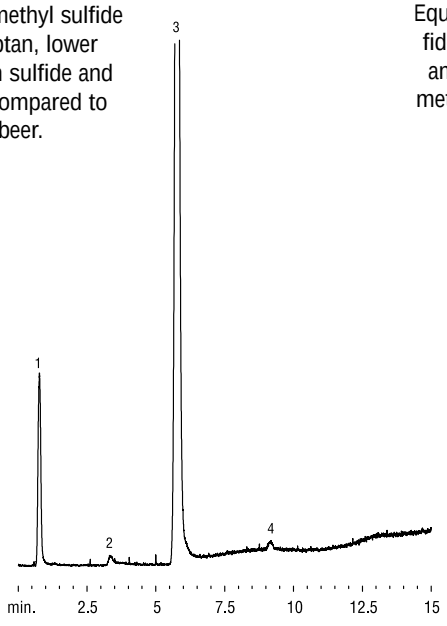
GC_FF00494

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)

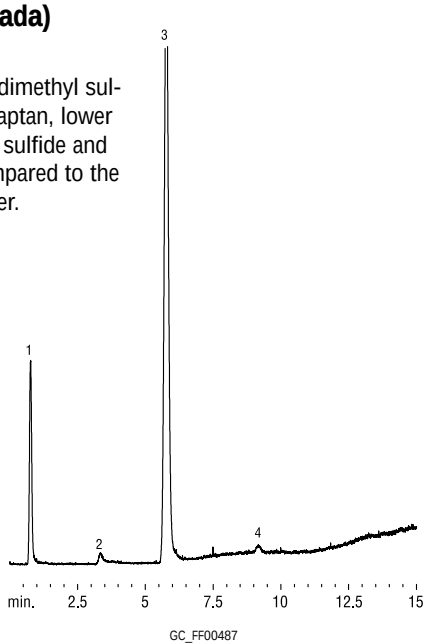
Higher amounts of dimethyl sulfide and/or ethyl mercaptan, lower amounts of hydrogen sulfide and methyl mercaptan, compared to the domestic beer.



GC_FF00483

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.



GC_FF00487

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 accomodate 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

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
	1.40	-20 to 240°C			16016
0.32mm	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
	1.00	-20 to 260°C	16051	16054	16057
	1.50	-20 to 250°C	16066	16069	16072
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

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

		Stainless Steel Tubing				SilcoSmooth™ Tubing			
On CarboBlack™ B	Mesh	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 $1x10^{-4}$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 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

www.restekcorp.com

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

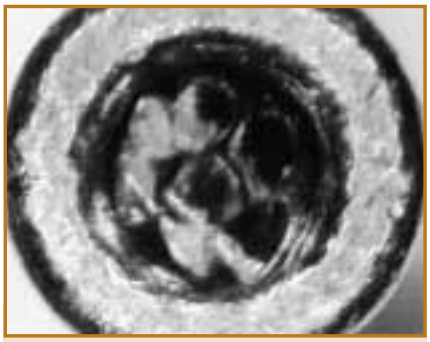
Purchase installation kit separately.

OD	ID (mm)	1-Meter	2-Meter
$^1/_{16}$"	1.0mm	19804	19805
0.95mm	0.75mm	19806	19807

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	—

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.

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

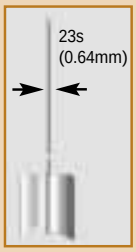
Description	100-pk.	1000-pk.
Silver Seal w/ PTFE/Gray Butyl Rubber	21761	21762
Silver Seal w/ PTFE/Silicone	21763	21764
Pressure Release Silver Seal w/ PTFE/Gray Butyl Rubber Septum <125°C	21765	21766
Pressure Release Silver Seal w/ PTFE/Silicone Septum >125°C	21767	21768

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.

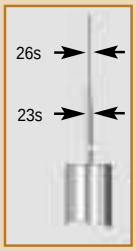
Needle Gauge for Agilent 7673 Syringes



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.

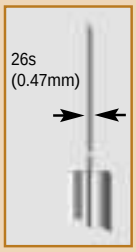
SuperfleX™ Flexible Plunger Syringe
Agilent 7673 Syringe
Gas-Tight Luer-lock Syringe



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 Ag



26s—Single Gauge Need

- On-column injection port
- Split/splitless injection po

Hamilton 10µL, Autosampler C
for Agilent 7673 Autosampler

Micropacked Columns

Drawings reproduced with permission from Hamilton.

Hamilton Syringes

Volume	Needle Term.	Needle Gauge	Needle Length	Point Style	Hamilton Model	Hamilton cat.#	Restek 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

** Autosampler cemented needle.*

*** Designated by Agilent as #80397.*

SGE Syringes

Volume	Needle Term.	Needle Gauge	Needle Length	Point Style	SGE Model	SGE cat.#	Restek 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-.063/0.47	002822	6-pk.	21215

** Fixed needle.*

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	

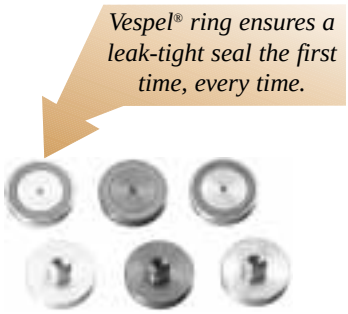
Inlet Liners for APEX ProSep™ 800 & ProSep™ 800 Plus GCs

	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.*

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.

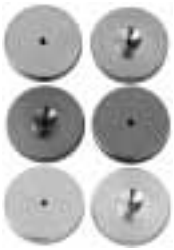


0.8mm ID Vespel® Ring Inlet Seal	**2-pk.**	**10-pk.**
Gold-Plated	21562	21563
Silcosteel®	21564	21565
Stainless Steel	21560	21561
1.2mm ID Vespel® Ring Inlet Seal	**2-pk.**	**10-pk.**
Gold-Plated	21568	21569
Silcosteel®	21570	21571
Stainless Steel	21566	21567

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.

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

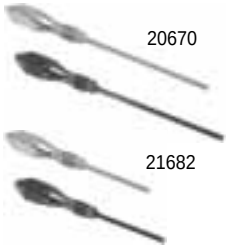
(Similar to Agilent part # G1531-80560.)

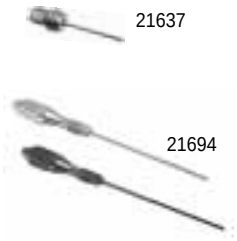
Description	qty.	cat.#	qty.	cat.#
Standard	ea.	21621	3-pk.	21682
High-Performance Silcosteel®	ea.	21620	3-pk.	21683

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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800-356-1688, ext. 3
or your local Restek representative.

Our extended hours are 8:00 a.m. to 6:00 p.m. EST, Monday thru Friday.

Technical Service Department

800-356-1688, ext. 4
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Our regular technical service hours are 8:00 a.m. to 7:00 p.m., Monday through Thursday, and 8:00 a.m. to 5:00 p.m. on Fridays.

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800-356-1688, ext. 4
or your local Restek representative.

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***Reach for Restek*—the company that chromatographers trust.**

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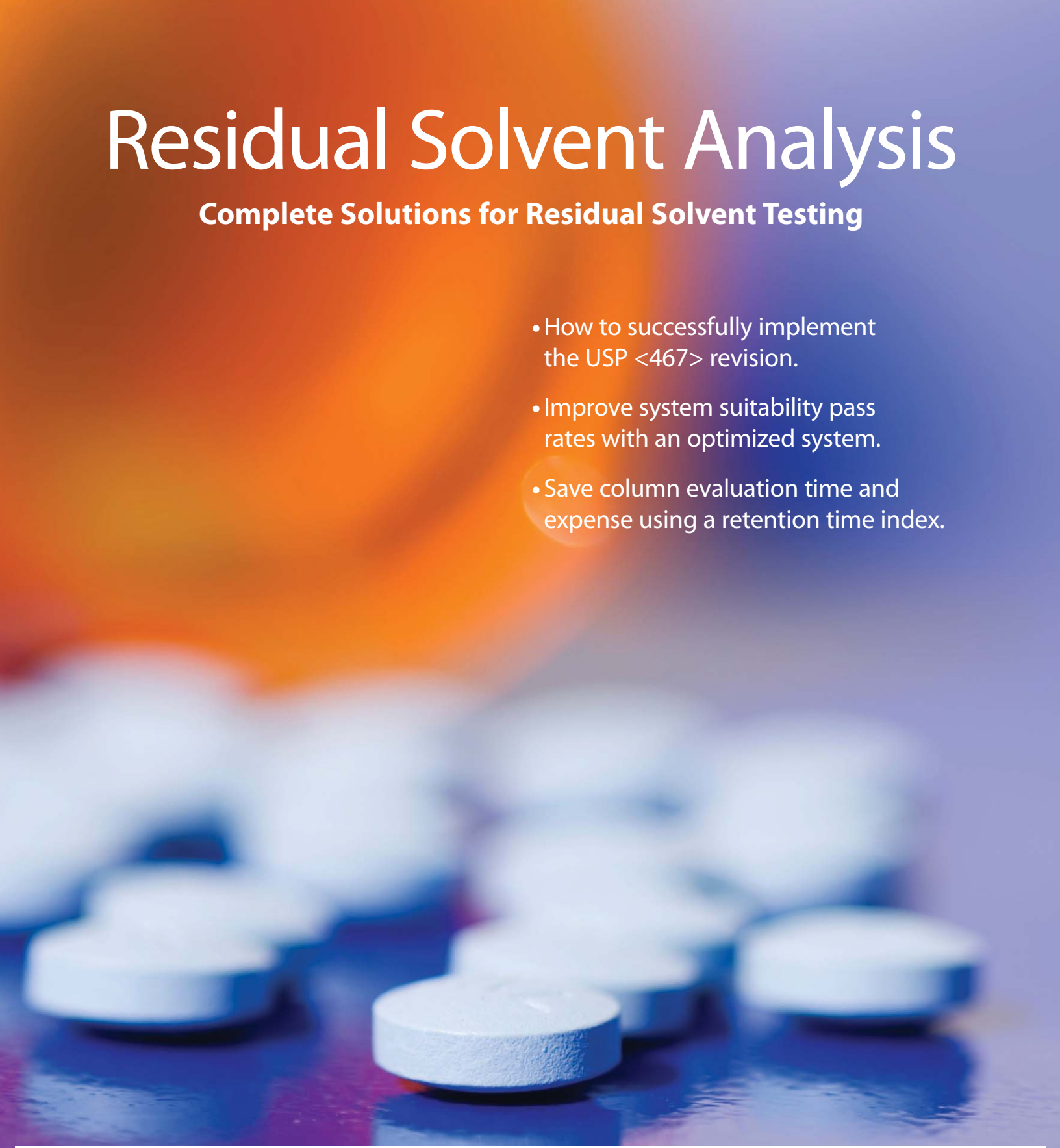
Residual Solvent Analysis

Complete Solutions for Residual Solvent Testing

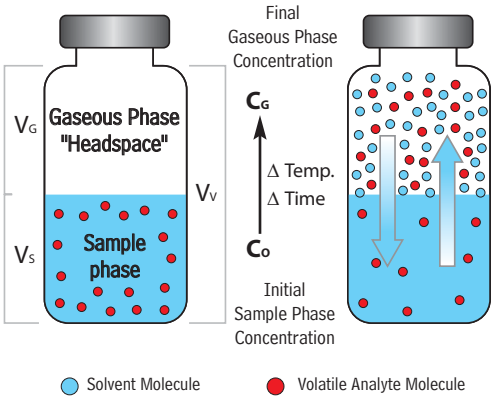
- How to successfully implement the USP <467> revision.
- Improve system suitability pass rates with an optimized system.
- Save column evaluation time and expense using a retention time index.



The Chemistry of Static Headspace Gas Chromatography

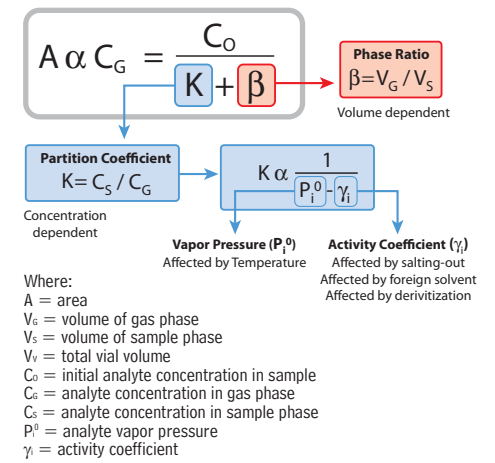
Improve Method Performance with Fundamentals

Figure 1 Volatile components partition into gaseous phase until equilibrium is reached.



Once the sample phase is introduced into the vial and the vial is sealed, volatile components diffuse into the gas phase until the headspace has reached a state of equilibrium as depicted by the arrows. The sample is then taken from the headspace.

Figure 2 Fundamental headspace relationship.



Where:
A = area
V_G = volume of gas phase
V_S = volume of sample phase
V_V = total vial volume
C_O = initial analyte concentration in sample
C_G = analyte concentration in gas phase
C_S = analyte concentration in sample phase
P_i^0 = analyte vapor pressure
γ_i = activity coefficient

Organic volatile impurities (OVIs), commonly referred to as residual solvents, are trace level chemical residues in drug substances and drug products that are byproducts of manufacturing or that form during packaging and storage. Drug manufacturers must ensure that these residues are removed, or are present only in limited concentrations. The International Conference on Harmonization (ICH) Q3C guideline lists the acceptable amounts of solvent residues that can be present. Methodology, both independently developed and compendial, should strive to coincide with this guideline. In this guide, we will take a comprehensive look at residual solvent analysis, in both theory and practice, and illustrate options for the practicing chromatographer.

The analysis of residual solvents is commonly performed using static headspace gas chromatography (HS/GC). The basic premise behind headspace analysis begins with the addition of an exact, known volume or weight of sample into a closed, sealed vial. This creates two distinct phases in the vial—a sample phase and a gaseous phase, or "headspace". Volatile components inside the sample phase, whether a solid or solution, can be extracted, or partitioned, from the sample phase into the headspace. An aliquot of the headspace can then be taken and delivered into a GC system for separation and detection. If we look at the anatomy of a headspace vial (Figure 1), we can begin to see the relationship of the vial components and how we can control these parameters to create analytical methods.

Residual solvent analysis by static HS/GC can be enhanced by careful consideration of two basic concepts—partition coefficient (K) and phase ratio (β). Partition coefficients and phase ratios work together to determine the final concentration of volatile compounds in the headspace of sample vials. Volatile components partition from the sample phase and equilibrate in the vial headspace. Striving for the lowest values for both K and β when preparing samples will result in higher concentrations of volatile analytes in the gas phase and, therefore, better sensitivity (Figure 2).

Controlling the Partition Coefficient

The partition coefficient (K) is defined as the equilibrium distribution of an analyte between the sample and gas phases. 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. K can be further described as a relationship between analyte vapor pressure (p_i^0) and activity coefficient (γ_i). In practice, K can be lowered by increasing the temperature at which the vial is equilibrated (vapor pressure) or by changing the composition of the sample matrix (activity coefficient) by adding an inorganic salt or a solvent of lesser solubility, often referred to as a foreign solvent. High salt concentrations and foreign solvents decrease analyte solubility in the sample phase (decrease activity) and promote transfer into the headspace, thus resulting in lower K values. The magnitude of this effect on K is not the same for all analytes. Compounds with inherent low K values in the matrix will experience little change in partition coefficient in response to the addition of a salt and temperature, while volatile compounds in a matrix of similar polarity will show the largest responses.

Adjusting the Phase Ratio

The phase ratio (β) is defined as the volume of the headspace over the volume of the sample in the vial. Lower values for β (i.e., larger sample sizes) will yield higher responses for compounds with inherently low K values. However, decreasing β will not always yield the increase in response needed to improve sensitivity. When β is decreased by increasing sample size, compounds with high K values will partition less into the headspace compared to compounds with low K values and yield correspondingly smaller changes in sensitivity.

Achieving USP<467> Compliance

Your Guide to Successfully Implementing the Revised Method

The USP general chapter <467> Residual Solvents is a widely used compendial method for identifying and quantifying residual solvents when no information is available on what solvents are likely to be present. In an attempt to harmonize with the ICH guidelines, the USP has proposed a more comprehensive method in the current USP 30/NF 25. This revision significantly increases the number of residual solvents to be routinely tested and includes three distinct procedures.[1]

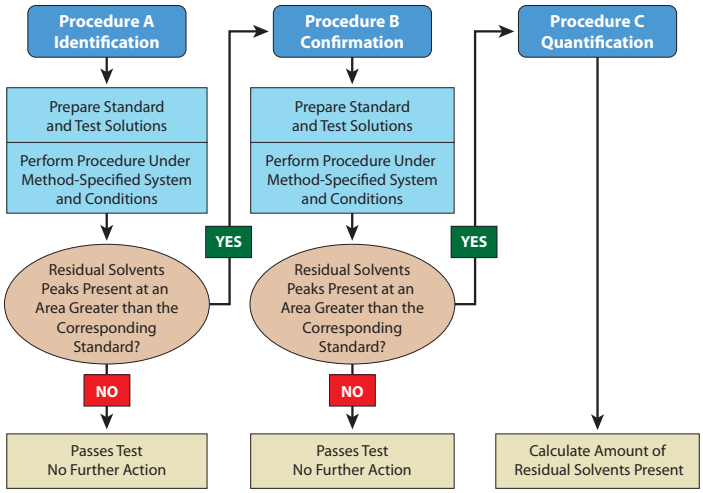
Initially set to become effective July 1, 2007, the implementation of the current version of USP <467> has been delayed until July 1, 2008. Until that time, the Other Analytical Procedures section of the previous version will be retained. However, in preparation for the implementation of the revised method, this application will comply with the procedure and criteria set forth in the USP30/NF25, second supplement (effective December 1, 2007) and the interim revision announcement.

Overview of Method

The revised USP <467> method consists of a static headspace extraction coupled with a gas chromatographic separation and flame ionization detection. In this guide we demonstrate the USP <467> application using two different types of headspace autosamplers. Procedure A was performed using a pressured loop autosampler and transfer line. Procedure B was performed using a heated syringe injection. Either system can be used to meet method requirements.

USP <467> is divided into two separate sections based upon sample solubility: water-soluble and water-insoluble articles. The methodology for both types of articles is similar, but the diluent used in both standard and sample preparations differs based upon the solubility of the test article. The test method consists of three procedures (A, B, and C), that are designed to identify, confirm, and then quantify residual solvents in drug substances and products (Figure 3).

Figure 3 Analytical flow chart for residual solvent testing under the revised USP <467> method.



[1] This number of analytes to be tested represents the sum of Class 1 and 2 residual solvents that can be effectively assayed using HS/GC. The actual number of analytes may be more if xylenes, ethyl benzene and *cis/trans* 1,2 dichloroethylene are differentiated, or if circumstances require the quantification of specific Class 3 residual solvents.

tech tip

Compatibility concerns?
Refer to the Septum Selection Guide at **www.restek.com/septaguide**

free literature

Download your free copy of our Technical Guide for Static Headspace Analysis from **www.restek.com/ovi**

lit. cat.# 59895A

Residual Solvents - Class 1

benzene	10mg/mL	1,1-dichloroethene	40
carbon tetrachloride	20	1,1,1-trichloroethane	50
1,2-dichloroethane	25		

In dimethyl sulfoxide, 1mL/ampul
cat. # 36279 (ea.)

Quantity discounts not available.

Residual Solvents Class 2 - Mix A (15 components)

acetonitrile	2.05mg/mL	methylcyclohexane	5.90
chlorobenzene	1.80	methylene chloride	3.00
cyclohexane	19.40	tetrahydrofuran	3.45
cis-1,2-dichloroethene	4.70	toluene	4.45
trans-1,2-dichloroethene	4.70	*m*-xylene	6.51
1,4-dioxane	1.90	*o*-xylene	0.98
ethylbenzene	1.84	*p*-xylene	1.52
methanol	15.00		

In dimethyl sulfoxide, 1mL/ampul
cat. # 36271 (ea.)

Residual Solvents Class 2 - Mix B (8 components)

chloroform	60µg/mL	nitromethane	50
1,2-dimethoxyethane	100	pyridine	200
n-hexane (C6)	290	tetralin	100
2-hexanone	50	trichloroethene	80

In dimethyl sulfoxide, 1mL/ampul
cat. # 36280 (ea.)

Quantity discounts not available.

Residual Solvents Class 2 - Mix C (8 components)

2-ethoxyethanol	800µg/mL	2-methoxyethanol (methyl Cellosolve®)	250
ethylene glycol	3,100	N-methylpyrrolidone	2,650
formamide	1,100	sulfolane	800
N,N-dimethylacetamide	5,450		
N,N-dimethylformamide	4,400		

In dimethyl sulfoxide, 1mL/ampul
cat. # 36273 (ea.)

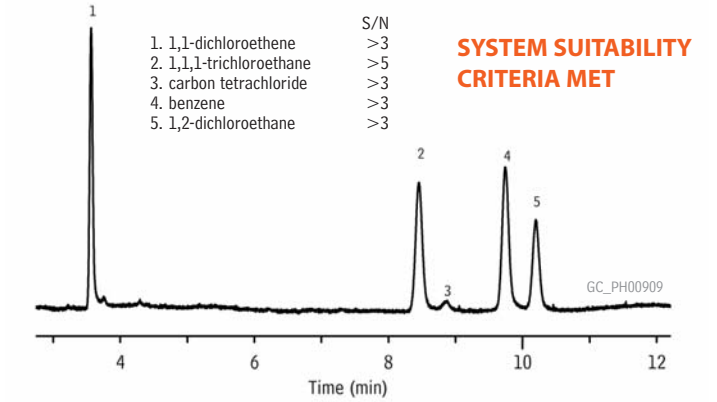
Analytical Reference Materials

The ICH guideline classifies residual solvents by class according to toxicity. Class 1 compounds are carcinogenic and pose a risk to both the consumer and the environment. The use of these solvents must be avoided or tightly controlled. Class 2 compounds are nongenotoxic animal carcinogens and their concentration should be limited. Both Class 1 and 2 compounds require chromatographic determination and are separated into 3 test mixes: Class 1 Mixture, Class 2 Mixture A, and Class 2 Mixture B. Class 3 compounds have low toxic potential. Concentration levels of up to 0.5% are acceptable and, therefore, they can be assayed by nonspecific techniques, such as weight loss on drying. Class 2 Mixture C is not used in the second supplement of USP 30/NF 25, but contains solvents that are not readily detectable by headspace analysis. These solvents should be assayed by other appropriately validated procedures.

Procedure A - Identification

Procedure A is the first step in the identification process and is performed on a G43 column to determine if any residual solvents are present in the sample at detectable levels. First, Class 1 standard and system suitability solutions and Class 2 Mix A standard solutions are assayed under the method-specified operating conditions to establish system suitability. All peaks in the Class 1 system suitability solution must have a signal-to-noise ratio not less than 3, the Class 1 standard solution must have a 1,1,1-trichloroethane response greater than 5, and the resolution of acetonitrile and dichloromethane must be not less than 1 in the Class 2 Mixture A solution. When system suitability has been achieved, the test solutions are assayed along with the Class 1 and Class 2 Mixtures A and B standard solutions. If a peak is determined in the sample that matches a retention time and has a greater response than that of a corresponding reference material, then Procedure B is performed for verification of the analyte. In the second supplement of USP 30/NF 25, an exemption is made for 1,1,1-trichloroethane, where a response greater than 150 times the peak response denotes an amount above the percent daily exposure limit. Figures 4 through 6 illustrate the analysis of Class 1, Class 2 Mixture A, and Class 2 Mixture B residual solvent mixes by Procedure A. The resolution between acetonitrile and dichloromethane was easily achieved using an Rtx®-1301 column.

Figure 4 USP residual solvent Class 1 standard solution on an Rtx®-1301 column (G43).

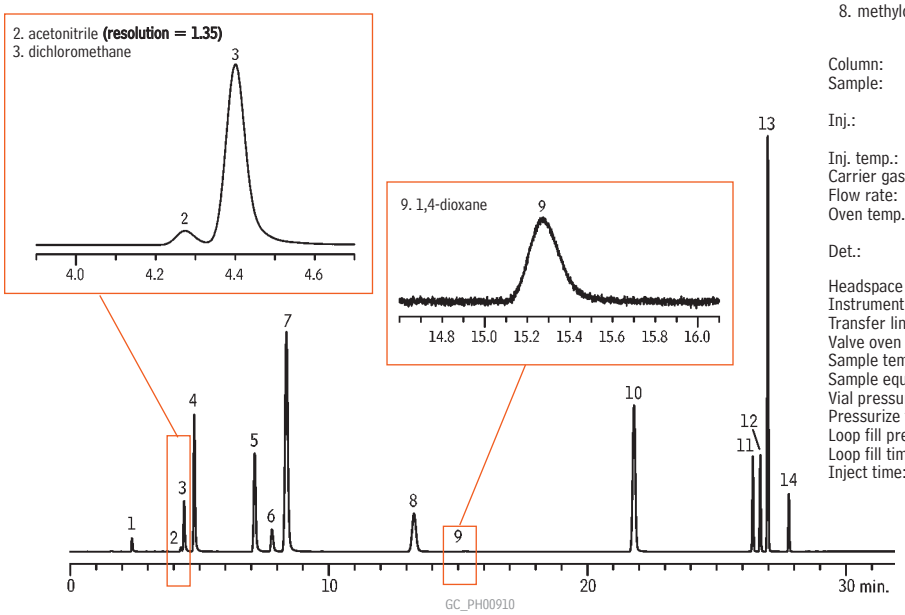


Column:	Rtx®-1301, 30m, 0.32mm ID, 1.8µm (cat.# 16092)	Headspace Conditions	
Sample:	USP <467> Class 1 standard solution (cat.# 36279) in 20mL headspace vial	Instrument:	Tekmar HT3
		Transfer line temp.:	105°C
Inj.:	headspace injection (split ratio 1:5), 1mm split liner, Siltek® deactivated (cat.# 20972-214.1)	Valve oven temp.:	105°C
		Sample temp.:	80°C
Inj. temp.:	140°C	Sample equil. time:	45 min.
Carrier gas:	helium, constant flow	Vial pressure:	10psi
Flow rate:	2.16mL/min., 35.3cm/sec.	Pressurize time:	0.5 min.
Oven temp.:	40°C for 20 min. to 240°C @ 10°C/min. (hold for 20 min.)	Loop fill pressure:	5psi
		Loop fill time:	2.00 min.
Det.:	FID @ 240°C	Inject time:	1.00 min.

Figure 5 USP residual solvent Class 2 Mixture A standard solution on an Rtx®-1301 column (G43).

SYSTEM SUITABILITY CRITERIA MET

1. methanol
2. acetonitrile (resolution=1.35)
3. dichloromethane
4. *trans*-1,2-dichloroethene
5. *cis*-1,2-dichloroethene
6. tetrahydrofuran
7. cyclohexane
8. methylcyclohexane
9. 1,4-dioxane
10. toluene
11. chlorobenzene
12. ethyl benzene
13. *m*-xylene/*p*-xylene
14. *o*-xylene



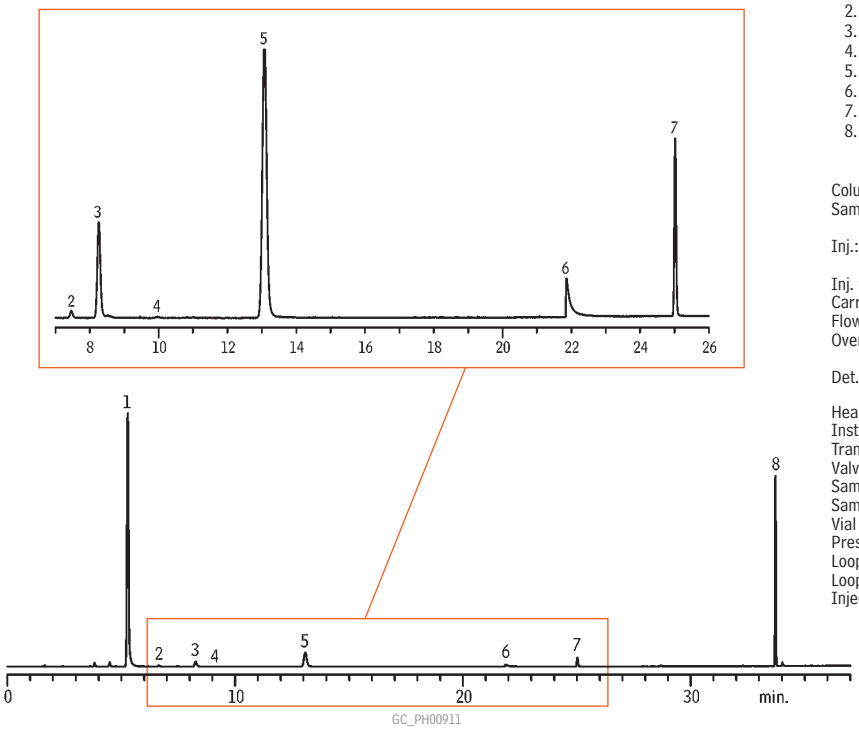
Column:	Rtx®-1301, 30m, 0.32mm ID, 1.8µm (cat.# 16092)
Sample:	USP <467> Class 2 Mixture B standard solution (cat.# 36280) in 20mL headspace vial
Inj.:	headspace injection (split ratio 1:5), 1mm split liner Siltek® deactivated (cat.# 20972-214.1)
Inj. temp.:	140°C
Carrier gas:	helium, constant flow
Flow rate:	2.16mL/min., 35.3cm/sec.
Oven temp.:	40°C for 20 min. to 240°C @ 10°C/min. (hold for 20 min.)
Det.:	FID @ 240°C

Headspace Conditions

Instrument:	Tekmar HT3
Transfer line temp.:	105°C
Valve oven temp.:	105°C
Sample temp.:	80°C
Sample equil. time:	45 min.
Vial pressure:	10psi
Pressurize time:	0.5 min.
Loop fill pressure:	5psi
Loop fill time:	2.00 min.
Inject time:	1.00 min.

Figure 6 USP residual solvent Class 2 Mixture B standard solution on an Rtx®-1301 column (G43).

1. hexane
2. nitromethane
3. chloroform
4. 1,2-dimethoxyethane
5. trichloroethylene
6. pyridine
7. 2-hexanone
8. tetralin



Column:	Rtx®-1301, 30m, 0.32mm ID, 1.8µm (cat.# 16092)
Sample:	USP <467> Class 2 Mixture B standard solution (cat.# 36280) in 20mL headspace vial
Inj.:	headspace injection (split ratio 1:5), 1mm split liner Siltek® deactivated (cat.# 20972-214.1)
Inj. temp.:	140°C
Carrier gas:	helium, constant flow
Flow rate:	2.16mL/min., 35.3cm/sec.
Oven temp.:	40°C for 20 min. to 240°C @ 10°C/min. (hold for 20 min.)
Det.:	FID @ 240°C

Headspace Conditions

Instrument:	Tekmar HT3
Transfer line temp.:	105°C
Valve oven temp.:	105°C
Sample temp.:	80°C
Sample equil. time:	45 min.
Vial pressure:	10psi
Pressurize time:	0.5 min.
Loop fill pressure:	5psi
Loop fill time:	2.00 min.
Inject time:	1.00 min.

Achieving USP<467> Compliance *(continued from page 5)*

Capillary Column—Procedure A

Rtx®-1301 (G43) Columns (fused silica)

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

ID	df (µm)	temp. limits	length	cat. #
0.32mm	1.80	-20 to 240°C	30-Meter	16092
0.53mm	3.00	-20 to 240°C	30-Meter	16085

Capillary Column—Procedure B

0.32mm	0.25	40 to 250°C	30-Meter	10624
0.53mm	0.25	40 to 250°C	30-Meter	10625

Procedure B - Confirmation

Once a residual solvent is identified and found to be above the percent daily exposure limit, Procedure B is performed to confirm analyte identity. A G16 capillary column is used here as a confirmation column, because it yields an alternate selectivity compared to a G43 column. The same standard and system suitability preparations are used in Procedures A and B. The system suitability requirements differ here in that the Class 1 standard solution must have a benzene response greater than 5 and the resolution of acetonitrile and *cis*-dichloroethene must not be less than 1 in the Class 2 Mixture A solution, a change from the original version. If the analyte identified in Procedure A again matches the retention time and exceeds the peak response of the reference materials (with the same exception to 1,1,1-trichloroethane), the analyst must quantify the analyte using Procedure C. Figures 7 through 9 illustrate the analysis of Class 1, Class 2 Mixture A, and Class 2 Mixture B residual solvent mixes on a Stabilwax® column. Again, the system suitability requirements were easily met.

Procedure C – Quantification

Once a residual solvent has been identified and verified, Procedure C is used to quantify the analyte by analyzing the sample against compound-specific reference materials. Individual standards are prepared by diluting the analyte in solution to a concentration of 1/20 of the concentration limit given under concentration limit Table 1 or 2 of the method. Following the procedure and instrument conditions in either Procedure A or B (whichever provides the most definitive results), a quantifiable result is produced. For water-insoluble articles, the same procedure is followed, except dimethylformamide or dimethylsulfoxide is used as the diluent.

Figure 7 USP residual solvent Class 1 standard solution on a Stabilwax® column (G16).

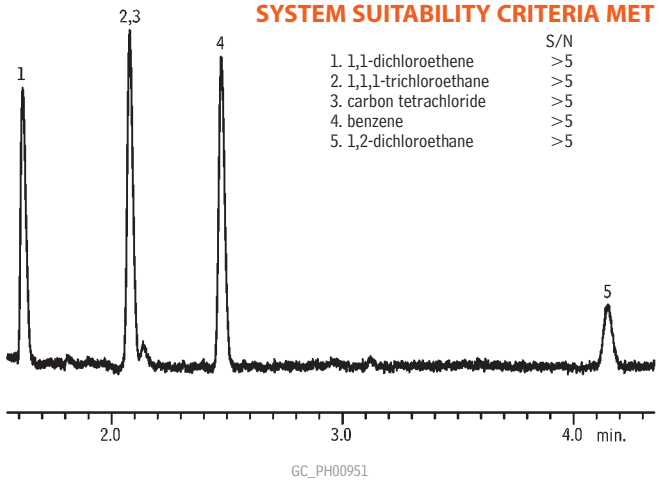


Figure 8 USP residual solvent Class 2 Mixture A standard solution on a Stabilwax® column (G16).

SYSTEM SUITABILITY CRITERIA MET—
RESOLUTION BETWEEN PEAKS 7 & 8 > 1.0

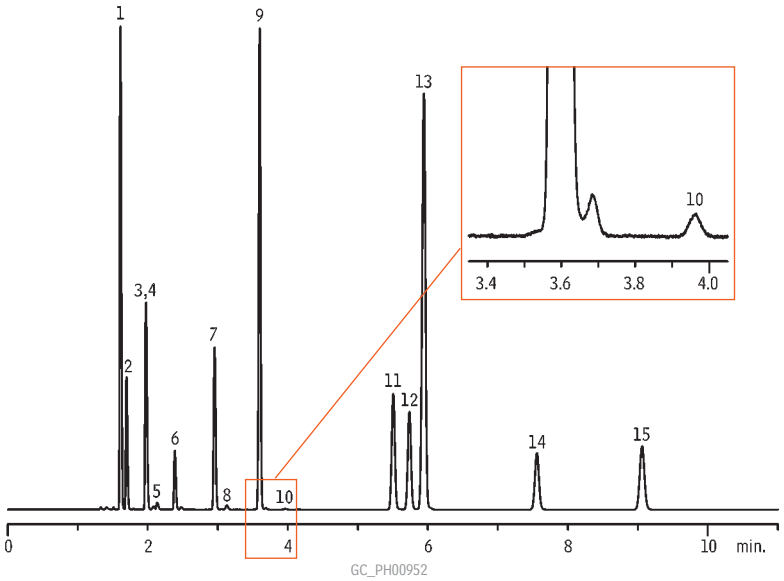
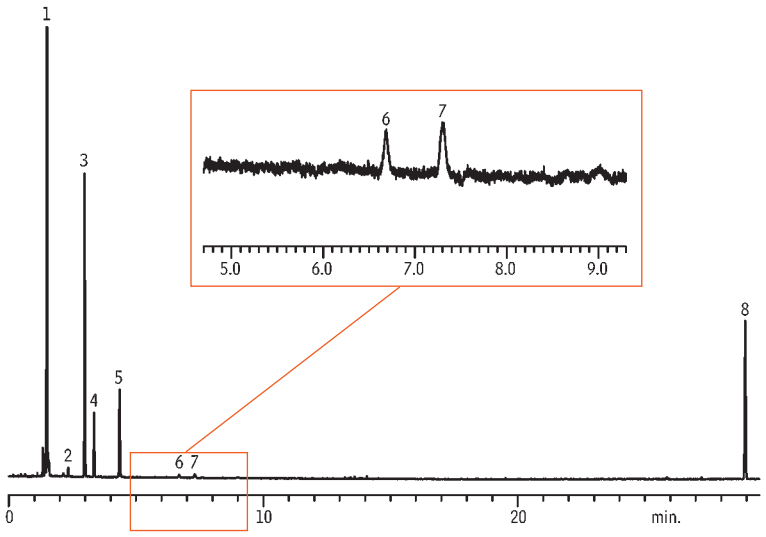


Figure 9 USP residual solvent Class 2 Mixture B standard solution on a Stabilwax® column (G16).



1. hexane
2. 1,2-dimethoxyethane
3. trichloroethylene
4. chloroform
5. 2-hexanone
6. nitromethane
7. pyridine
8. tetralin

Column:	Stabilwax®, 30m, 0.32mm ID, 0.25µm (cat.# 10624)
Sample:	USP Stock Standard Residual Solvents Class 2 Mix B (cat.# 36272) in 20mL headspace vial (cat.# 24685), water diluent
Inj.:	headspace injection (split ratio 1:5), 2mm splitless liner IP deactivated (cat.# 20712)
Inj. temp.:	140°C
Carrier gas:	helium, constant flow
Flow rate:	2.15mL/min., 35.2cm/sec.
Oven temp.:	50°C for 20 min. to 165°C @ 6°C/min. (hold for 20 min.)
Det.:	FID @ 250°C

Headspace Conditions

Instrument:	Overbrook Scientific HT200H
Syringe temp.:	100°C
Sample temp.:	80°C
Sample equil. time.:	45 min.
Injection vol.:	1.0mL
Injection speed:	setting 8
Injection dwell:	5 sec.

Optimize Your Testing Procedure

Tools, Tips, & Techniques for Improving Method Performance

Use Smaller Bore Liners for Better Efficiency

1mm Split Liners for Agilent GCs

ID* x OD & Length	qty.	cat.#
1mm Split**		
1.0mm x 6.3mm x 78.5mm	ea.	20972
1.0mm x 6.3mm x 78.5mm	5-pk.	20973

2mm Splitless Liners for Agilent GCs

ID* x OD & Length	qty.	cat.#
2mm Splitless		
2.0mm x 6.5mm x 78.5mm	ea.	20712
2.0mm x 6.5mm x 78.5mm	5-pk.	20713
2.0mm x 6.5mm x 78.5mm	25-pk.	20714

Split Liners for Varian 1075/1077 GCs

ID* x OD & Length	qty.	cat.#
1mm Split		
1.0mm x 6.3mm x 72mm	ea.	20970
1.0mm x 6.3mm x 72mm	5-pk.	20971

Split Liners for Shimadzu GCs

ID* x OD & Length	qty.	cat.#
1mm Split		
1.0mm x 5.0mm x 95mm	ea.	20976
1.0mm x 5.0mm x 95mm	5-pk.	20977
1.0mm x 5.0mm x 95mm	25-pk.	20978

SPME Liners for Shimadzu 17A, 2010, and 2014 GCs

ID* x OD & Length	qty.	cat.#
SPME Liner		
.75mm x 5.0mm x 95mm	ea.	22278
.75mm x 5.0mm x 95mm	5-pk.	22279

Zero Dilution Liners for PerkinElmer Auto SYS™ and Clarus GCs

ID* x OD & Length	qty.	cat.#
Zero Dilution Inner Liner		
1.0mm x 2.0mm x 73mm	ea.	22990
1.0mm x 2.0mm x 73mm	5-pk.	22991

Zero Dilution Outer Liner		
2.5mm x 6.2mm x 90mm	ea.	22992
2.5mm x 6.2mm x 90mm	5-pk.	22993

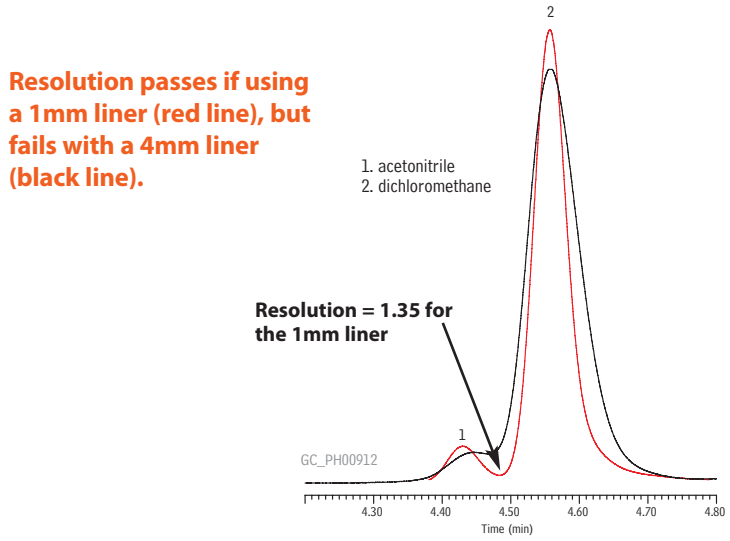
*Nominal ID at syringe needle expulsion point.
**Use this liner for increased sensitivity.

Implementing the revised method for USP<467> can be difficult if the instrument is not optimized correctly. 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. While the second supplement contains a change that allows for modifications to the split ratio, column and liner choices are critical to analytical success.

Use Smaller Bore Liners for Better Resolution

The function of an injection port in headspace analysis is very different than in direct liquid injection. In direct injection, the sample is vaporized in the injection port and larger volume liners (e.g., 4mm) are typically used since the liner must be able to accommodate the solvent expansion volume. In contrast, in headspace analysis, the sample is vaporized inside the headspace vial and the resulting gas sample is simply transferred into the injection port via a transfer line or syringe injection. Since solvent vaporization does not occur in the liner, a large volume liner is not needed and, in fact, the use of one can cause deleterious effects such as band broadening and decreased peak efficiency. For headspace applications, a smaller bore liner, preferably 1mm, is recommended. The smaller liner volume reduces band broadening by increasing linear velocity in the liner allowing faster sample transfer and improving resolution (Figure 10).

Figure 10 Improve system suitability pass rates using smaller bore liners.



Speed Up Method Development Using a Retention Time Index

ICH guideline Q3C states that residual solvents need only be tested when production or purification processes are known to result in the presence of such solvents. Therefore, in many cases exhaustive testing is not needed and individual validated methods for smaller, specific analyte lists are an option. To simplify column selection and reduce method development time, Restek has created a retention time index for ICH Class 1, 2, and 3 residual solvents on various phases (Table I). To use this index, simply locate the analytes of interest on the list and determine which phase gives the optimal amount of resolution—or difference in retention time—between your target compounds. A critical coelution is indicated by a failure to achieve a retention time difference of greater than 1.5 minutes.

Table I Reduce method development time—use a retention time index for column selection.

Retention time data collected using the following conditions:
G16 Stabilwax®: 30m, 0.25mm ID, 0.5µm df, Phase ratio: 125, Oven program: 40ºC, hold 1 min., to 190ºC @ 4ºC/min., hold 15 min., Carrier flow: 1.2mL/min., Dead time: 1.38 min. @ 45ºC
G16 Rtx®-WAX: 30m, 0.25mm ID, 0.5µm df, Phase ratio: 125, Oven program: 40ºC, hold 1 min., to 190ºC @ 4ºC/min., hold 15 min., Carrier flow: 1.2mL/min., Dead time: 1.40 min. @ 45ºC
G43 Rtx®-1301: 30m, 0.25mm ID, 1.0µm df, Phase ratio: 63, Oven program: 40ºC, hold 1 min., to 190ºC @ 4ºC/min., hold 15 min., Carrier flow: 1.2mL/min., Dead time: 1.40 min. @ 45ºC
G27 Rxi®-5ms: 30m, 0.25mm ID, 1.0µm df, Phase ratio: 63, Oven program: 40ºC, hold 1 min., to 190ºC @ 4ºC/min., hold 15 min., Carrier flow: 1.1mL/min., Dead time: 1.49 min. @ 45ºC
G1 Rtx®-1: 60m, 0.53mm ID, 3.00µm df, Phase ratio: 43, Oven program: 30ºC, hold 4 min., to 220ºC @ 4ºC/min., Carrier flow: 6.3mL/min., Dead time: 2.54 min. @ 35ºC
Rtx®-200: 60m, 0.53mm ID, 3.00µm df, Phase ratio: 43, Oven program: 30ºC, hold 4 min., to 220ºC @ 4ºC/min., Carrier flow: 7.8mL/min., Dead time: 2.22 min. @ 35ºC

Carrier gas: helium Compound	ICH Class	G16 Stabilwax® Retention Time	G16 Rtx®-WAX Retention Time	G43 Rtx®-1301 Retention Time	G27 Rxi®-5ms Retention Time	G1 Rtx®-1 Retention Time	NA Rtx®-200 Retention Time
1,1,1-trichloroethane	1	3.96	3.49	5.43	5.40	10.82	8.35
1,1,2-trichloroethene	2	15.72	14.28	10.99	9.77	16.75	14.94
1,1-dichloroethene	1	2.23	2.04	2.79	4.41	5.73	4.16
1,2-dichloroethane	1	8.80	7.68	6.15	5.46	10.38	9.74
cis-1,2-dichloroethene	2	6.50	5.65	4.79	2.88	8.71	7.11
trans-1,2-dichloroethene	2	3.63	3.20	3.55	3.54	7.17	5.16
1,2-dimethoxyethane	2	4.80	4.18	6.03	5.54	10.98	10.63
1,4-dioxane	2	8.55	7.49	7.86	7.26	13.54	14.34
1-butanol	3	11.13	10.08	7.18	5.76	11.49	10.13
1-pentanol	3	14.95	13.75	11.19	9.44	16.99	14.95
1-propanol	3	7.69	6.80	4.20	3.37	6.81	6.13
2-butanol	3	7.25	6.44	5.08	4.16	8.51	7.69
2-ethoxyethanol	2	13.99	12.70	8.69	7.36	13.91	13.99
2-methoxyethanol	2	12.42	11.11	6.02	5.14	9.83	10.74
2-methyl-1-propanol	3	9.32	8.40	6.00	4.79	*	*
2-propanol	3	4.81	4.25	3.00	2.55	4.91	4.69
3-methyl-1-butanol	3	13.42	12.25	9.86	8.26	15.28	13.55
acetic acid	3	22.47	20.34	6.52	4.61	8.84	8.96
acetone	3	3.02	2.64	2.89	2.50	4.64	7.68
acetonitrile	2	6.91	5.83	3.28	2.47	4.32	8.89
anisole	3	18.65	17.09	17.12	16.28	25.00	22.84
benzene	1	5.23	4.54	5.98	3.83	11.63	9.17
butyl acetate	3	8.86	7.88	12.12	11.38	19.43	19.63
carbon tetrachloride	1	3.96	3.49	5.61	5.90	11.89	7.42
chlorobenzene	2	13.91	12.54	13.55	13.14	21.56	18.48
chloroform	2	7.31	6.41	5.23	4.64	9.18	6.66
cumene	3	12.36	11.17	16.66	16.69	25.88	20.90
cyclohexane	2	2.16	2.01	5.37	5.89	*	*
dichloromethane	2	5.01	4.33	3.31	3.06	5.87	4.88
dimethylsulfoxide	3	26.47	24.43	16.62	13.01	18.81	30.95
ethanol	3	4.98	4.37	2.52	2.19	4.03	3.80
ethyl acetate	3	4.08	3.56	4.87	4.44	9.04	10.35
ethyl benzene	2	10.72	9.58	13.86	13.81	22.54	18.18
ethyl ether	3	1.72	1.63	2.58	2.67	5.34	3.87
ethyl formate	3	3.16	2.78	3.00	2.78	5.46	6.48
ethylene glycol	2	28.06	26.23	10.77	6.63	12.59	13.86
formamide	2	32.99	30.93	11.85	7.30	12.72	19.93
formic acid	3	24.64	22.09	5.19	2.60	5.59	5.06
heptane	3	1.98	1.86	6.34	6.98	14.18	7.84
hexane	2	1.65	1.58	3.77	4.11	9.06	4.86
isobutyl acetate	3	6.99	6.18	10.39	9.69	17.35	18.02
isopropyl acetate	3	4.26	3.74	6.19	5.71	11.47	12.38
methanol	2	4.23	3.64	1.96	1.80	3.14	2.93
methyl acetate	3	3.19	2.80	3.17	2.93	5.80	7.10
methylbutyl ketone	2	9.10	8.05	11.81	10.50	17.94	20.81
methylcyclohexane	2	2.50	2.30	7.31	7.95	15.49	9.21
methylethyl ketone	3	4.33	3.76	4.90	4.09	7.99	11.55
methylisobutyl ketone	3	6.84	5.97	9.64	8.49	15.35	18.41
m-xylene	2	11.21	10.04	15.46	14.17	23.01	18.78
N,N-dimethylacetamide	2	20.75	19.01	12.95	13.96	21.42	30.00
N,N-dimethylformamide	2	18.04	16.26	13.09	10.23	16.52	26.19
nitromethane	2	11.82	10.31	4.84	3.53	6.30	12.01
N-methylpyrrolidone	2	29.84	27.86	25.09	21.85	29.99	38.08
o-xylene	2	12.79	11.51	15.46	15.26	24.23	20.33
pentane	3	1.49	1.45	2.39	2.62	5.36	3.29
propyl acetate	3	5.98	5.29	8.03	7.44	*	*
p-xylene	2	10.98	9.82	14.29	15.27	22.99	18.69
pyridine	2	12.64	11.24	9.60	8.57	15.40	16.45
sulfolane	2	47.62	43.31	34.02	28.90	36.76	48.67
tert-butylmethyl ether	3	1.94	1.82	3.50	3.59	7.52	5.73
tetrahydrofuran	3	3.63	3.19	5.12	4.90	9.81	9.48
tetralin	2	25.12	23.48	27.49	27.44	37.27	31.72
toluene	2	7.86	6.91	9.80	9.66	17.36	14.00
1,1-diethoxypropane	—	5.42	4.84	11.39	11.38	19.82	15.08
2,2-dimethoxypropane	—	3.11	2.79	5.48	5.55	11.37	8.67
2-chloropropane	—	1.96	1.82	2.67	2.66	5.20	4.61
2-methylpentane	—	1.58	1.52	3.22	3.56	7.72	4.32
acetaldehyde	—	2.05	1.85	1.86	1.84	3.14	3.90
chloroethane	—	1.83	1.71	2.14	2.10	3.97	3.55
chloromethane	—	1.63	1.55	1.70	1.70	3.01	2.73
ethylene oxide	—	2.05	1.86	1.89	2.02	3.59	3.92
formaldehyde	—	2.25	1.57	1.68	1.58	2.66	2.59
isoamyl acetate	—	10.51	9.43	14.84	14.18	22.80	22.62
isooctane	—	1.85	1.75	5.84	6.59	13.66	8.07
isopropyl ether	—	1.86	1.76	4.03	4.23	9.03	5.83
methyl cyclopentane	—	1.91	1.79	4.50	4.93	10.41	5.81
methyl isopropyl ketone	—	4.93	4.29	6.58	5.69	11.04	14.47
methylal	—	2.26	2.06	2.84	2.82	5.65	5.09
trichloroethene	—	6.50	5.70	7.07	7.05	13.58	9.75
water	—	8.24	7.18	1.74	1.68	2.75	2.57

* Not determined

Restek Offers An Extensive Selection of Capillary Columns

For Successful Method Development & Validation

free literature

Genuine Restek Replacement Parts

Use our handy new Genuine Restek Replacement Parts (GRRP) mini-catalogs to help you select the supplies and replacement parts you need for your specific GC. We now have customized GRRP mini-catalogs for each major instrument manufacturer to simplify your product search. Download these and other pieces from our website at **www.restek.com/grrp.**

For Agilent GCs
(lit. cat.# 59627F)

For Agilent 5890 GCs
(lit. cat.# 580216)

For PerkinElmer GCs
(lit. cat.# 580038)

For Shimadzu GCs
(lit. cat.# 580037)

For Thermo Scientific GCs
(lit. cat.# 580039)

For Varian GCs
(lit. cat.# 59224A)

Rtx®-624 Columns (fused silica) G43

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

ID	df (µm)	temp. limits	30-Meter	60-Meter
0.25mm	1.40	-20 to 240°C	10968	10969
0.32mm	1.80	-20 to 240°C	10970	10972
0.53mm	3.00	-20 to 240°C	10971	10973
ID	**df (µm)**	**temp. limits**	**20-Meter**	**40-Meter**
0.18mm	1.00	-20 to 240°C	40924	40925

Rtx®-1301 Columns (fused silica) G43

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

ID	df (µm)	temp. limits*	30-Meter	60-Meter
0.25mm	0.50	-20 to 270°C	16038	16041
	1.00	-20 to 260°C	16053	16056
	1.40	-20 to 240°C		16016
0.32mm	0.50	-20 to 270°C	16039	16042
	1.00	-20 to 260°C	16054	16057
	1.50	-20 to 250°C	16069	16072
	1.80	-20 to 240°C	16092	16093
0.53mm	0.50	-20 to 270°C	16040	16043
	1.00	-20 to 260°C	16055	16058
	1.50	-20 to 250°C	16070	16073
	3.00	-20 to 240°C	16085	16088

Stabilwax® Columns (fused silica) G16

(Crossbond® Carbowax® polyethylene glycol)

ID	df (µm)	temp. limits	30-Meter	60-Meter
0.25mm	0.25	40 to 250°C	10623	10626
	0.50	40 to 250°C	10638	10641
0.32mm	0.25	40 to 250°C	10624	10627
	0.50	40 to 250°C	10639	10642
	1.00	40 to 240/250°C	10654	10657
0.53mm	1.00	40 to 240/250°C	10655	10658
	1.50	40 to 230/240°C	10669	10672
	2.00	40 to 220/230°C	10670	

also **available**

Custom Column Lengths:
If you do not see the column dimension you need, call our customer service team, and we will make the column for you.

Rxi®-5ms Columns (fused silica) G27

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	30-Meter	60-Meter
0.25mm	0.50	-60 to 330/350°C	13438	13441
	1.00	-60 to 330/350°C	13453	13456
0.32mm	0.50	-60 to 330/350°C	13439	13442
	1.00	-60 to 330/350°C	13454	13457
0.53mm	1.00	-60 to 330/350°C	13455	
	1.50	-60 to 330/350°C	13470	
ID	**df (µm)**	**temp. limits**	**20-Meter**	
0.18mm	0.18	-60 to 330/350°C	13402	
	0.30	-60 to 330/350°C	13409	
	0.36	-60 to 330/350°C	13411	

Rtx®-1 Columns (fused silica) G1

(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	30-Meter	60-Meter
0.25mm	0.50	-60 to 330/350°C	10138	10141
	1.00	-60 to 320/340°C	10153	10156
0.32mm	1.00	-60 to 320/340°C	10154	10157
	1.50	-60 to 310/330°C	10169	10172
	3.00	-60 to 280/300°C	10184	10187
	4.00	-60 to 280/300°C	10198	
	5.00	-60 to 260/280°C	10178	10180
0.53mm	1.50	-60 to 310/330°C	10170	10173
	3.00	-60 to 270/290°C	10185	10188
	5.00	-60 to 270/290°C	10179	10183
	7.00	-60 to 240/260°C	10192	10193
ID	**df (µm)**	**temp. limits**	**20-Meter**	**40-Meter**
0.18mm	0.20	-60 to 330/350°C	40102	40103
	0.40	-60 to 320/340°C	40111	40112

Rtx®-200 Columns (fused silica)

(Crossbond® trifluoropropylmethyl polysiloxane)

ID	df (µm)	temp. limits*	30-Meter	60-Meter
0.25mm	0.50	-20 to 310/330°C	15038	15041
	1.00	-20 to 290/310°C	15053	15056
0.32mm	1.00	-20 to 290/310°C	15054	15057
	1.50	-20 to 280/300°C	15069	15072
0.53mm	1.00	-20 to 290/310°C	15055	15058
	1.50	-20 to 280/300°C	15070	15073
	3.00	-20 to 260/280°C	15085	15088
ID	**df (µm)**	**temp. limits**	**20-Meter**	**40-Meter**
0.18mm	0.20	-20 to 310/330°C	45002	45003
	0.40	-20 to 310/330°C	45011	45012

*Maximum temperatures listed are for 15- and 30-meter lengths. Longer lengths may have a slightly reduced maximum temperature.

Simplify Lab Work with Innovative Accessories

Dual Vespel® Ring Inlet 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 into the carrier gas, increasing column lifetime.


Patented.

Washerless, leak-tight seals for Agilent GCs

0.8mm ID Dual Vespel Ring Inlet Seal	2-pk./price	10-pk./price
Gold-Plated	21240	21241
Siltek Treated	21242	21243
Stainless Steel	21238	21239
1.2mm ID Dual Vespel Ring Inlet Seal	**2-pk./price**	**10-pk./price**
Gold-Plated	21246	21247
Siltek Treated	21248	21249
Stainless Steel	21244	21245

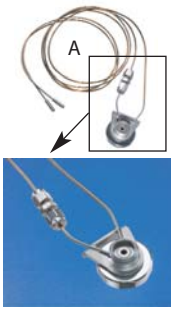
Dual Vespel® Ring Cross-Disk Inlet Seals for Agilent GCs

- Ideal for high-flow split applications.
- Washerless, leak-tight seals.

0.8mm ID Dual Vespel Ring Cross-Disk Inlet Seal	2-pk./price	10-pk./price
Gold-Plated	22083	22084
Siltek Treated	22085	22086
Stainless Steel	22087	22088

Injection Port Weldments for Agilent GCs

Easily attach your autosampler with pre-installed low dead volume fittings.

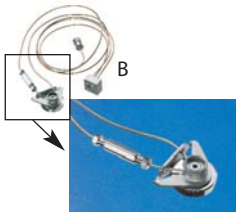


For Agilent GCs with Tekmar Transfer Lines

Description	qty.	cat.#
A) Weldment for Agilent 6890 GCs	ea.	22664
Weldment for Agilent 6890 GCs with optional canister filter	ea.	22668
Weldment for Agilent 5890 GCs	ea.	22666

For Agilent GCs with OI Purge and Trap Systems

Description	qty.	cat.#
B) Weldment for Agilent 6890 GCs	ea.	22665
Weldment for Agilent 6890 GCs with optional canister filter	ea.	22669
Weldment for Agilent 5890 GCs	ea.	22667



FID Replacement Jets

Standard Version

- Engineered with a fluted tip to guide the capillary column into the jet.
- Threads specially coated for easy installation and removal.
- Special processing ensures the highest degree of cleanliness.

High-Performance Version

- Similar to the standard version, but Siltek® treated.
- Extremely inert, for use with active compounds.

Capillary Adaptable FID Replacement Jet for Agilent 5890/6890/6850 GCs

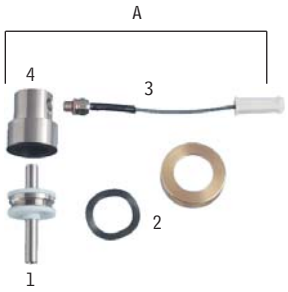
0.011-Inch ID Tip	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
Standard, 0.011-Inch ID Tip	19244-80560	ea.	20670	3-pk.	20671
High-Performance Siltek Treated, 0.011-Inch ID Tip	19244-80560	ea.	20672	3-pk.	20673

Capillary Dedicated FID Replacement Jet for Agilent 6890/6850/7890 GCs

0.011-Inch ID Tip	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
Standard, 0.011-Inch ID Tip	G1531-80560	ea.	21621	3-pk.	21682
High-Performance Siltek Treated, 0.011-Inch ID Tip	G1531-80560	ea.	21620	3-pk.	21683

did you **know**?

Restek carries a full line of FID replacement jets. Visit **www.restek.com** for a complete selection.



Direct Replacement FID Collector Assembly Kit for Agilent 6890/6850/7890 GCs

- Constructed of high-quality stainless steel.
- Meets or exceeds manufacturer's performance.

Description	Similar to Agilent part #	qty.	cat.#
A) FID Collector Assembly Kit (includes insulator)	G1531-60690	kit	21699
FID Collector Assembly Kit w/Siltek Ignitor Castle	—	kit	21132

Replacement FID Parts for Agilent 6890/6850/7890 GCs

- Meets or exceeds manufacturer's performance.

Description	Similar to Agilent part #	qty.	cat.#
1) FID Collector (includes insulators)	G1531-20690 G1531-20700	ea.	21139
2) FID Collector Nut and Washer	19231-20940 5181-3311	set	21136
3) FID Ignitor*	19231-60680	ea.	21001
4) FID Ignitor Castle	19231-20910	ea.	21137
Siltek FID Ignitor Castle	—	ea.	21135

*Also fits OI Analytical 4410 detector (similar to OI part # 191833).

The New Restek Electronic Leak Detector!

We are pleased to introduce the new, enhanced Restek Electronic Leak Detector. With our new unit you'll receive the great performance that you've come to trust from our current Leak Detector; but with new features designed with the end-user in mind, including:

- A sleek, new ergonomic, hand-held design.
- Rugged side grips for added durability.
- Handy probe storage for cleanliness.
- Longer battery lifetime.
- Automatic shut-off capabilities.
- A convenient carrying and storage case.
- A universal power adapter set (US, European, UK and Australian plugs included).

Available July 2008

Backed by a 1 year warranty, the new Restek Leak Detector will again set an industry standard for performance and affordability in a hand-held Leak Detector.

We will have units available for delivery in July 2008. Don't miss this opportunity to reserve your new Leak Detector. Call Restek customer service to reserve yours today! To find out more, visit **www.restek.com/leakdetector.**

Description	qty	cat.#
Leak Detector with Universal Adapter Set	ea	22839

Order your unit today! 1-800-356-1688, ext. 3

tech tip

Avoid using liquid leak detectors on a capillary system! Liquids can be drawn into the system.

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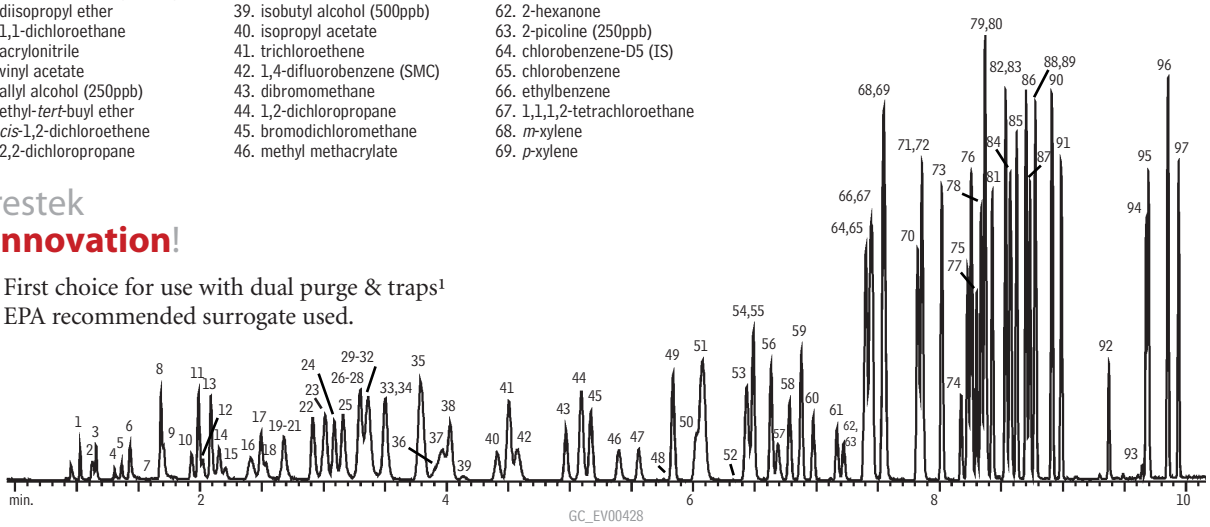
Volatiles

Volatile organic compounds (VOCs) are usually analyzed using a purge and trap system connected to a GC. The column used must have a selective stationary phase to resolve the volatile pollutants, have a sufficient film thickness to retain and resolve the low boiling volatile compounds (i.e., dichlorodifluoromethane), and must be thermally stable to elute the high boiling volatiles compounds (i.e., hexachlorobutadiene & naphthalene).

The first fused silica columns used for analyzing volatiles were based on diphenyl/dimethyl polysiloxane stationary phases. However, resolution of gases has always been problematic with these phases. Restek designed the Rtx®-VMS column specifically to optimize separation of volatiles in the most commonly used EPA volatiles methods. A faster oven ramp rate is possible because these compounds elute farther apart on the Rtx®-VMS phase, eliminating partial coelutions that interfere with quantification. Using the EPA suggested surrogates (i.e., chlorobenzene-d5) analysis time can be less than 10 minutes with a narrow bore column, allowing you to connect two purge and trap units to one GC/MS instrument – significantly increasing sample throughput.

Figure 1 Excellent resolution of bromomethane and chloroethane, as well as challenging isomer pairs like 2-/4-chlorotoluene on the Rtx®-VMS column.

1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. ethanol (2500ppb)
8. 1,1-dichloroethene
9. carbon disulfide (40ppb)
10. allyl chloride
11. methylene chloride
12. acetone
13. *trans*-1,2-dichloroethene
14. methyl *tert*-butyl ether
15. *tert*-butyl alcohol (100ppb)
16. diisopropyl ether
17. 1,1-dichloroethane
18. acrylonitrile
19. vinyl acetate
20. allyl alcohol (250ppb)
21. ethyl-*tert*-buyl ether
22. *cis*-1,2-dichloroethene
23. 2,2-dichloropropane
24. bromochloromethane
25. chloroform
26. ethyl acetate
27. carbon tetrachloride
28. methyl acrylate
29. propargyl alcohol (500ppb)
30. dibromofluoromethane (SMC)
31. tetrahydrofuran
32. 1,1,1-trichloroethane
33. 2-butanone
34. 1,1-dichloropropene
35. benzene
36. pentafluorobenzene (IS)
37. *tert*-amyl-methyl ether
38. 1,2-dichloroethane
39. isobutyl alcohol (500ppb)
40. isopropyl acetate
41. trichloroethene
42. 1,4-difluorobenzene (SMC)
43. dibromomethane
44. 1,2-dichloropropane
45. bromodichloromethane
46. methyl methacrylate
47. *n*-propyl acetate
48. 2-chloroethanol (2500ppb)
49. *cis*-1,3-dichloropropene
50. toluene-d8 (SMC)
51. toluene
52. pyridine (250ppb)
53. tetrachloroethene
54. 4-methyl-2-pentanone
55. *trans*-1,3-dichloropropene
56. 1,1,2-trichloroethane
57. ethyl methacrylate
58. dibromochloromethane
59. 1,3-dichloropropane
60. 1,2-dibromoethane
61. *n*-butyl acetate
62. 2-hexanone
63. 2-picoline (250ppb)
64. chlorobenzene-D5 (IS)
65. chlorobenzene
66. ethylbenzene
67. 1,1,1,2-tetrachloroethane
68. *m*-xylene
69. *p*-xylene
70. *o*-xylene
71. stryrene
72. bromoform
73. isopropylbenzene
74. 4-bromo-1-fluorobenzene (SMC)
75. bromobenzene
76. *n*-propylbenzene
77. 1,1,2,2-tetrachloroethane
78. 2-chlorotoluene
79. 1,3,5-trimethylbenzene
80. 1,2,3-trichloropropane
81. 4-chlorotoluene
82. *tert*-butylbenzene
83. pentachloroethane
84. 1,2,4-trimethylbenzene
85. *sec*-butylbenzene
86. *p*-isopropyltoluene
87. 1,3-dichlorobenzene
88. 1,4-dichlorobenzene-d4 (IS)
89. 1,4-dichlorobenzene
90. *n*-butylbenzene
91. 1,2-dichlorobenzene
92. 1,2-dibromo-3-chloropropane
93. nitrobenzene (250ppb)
94. hexachlorobutadiene
95. 1,2,4-trichlorobenzene
96. naphthalene
97. 1,2,3-trichlorobenzene



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innovation!

- First choice for use with dual purge & traps[1]
- EPA recommended surrogate used.

Column:	Rtx®-VMS 20m, 0.18 mm ID, 1.00µm (cat.# 49914)	Interface:	0.53mm ID Silcosteel® tubing transfer line
Conc.:	10ppb in 5mL of RO water unless otherwise noted; ketones at 2.5X		1:40 split at injection port. 1mm ID liner.
Concentrator:	Tekmar LSC-3100 Purge and Trap	Oven temp.:	50°C (hold 4 min.) to 100°C @ 18°C/min. (hold 0 min.) to 230°C @ 40°C/min. (hold 3 min.)
Trap:	Vocarb 3000 (type K)	Carrier gas:	helium @ ~1.0mL/min. constant flow
Purge:	11 min. @ 40mL/min. (ambient temperature)		Adjust dichlorodifluoromethane to a retention time of 1.03 min. @ 50°C.
Dry purge:	1 min. @ 40mL/min.		
Desorb preheat:	245°C	Detector:	Agilent 5973 MSD
Desorb:	250°C for 2 min., flow 40mL/min.	Scan range:	35-300amu
Bake:	260°C for 8 min.		

[1]A.L. Hilling and G. Smith, Environmental Testing & Analysis, 10(3), 15-19, 2001.

Recommended Column

Rtx®-VMS Columns (fused silica)
(proprietary Crossbond® phase)

ID	df (µm)	temp. limits	length	cat. #
0.18mm	1.00	-40 to 240/260°C	20-Meter	49914

Analytical Reference Materials

8260A Internal Standard Mix
chlorobenzene-d5 fluorobenzene
1,4-dichlorobenzene-d4
2,500µg/mL each in P&T methanol, 1mL/ampul
cat. # 30241 (ea.)

8260 Internal Standard Mix
chlorobenzene-d5 1,4-difluorobenzene
1,4-dichlorobenzene-d4 pentafluorobenzene
2,500µg/mL each in P&T methanol, 1mL/ampul
cat. # 30074 (ea.)

8260A Surrogate Mix
4-bromofluorobenzene 1,2-dichloroethane-d4
dibromofluoromethane toluene-d8
2,500µg/mL each in P&T methanol, 1mL/ampul
cat. # 30240 (ea.)

8260 Surrogate Mix
4-bromofluorobenzene toluene-d8
dibromofluoromethane
2,500µg/mL each in P&T methanol, 1mL/ampul
cat. # 30073 (ea.)

8260B MegaMix® Calibration Mix (76 components)
Please visit us online for compound list
2,000µg/mL each in P&T methanol, 1mL/ampul
cat. # 30633 (ea.)

8260B MegaMix® Calibration Mix Kit
30633: 8260B MegaMix®
30265: 2-chlorethyl vinyl ether
Contains 1mL each of these mixtures.
cat. # 30475 (kit)

502.2 Calibration Mix #1 (gases)
bromomethane dichlorodifluoromethane (CFC-12)
chloroethane trichlorofluoromethane (CFC-11)
chloromethane vinyl chloride
200µg/mL each in P&T methanol, 1mL/ampul
cat. # 30439 (ea.)
2,000µg/mL each in P&T methanol, 1mL/ampul
cat. # 30042 (ea.)

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
cat. # 30006 (ea.)

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
cat. # 30465 (ea.)

Reduce Dead Volume, Contamination, & Cold Spots

The injection port can be a source for dead volume, which is especially critical when dealing with a sample in the gas phase. The severity of the problem is a combination of the inside diameter of the injection port liner and the total desorb flow through the port. To reduce dead volume in the injection port, use a 1mm ID inert split liner. Always be sure to use insulation where the transfer line attaches to the inlet line since this is a cold spot that will condense high molecular weight analytes.

Transfer lines often are the first place contamination occurs. When the response factor for bromoform fails the method criteria, changing the transfer line is the first step to getting the system working again. Replace your transfer line with our Siltek® deactivated tubing, for optimum performance.

1mm Split Inlet Liner for Agilent GCs

ID*/OD & Length (mm)	cat.# ea.	cat.# 5-pk.
1.0 ID 6.3 OD x78.5	20972	20973

*Nominal ID at syringe needle expulsion point.
Also available with Siltek® deactivation, upon request.

Siltek®/Sulfinert® Treated Coiled 304 Grade Stainless Steel Tubing

Our most popular grade of tubing.
- chromatography applications.
- gas delivery systems.
- lower pressures.
- inert applications.

ID	OD	cat.#	5-24 ft.	25-199 ft.	200-399 ft.	>400 ft.
0.040" (1.02mm)	1/16" (1.59mm)	22505				

*0.020" wall thickness

An extra charge is applied for cutting Siltek®/Sulfinert®, Silcosteel®, or Silcosteel®-CR tubing, calculated from the total number of pieces produced for each line item.

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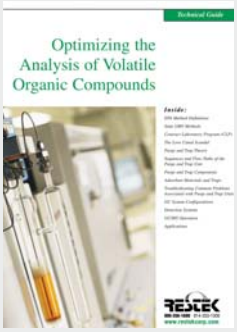
Optimizing the Analysis of Volatile Organic Compounds
lit. cat.# 59887A

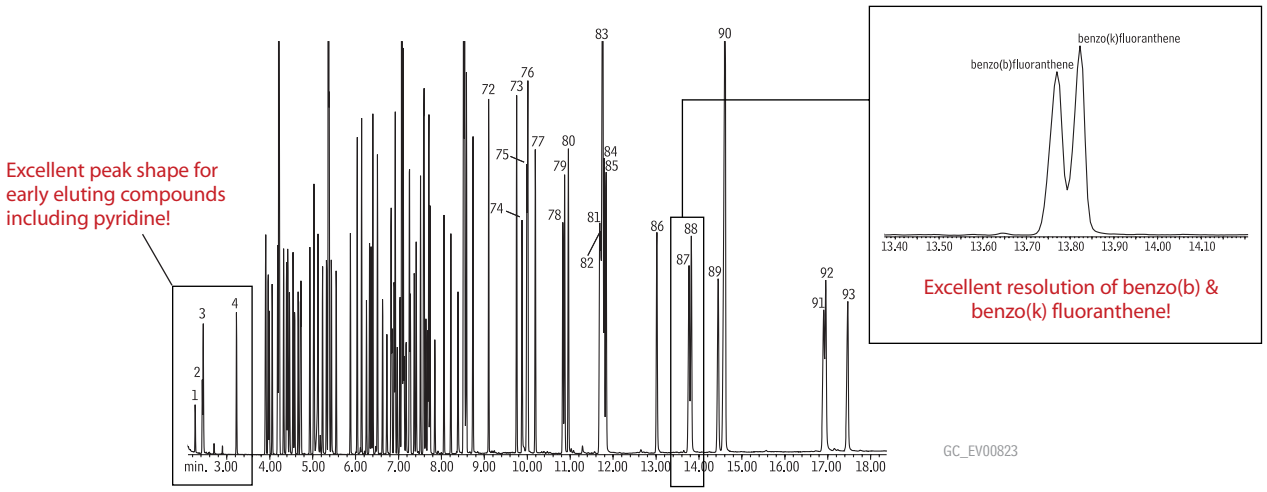
Contact your Restek representative, to request your free copy!



Semivolatiles

Semivolatile analysis is a challenging area covering a wide range of compound classes – neutral, acidic, and basic compounds, including anilines, phenols, PAHs, and more – that differ in both volatility and reactivity. While the chromatography is complicated by a broad list of target analytes, many problems can be avoided by proper attention to the inlet system and an informed column choice.

Figure 1 Separate greater than 90 semivolatile compounds in less than 18 minutes, using an Rxi™-5ms column.



For complete identifications, please visit www.restek.com/rxi

Column:	Rxi™-5ms, 30m, 0.25mm ID, 0.25µm (cat.# 13423)
Sample:	US EPA Method 8270D mix: 8270 MegaMix™ (cat.# 31850), Benzoic Acid Standard (cat.# 31879), Benzidine Standard (cat.# 31852), Acid Surrogate Mix (cat.# 31025), B/N Surrogate Standard Mix (cat.# 31887), 1,4-Dioxane (cat.# 31853)
Inj.:	1.0µL, 10ppm each analyte (10ng on column), splitless (hold 0.1 min.) 4mm Drilled Uniliner® inlet liner (hole at bottom) (cat.# 20756)
Instrument:	Agilent 6890
Inj. temp.:	250°C
Carrier gas:	helium, constant flow
Flow rate:	1.2mL/min.
Oven temp.:	50°C (hold 0.5 min.) to 265°C @ 25°C/min., to 330°C @ 6°C/min. (hold 2 min.)
Det.:	Agilent 5973 GC/MS
Transfer line temp.:	280°C
Scan range:	35-550 amu
Solvent delay:	2 min.
Tune:	DFTPP
Ionization:	EI

1. 1,4-dioxane
2. N-nitrosodimethylamine •
3. pyridine •
4. 2-fluorophenol
5. phenol-d6
6. phenol •
7. aniline •
8. bis(2-chloroethyl) ether •
9. 2-chlorophenol •
10. 1,3-dichlorobenzene •
11. 1,4-dichlorobenzene-d4
12. 1,4-dichlorobenzene •
13. benzyl alcohol •
14. 1,2-dichlorobenzene •
15. 2-methylphenol •
16. bis(2-chloroisopropyl) ether •
17a. 4-methylphenol •
17b. 3-methylphenol •
18. N-nitroso-di-*n*-propylamine •
19. hexachloroethane •
20. nitrobenzene-d5
21. nitrobenzene •
22. isophorone •
23. 2-nitrophenol •
24. 2,4-dimethylphenol •
25. benzoic acid
26. bis(2-chloroethoxy)methane •
27. 2,4-dichlorophenol •
28. 1,2,4-trichlorobenzene •
29. naphthalene-d8
30. naphthalene •
31. 4-chloroaniline •
32. hexachlorobutadiene •
33. 4-chloro-3-methylphenol •
34. 2-methylnaphthalene •
35. 1-methylnaphthalene •
36. hexachlorocyclopentadiene •
37. 2,4,6-trichlorophenol •
38. 2,4,5-trichlorophenol •
39. 2-fluorobiphenyl
40. 2-chloronaphthalene •
41. 2-nitroaniline •
42. 1,4-dinitrobenzene •
43. dimethyl phthalate •
44. 1,3-dinitrobenzene •
45. 2,6-dinitrotoluene •
46. acenaphthylene •
47. 1,2-dinitrobenzene •
48. 3-nitroaniline •
49. acenaphthene-d10
50. acenaphthene •
51. 2,4-dinitrophenol •
52. 4-nitrophenol •
53. dibenzofuran •
54. 2,4-dinitrotoluene •
55. 2,3,4,6-tetrachlorophenol •
56. 2,3,5,6-tetrachlorophenol •
57. diethyl phthalate •
58. 4-chlorophenyl phenyl ether •
59. fluorene •
60. 4-nitroanaline •
61. 4,6-dinitro-2-methylphenol •
62. N-nitrosodiphenylamine (as diphenylamine) •
63. 1,2-diphenylhydrazine (as azobenzene) •
64. 2,4,6-tribromophenol
65. 4-bromophenyl phenyl ether •
66. hexachlorobenzene •
67. pentachlorophenol •
68. phenanthrene-d10
69. phenanthrene •
70. anthracene •
71. carbazole •
72. di-*n*-butyl phthalate •
73. fluoranthene •
74. benzidine
75. pyrene-d10
76. pyrene •
77. *p*-terphenyl-d14
78. 3,3'-dimethylbenzidine
79. butyl benzyl phthalate •
80. bis(2-ethylhexyl) adipate •
81. 3,3'-dichlorobenzidine
82. benzo(a)anthracene •
83. chrysene-d12
84. chrysene •
85. bis(2-ethylhexyl) phthalate •
86. di-*n*-octyl phthalate •
87. benzo(b)fluoranthene •
88. benzo(k)fluoranthene •
89. benzo(a)pyrene •
90. perylene-d12 •
91. indeno(1,2,3-cd)pyrene •
92. dibenzo(a,h)anthracene •
93. benzo(ghi)perylene •

• 8270 MegaMix® components

Recommended Columns

Rxi™-5ms Columns (fused silica)

(Crossbond® 5% diphenyl / 95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.18mm	0.18	-60 to 330/350°C	20-Meter	13402
0.18mm	0.30	-60 to 330/350°C	20-Meter	13409
0.25mm	0.25	-60 to 330/350°C	30-Meter	13423
0.25mm	0.40	-60 to 330/350°C	30-Meter	13481

Rtx®-5Sil MS Columns (fused silica)

(Crossbond®, selectivity similar to 5% diphenyl/95% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.18mm	0.18	-60 to 330/350°C	20-Meter	42702
0.18mm	0.36	-60 to 330/350°C	20-Meter	42704
0.25mm	0.25	-60 to 330/350°C	30-Meter	12723
0.25mm	0.50	-60 to 330/350°C	30-Meter	12738

Analytical Reference Materials

SV Internal Standard Mix

acenaphthene-d10 naphthalene-d8
chrysene-d12 perylene-d12
1,4-dichlorobenzene-d4 phenanthrene-d10

2,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31206 (ea.)

4,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31006 (ea.)

B/N Surrogate Mix (4/89 SOW)

2-fluorobiphenyl *p*-terphenyl-d14
nitrobenzene-d5

1,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31024 (ea.)

5,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31062 (ea.)

5,000µg/mL each in methylene chloride, 5mL/ampul
cat. # 31086 (ea.)

5,000µg/mL each in methylene chloride, 10mL/ampul
cat. # 33028 (ea.)

Acid Surrogate Mix (4/89 SOW)

2-fluorophenol 2,4,6-tribromophenol
phenol-d6

2,000µg/mL each in methanol, 1mL/ampul
cat. # 31025 (ea.)

10,000µg/mL each in methanol, 1mL/ampul
cat. # 31063 (ea.)

10,000µg/mL each in methanol, 5mL/ampul
cat. # 31087 (ea.)

10,000µg/mL each in methylene chloride, 10mL/ampul
cat. # 33029 (ea.)

GC/MS Tuning Mixture

benzidine DFTPP
4,4'-DDT pentachlorophenol

1,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31615 (ea.)

605 Benzidines Calibration Mix

benzidine 3,3'-dichlorobenzidine

2,000µg/mL each in methanol, 1mL/ampul
cat. # 31030 (ea.)

2,000µg/mL each in methylene chloride, 1mL/ampul
cat. # 31834 (ea.)

8270 Matrix Spike Mix (76 components)

200µg/mL each in methanol:methylene chloride (80:20), 5mL/ampul
cat. # 31687 (ea.)

8270 MegaMix® (76 components)

1,000µg/mL each in methylene chloride, 1mL/ampul, •
cat. # 31850 (ea.)

• Refer to figure for compound list

Inert Sample Path Increases Accuracy

Injection port liners are designed in many configurations, four of which are commonly used for semivolatiles analysis: the single gooseneck, double gooseneck, cyclo double gooseneck, and the Drilled Uniliner®. While all four liner types are used for 8270 analysis, we recommend the Drilled Uniliner® when using constant flow, and the cyclo double gooseneck with pressure pulse conditions.

Liners shown are for Agilent instruments; liners for other instrument brands also are available. For a complete list of liners and seals refer to our catalog or website.

Gooseneck Splitless (4mm)

4.0 ID 6.5 OD x 78.5 20799 5 pk.

Cyclo Double Gooseneck (4mm)

4.0 ID 6.5 OD x 78.5 20896 5 pk.

Double Gooseneck Splitless (4mm)

4.0 ID 6.5 OD x 78.5 20785 5 pk.

Drilled Uniliner® (hole on bottom)

4.0 ID 6.3 OD x 78.5 20771 5 pk.

The Drilled Uniliner® is the most inert liner because the metal injection port outside the glass liner does not contact the sample path – the sample is virtually "funneled" into the column. Also, when using the Drilled Uniliner® inlet seals do not need to be replaced – a savings in maintenance cost and time. The cyclo double gooseneck liner is recommended with pressure pulse conditions. Its corkscrew type sample path enhances sample vaporization and helps prevent sample contact with metal surfaces below the liner. When using a gooseneck type liner, however, routinely replacing the inlet seal below the liner is critical. Gold plated and Siltek® treated liners and seals both ensure an inert sample path, however, Siltek® treated surfaces are more resistant to abrasion during cleaning.

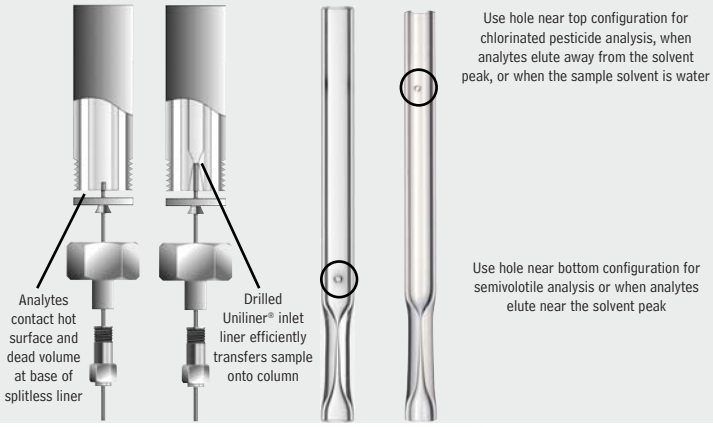
0.8mm ID Dual Vespel® Ring Inlet Seal

Siltek® Treated	21242	2-pk.
Stainless Steel	21238	2-pk.
Gold Plated	21240	2-pk.

restek **innovation**!

The Drilled Uniliner®

The Drilled Uniliner® provides the most inert sample pathway of all inlet liners for splitless injection techniques. This liner connects directly to the column, eliminating contact between the active compounds and active metal surfaces in the injector, and ensuring an inert sample pathway for analyte transfer from the injection port to the column.



Organochlorine Pesticides and PCB

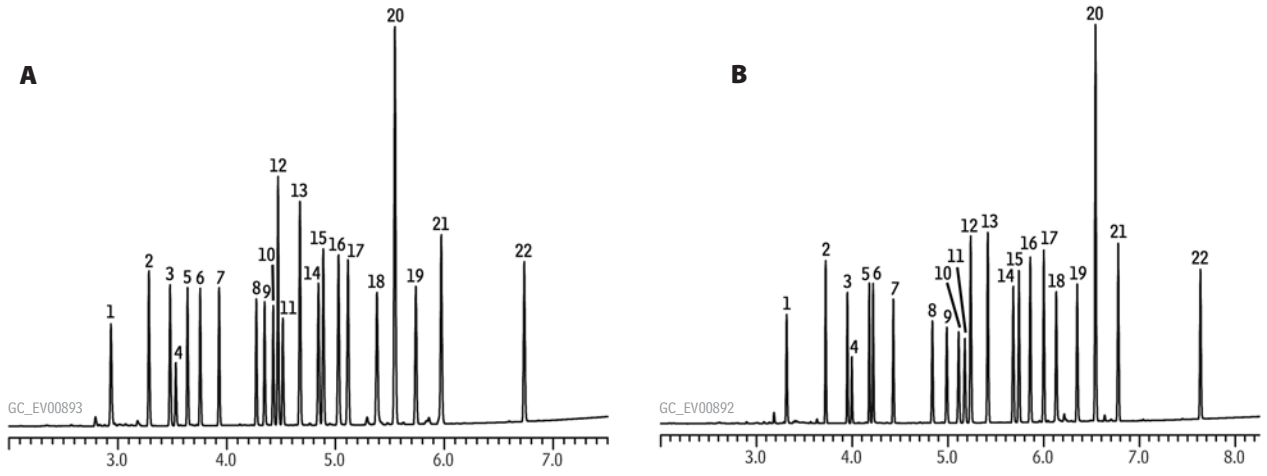
In organochlorine pesticide analysis, careful consideration of the instrument set-up and column choice can greatly improve sample throughput – reducing costs and saving time. The most critical aspects of the inlet system are inertness and efficiency of target analyte transfer to the analytical column. For pesticide and PCB analysis we recommend the Drilled Uniliner® for its unsurpassed inertness (see page 5).

In the analysis shown, 20m x 0.18mm ID Rtx®-CLPesticides and Rtx®-CLPesticides2 primary and confirmation columns were used. We connected a 5m x 0.53mm guard column to the dual analytical columns, using a SeCure™ "Y" connector kit. These columns have been specifically designed to resolve the chlorinated pesticides when used in parallel under the same temperature program and inlet backpressure. As shown in Figure 1, all the organochlorine pesticide compounds are baseline resolved in less than 8 minutes.

Figure 1 Organochlorine pesticides on Rtx®-CLPesticides and Rtx®-CLPesticides2 columns.

5%Column:	**A:** Rtx®-CLPesticides, 20m, 0.18mm ID, 0.18µm (cat.# 42102) and **B:** Rtx®-CLPesticides2, 20m, 0.18mm ID,0.14µm (cat.# 42302) with 5m x 0.53mm ID intermediate-polarity deactivated guard tubing (cat.# 10045), connected using SeCure™ "Y" Connector Kit (cat.# 20276) with Universal "Y" Press-Tight® Connector
Sample:	Organochlorine Pesticide Mix AB #2 (cat.# 32292), 8-80µg/mL each component in hexane/toluene, Pesticide Surrogate Mix (cat.# 32000), 200µg/mL each component in acetone
Inj.:	0.5µL splitless (hold 0.75 min.), 2mm single gooseneck inlet liner (cat.# 20796)
Inj. temp.:	250°C
Carrier gas:	helium, constant flow
Linear velocity:	20cm/sec. @ 140°C
Oven temp.:	140°C (hold 1 min.) to 250°C @ 35°C/min. (hold 1 min.) to 330°C @ 35°C/min. (hold 3 min.)
Det.:	ECD @ 350°C

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



Recommended Columns

Rtx®-CLPesticides Columns (fused silica)

ID	df (µm)	temp. limits	length	cat. #
0.18mm	0.18	-60 to 310/330°C	20-Meter	42102
0.25mm	0.25	-60 to 320/340°C	30-Meter	11123
0.32mm	0.50	-60 to 320/340°C	30-Meter	11139
0.53mm	0.50	-60 to 300/320°C	30-Meter	11140

Rtx®-CLPesticides2 Columns (fused silica)

ID	df (µm)	temp. limits	length	cat. #
0.18mm	0.14	-60 to 310/330°C	20-Meter	42302
0.25mm	0.20	-60 to 320/340°C	30-Meter	11323
0.32mm	0.50	-60 to 320/340°C	30-Meter	11325
0.53mm	0.42	-60 to 300/320°C	30-Meter	11340

did you know?

We can supply all your sample extract clean-up needs.
See our catalog or website for details.

Analytical Reference Materials

Organochlorine Pesticide Mix AB #1

(20 components)

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 each in hexane:toluene (1:1), 1mL/ampul

cat. # 32291 (ea.)

Organochlorine Pesticide Mix AB # 3

(20 components)

same listing as Organochlorine Pesticide Mix AB #1, shown above.

2,000µg/mL each in hexane:toluene (1:1), 1mL/ampul

cat. # 32415 (ea.)

Pesticide Surrogate Mix

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

200µg/mL each in acetone, 1mL/ampul

cat. # 32000 (ea.)

Pesticide Surrogate Mix

decachlorobiphenyl	200µg/mL
2,4,5,6-tetrachloro-*m*-xylene	100

In P&T methanol, 1mL/ampul

cat. # 32453 (ea.)

Organochlorine Pesticide System Evaluation Mix

4,4'-DDT 200µg/mL endrin 100µg/mL

In MTBE, 1mL/ampul

cat. # 32417 (ea.)

508.1 GC Degradation Check Mix

4,4'-DDT endrin

100µg/mL each in ethyl acetate, 1mL/ampul

cat. # 32093 (ea.)

Technical Chlordane, Toxaphene Solutions

Compound	cat.# (ea.)
1,000µg/mL in hexane, 1mL/ampul	
chlordane (technical)	32021
toxaphene	32005
2,000µg/mL in methanol, 1mL/ampul	
chlordane (technical)	32016
toxaphene	32015
5,000µg/mL in isooctane, 1mL/ampul	
chlordane (technical)	32072
toxaphene	32071

also **available**

Aroclor® Solutions!

Visit our website, see our newest catalog, or call your Restek representative for details.

Increase Sample Throughput Using Dual Analytical Columns and a "Y" Connector Union

Most laboratories need to confirm the compound identification obtained on one column with a second column of different selectivity. This is best achieved by making a single injection onto a guard column which is connected to two analytical columns, using a "Y" splitter. This allows data to be collected from both columns simultaneously, allowing samples to be processed without waiting for the confirmation result.

Rtx®-CLPesticides Column Kits

0.25mm ID Rtx®-CLPesticides Kit cat.# 11199 (kit), Includes:	cat.#
30m, 0.25mm ID, 0.25µm Rtx®-CLPesticides Column	11123
30m, 0.25mm ID, 0.20µm Rtx®-CLPesticides2 Column	11323
Universal Angled "Y" Press-Tight® Connector	20403
5m, 0.25mm ID Siltek® Guard Column	10026

0.32mm ID Rtx®-CLPesticides Kit cat.# 11198 (kit), Includes:	cat.#
30m, 0.32mm ID, 0.50µm Rtx®-CLPesticides Column	11139
30m, 0.32mm ID, 0.25µm Rtx®-CLPesticides2 Column	11324
Universal Angled "Y" Press-Tight® Connector	20403
5m, 0.32mm ID Siltek® Guard Column	10027

0.53mm ID Rtx®-CLPesticides Kit cat.# 11197 (kit), Includes:	cat.#
30m, 0.53mm ID, 0.50µm Rtx®-CLPesticides Column	11140
30m, 0.53mm ID, 0.42µm Rtx®-CLPesticides2 Column	11340
Universal Angled "Y" Press-Tight® Connector	20403
5m, 0.53mm ID IP Deactivated Guard Column	10045

"Y" connectors

"Y" connectors are available in both metal and glass. Glass connectors offer the best chromatography, but are prone to leaks. To eliminate leaks we developed the SeCure™ "Y" connector, which takes advantage of our Press-Tight® connector and adds mechanical strength to hold the columns in place. A second connector, the MXT™ "Y"-Union, is available for fused silica columns.

SeCure™ "Y" - The most secure connector available!

Kits include: SeCure™ "Y" connector body, 3 knurled nuts, "Y" Universal Press-Tight® union, 3 ferrules.

Ferrules Fit Column ID	qty.	cat.#
0.25/0.28mm	kit	20276
0.32mm	kit	20277
0.45/0.53mm	kit	20278

MXT™ "Y"-Union Connector Kits for Fused Silica Columns

Each kit contains the MXT™ union, three 1/32-inch 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

Volatile Organic Compounds in Air

One of the most widely used VOC methods for ambient air monitoring specifies sample collection with a specially prepared stainless steel canister, followed by GC/MS analysis. Restek can support all facets of your air monitoring program – from state-of-the-art sampling equipment to high quality analytical reference standards.

An inert canister surface is critical to obtaining accurate sample results. Restek offers a complete line of TO-Cans™ (Summa® canisters) which are electropolished and extensively cleaned prior to shipping to ensure a high-quality passivated surface for improved analyte stability. No weld marks on the spheres further reduce the occurrence of active sites. For reactive compounds, such as sulfur-containing components, a SilcoCan™ is your best canister choice. SilcoCan™ canisters are deactivated with Siltek® surface treatment resulting in exceptional inertness and maximum sample stability, even for low level sulfur compounds.

Optional gauge

- Quickly confirm vacuum or pressure inside canister.
- Monitor pressure changes.
- Fully protected by canister frame.
- Can be heated to 90°C during cleaning.

Siltek® tee

High-quality vacuum gauge

Enhanced valve and canister bracket
Canister holder and valve bracket protect canister, tube stub, and valve.

2-3 Port high quality valve
Metal-to-metal seal, 2/3 turn with stainless steel diaphragm.

We consider your TO-Cans™ and SilcoCans™ to be an investment and offer check-ups and reconditioning when needed.

Serial-controlled label
For quick, sure identification.

TO-Can™ Air Monitoring Canisters

Optimized for US EPA Methods TO-14 and TO-15, and ASTM D5466

Description	qty.	cat.#
6L Volume*		
TO-Can™ Canister, 1/4" Valve	ea.	24174
TO-Can™ Canister with Gauge, 1/4" Valve	ea.	24178
TO-Can™ Canister with No Valve	ea.	22096

SilcoCan™ Air Monitoring Canisters

Ideal for low-level reactive sulfur (1-20ppb), TO-14, or TO-15 compounds

Description	qty.	cat.#
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
SilcoCan™ Canister with No Valve	ea.	22092
Replacement 1/4" Valves for Air Monitoring Canisters		
1/4" Replacement Valve (2-port)	ea.	24145
1/4" Siltek® Replacement Valve (2-port)	ea.	24144
1/4" Replacement Valve (3-port)	ea.	24147
1/4" Siltek® Replacement Valve (3-port)	ea.	24146

Restek canisters are originally equipped with high-quality Parker Hannifin diaphragm valves. Each valve is helium leak-tested to 4 x 10^{-9}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.

All configurations also available in 1L, 3L, and 15L volumes.

Recommended Columns

Rxi™-1ms Columns (fused silica)

(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.32mm	1.00	-60 to 330/350°C	60-Meter	13357

Analytical Reference Materials

TO-15 62 Component Mix (62 components)

Cylinder Construction:	aluminum
Cylinder Size:	8 x 24 cm.
Volume/Pressure:	104 liters of gas @ 1800psig
Cylinder Fitting:	CGA-180 outlet
Weight:	1.5 lbs./0.7 kg

acetone
benzene
benzyl chloride*
bromodichloromethane
bromoform
bromomethane
1,3-butadiene
2-butanone (MEK)
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-dichlorotetrafluoroethane (Freon® 114)
heptane
hexachloro-1,3-butadiene
hexane
2-hexanone (MBK)
4-methyl-2-pentanone (MIBK)
methylene chloride
methyl *tert*-butyl ether (MTBE)
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

In nitrogen, 104 liters @ 1800psig

1ppm cat. # 34436 (ea.)

100ppb cat. # 34437 (ea.)

*Stability of this compound cannot be guaranteed.

TO-14A Internal Standard/Tuning Mix

Cylinder Construction:	aluminum
Cylinder Size:	8 x 24 cm.
Volume/Pressure:	104 liters of gas @ 1800psig
Cylinder Fitting:	CGA-180 outlet
Weight:	1.5 lbs./0.7 kg

bromochloromethane
1-bromo-4-fluorobenzene (4-bromofluorobenzene)
chlorobenzene-d5
1,4-difluorobenzene

In nitrogen, 104 liters @ 1800psig

1ppm cat. # 34408 (ea.)

100ppb cat. # 34425 (ea.)

Simplify Sampling, Increase Accuracy & Efficiency

Air Canister Heating Jacket

Our heating jackets can help you prepare your canisters for sampling faster and more efficiently. The jacket's novel design ensures complete cleaning by heating the canister and valve together and prevents condensation, ensuring more accurate results. Two temperature settings, 75ºC and 150ºC. Fits all canisters up to 6L in size.

Description	qty.	cat.#
Air Canister Heating Jacket	ea.	24123

*Not CE certified.

The ultimate in controlled heating, for reliably cleaning your air canisters!

Passive Air Sampling Kits

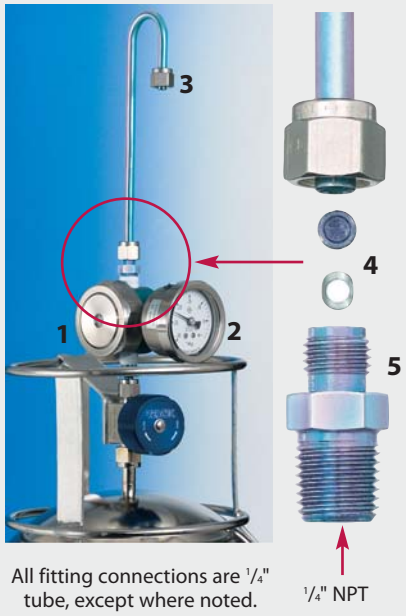
Our passive sampling kits include all hardware required for field sampling (except the canister) and assemble easily. Our kit was designed to reduce the number of potential leak sites and is available in seven flow ranges, and in stainless steel or with Siltek® surface treatment. Individual parts are also available.

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 vacuum change during sampling.

3. ¼-inch Siltek® sample inlet
The 0.3m x ¼-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.

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.



All fitting connections are ¼" tube, except where noted.

¼" NPT

See our catalog for other canister volumes and sampling times.

5. Interchangeable critical orifice
An interchangeable ruby critical orifice allows you to control the flow with very high precision. To select the correct critical orifice for your sample, see table below. Available in stainless steel, or Siltek® treated for optimum inertness.

Sampling Time 6 Liter	Flow (sccm)	Orifice size	Siltek® Treated Sampling Kits*	Stainless Steel Sampling Kits*
125 hour	0.5–2	0.0008"	24217	24216
24 hour	2–4	0.0012"	24160	24165
12 hour	4–8	0.0016"	24161	24166
8 hour	8–20	0.0020"	24162	24167
3 hour	20–40	0.0030"	24163	24168
1.5 hour	40–80	0.0060"	24164	24169
0.5 hour	80–350	0.0090"	22101	22100

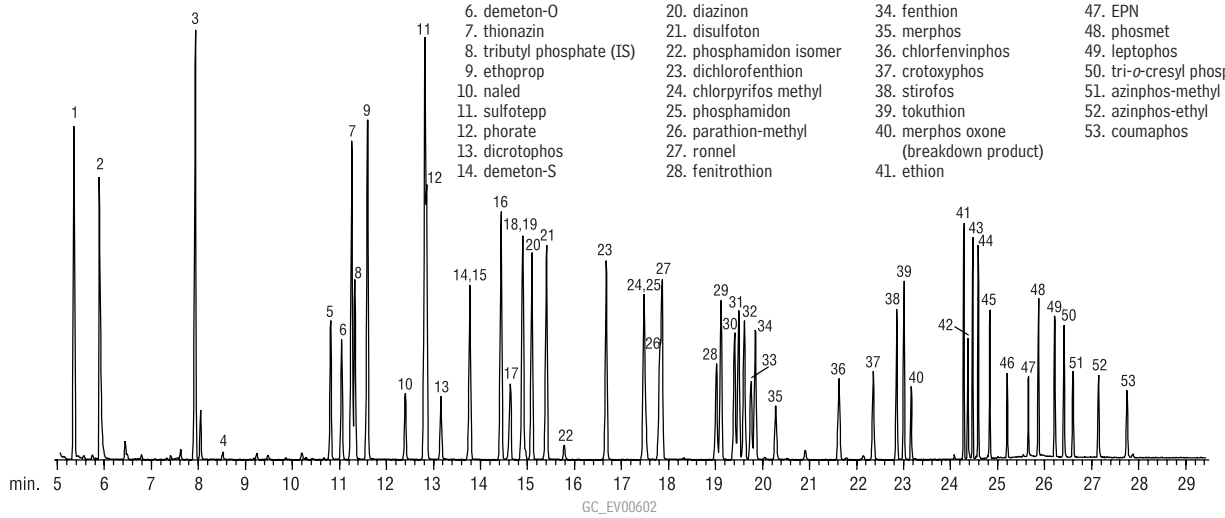
*Air sampling canisters sold separately. Available in 400cc, 1L, 3L, 6L, and 15L volumes.

Organophosphorus Pesticides

Organophosphorus pesticides (OPPs) are commonly used as insecticides, fungicides, and herbicides. Due to their widespread use however, they have become an environmental concern. We reccommend the Rtx®-OPPesticides2 column for separating organophosphorus pesticides (OPP). Separation is improved, and analysis time is significantly reduced, compared to other columns. The extended upper temperature limit of this phase (330°C) allows analysts to bake out high molecular weight contamination typically associated with pesticide samples. The low bleed column is a perfect match for sensitive detection systems.

Figure 1 Organophosphorus pesticides on an Rtx®-OPPesticides2 column.

1. dichlorvos
2. hexamethylphosphoramide
3. mevinphos
4. trichlorfon
5. TEPP
6. demeton-O
7. thionazin
8. tributyl phosphate (IS)
9. ethoprop
10. naled
11. sulfotepp
12. phorate
13. dicrotophos
14. demeton-S
15. monocrotophos
16. terbufos
17. dimethoate
18. dioxathion
19. fonophos
20. diazinon
21. disulfoton
22. phosphamidon isomer
23. dichlorofenthion
24. chlorpyrifos methyl
25. phosphamidon
26. parathion-methyl
27. ronnel
28. fenitrothion
29. aspon
30. malathion
31. chlorpyrifos
32. trichloronate
33. parathion-ethyl
34. fenthion
35. merphos
36. chlorfenvinphos
37. crotoxyphos
38. stirofos
39. tokuthion
40. merphos oxone (breakdown product)
41. ethion
42. fensulfothion
43. bolstar
44. carbophenothion
45. famphur
46. triphenyl phosphate (SS)
47. EPN
48. phosmet
49. leptophos
50. tri-*o*-cresyl phosphate
51. azinphos-methyl
52. azinphos-ethyl
53. coumaphos



Column:	Rtx®-OPPesticides2, 30m, 0.25mm ID, 0.25µm (cat.# 11243)	Inj. temp.:	250°C
Sample:	US EPA Method 8141A Custom Standard Mix 1µL 100ppm (100ng on column)	Carrier gas:	helium, constant flow
	Triphenylphosphate Standard (cat.# 32281)	Flow rate:	1.0mL/min.
	Tributylphosphate Standard (cat.# 32280)	Oven temp.:	80°C (hold 0.5 min.) to 140°C @ 20°C/min. to 210°C @ 4°C/min. (hold 1 min.) to 280°C @ 30°C (hold 5 min.)
	8140/8141 OP Pesticides Calibration Mix A (cat.# 32277)		
	8141 OP Pesticides Calibration Mix B (cat.# 32278)	Det:	MS
	Custom Mixes: Call Restek for Information	Transfer line temp.:	280°C
Inj.:	1.0µL splitless (hold 0.4 min.), 4mm double gooseneck inlet liner (cat.# 20785)	Scan range:	35-400amu
		Ionization:	EI

Recommended Columns

Rtx®-OPPesticides2 Columns (fused silica)

ID	df (µm)	temp. limits	length	cat. #
0.18mm	0.20	-20 to 310/330°C	20-Meter	11244
0.25mm	0.25	-20 to 310/330°C	30-Meter	11243
0.32mm	0.32	-20 to 310/330°C	30-Meter	11241
0.53mm	0.50	-20 to 310/330°C	30-Meter	11242

Sample Preparation

CarboPrep™ Cartridges

SPE Cartridge	Tube Volume, Bed Weight	qty.	cat#
CarboPrep™ 90	3mL, 250mg	50-pk.	26091
CarboPrep™ 90	6mL, 500mg	30-pk.	26092

Excellent for Pesticide Residue Cleanup!

did you know?

We can supply all your organophosphate standards—
See our catalog or website for details.

Carbamates by HPLC

Carbamates are widely used insecticides that pose a health risk as endocrine disruptors. Our Ultra Carbamate column, in a 50mm length, separates common carbamates in less than 10 minutes (Figure 1), significantly less than the time required by traditional C18 columns. In addition to the best column choice for the analysis, we offer reference mixes for Method 531 carbamates, a performance check mix, and the specified internal standard, 4-bromo-3,5-dimethylphenyl-N-methylcarbamate (BDMC).

Figure 1 Carbamate pesticides on an Ultra Carbamate column.

Peak List:
1. aldicarb sulfone
2. aldicarb sulfoxide
3. oxamyl
4. methomyl
5. 3-hydroxycarbofuran
6. aldicarb
7. propoxur
8. carbofuran
9. carbaryl
10. methiocarb
11. 4-bromo-3,5-dimethylcarbamate

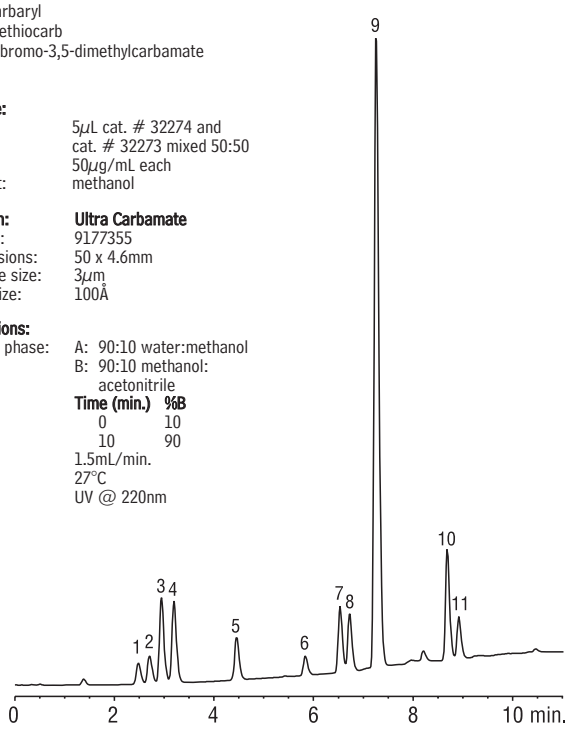
Sample:
Inj.: 5µL cat. # 32274 and cat. # 32273 mixed 50:50
Conc.: 50µg/mL each
Solvent: methanol

Column: **Ultra Carbamate**
Cat. #: 9177355
Dimensions: 50 x 4.6mm
Particle size: 3µm
Pore size: 100Å

Conditions:
Mobile phase: A: 90:10 water:methanol
B: 90:10 methanol: acetonitrile

Time (min.)	**%B**
0	10
10	90

Flow: 1.5mL/min.
Temp.: 27°C
Det.: UV @ 220nm



LC_0225

free literature

Simple, Sensitive HPLC/UV Analysis for Paraquat and Diquat

These highly charged quaternary amines are poorly retained on alkyl stationary phases. Using only acetonitrile, water, and a solvation-blocking reagent, our separation system alters the interactions among analyte, mobile phase, and stationary phase, and promotes solubility of the analytes in the stationary phase. In our system, the detection limit is 6ppb for either herbicide, and the analysis is completed in less than 10 minutes. An optimized solid phase extraction cartridge concentrates the herbicides for the analysis.

Lit. cat. # 580006

Recommended Columns

Ultra Carbamate Columns

Physical Characteristics:

particle size: 3µm or 5µm, spherical
pore size: 100Å
pH range: 2.5 to 7.5
temperature limit: 80°C

3µm Column	**cat. #**
50mm (2.1mm ID)	9177352
50mm (4.6mm ID)	9177355
5µm Column	**cat. #**
250mm (4.6mm ID)	9177575

*For post-column derivatization / fluorescence detection applications for a 4.6mm ID column the total system dead volume, including the post-column reactor, must be less than 650µL. For standard post-column reactor systems, we recommend a 250 x 4.6mm, 5µm column. Call Restek technical service for more information.

ordering note
For guard cartridges for these columns, visit our website at **www.restek.com**.

Analytical Reference Materials

531.1 Carbamate Pesticide Calibration Mixture

(10 components)

aldicarb
aldicarb sulfone
aldicarb sulfoxide
carbaryl (Sevin®)
carbofuran
3-hydroxycarbofuran
methiocarb
methomyl
oxamyl
propoxur (Baygon®)

100µg/mL each in methanol, 1mL/ampul
cat. # 32273 (ea.)

531.2 Carbamate Pesticide Calibration Mixture

(11 components)

aldicarb
aldicarb sulfone
aldicarb sulfoxide
carbaryl (Sevin®)
carbofuran
3-hydroxycarbofuran
methiocarb
methomyl
1-naphthol
oxamyl
propoxur (Baygon®)

100µg/mL in acetonitrile, 1mL/ampul
cat. # 32435 (ea.)

Internal Standard

4-bromo-3,5-dimethylphenyl-N-methylcarbamate (BDMC)
100µg/mL in methanol, 1mL/ampul
cat. # 32274 (ea.)

531.1 Performance Check Mix

aldicarb sulfoxide	100µg/mL	3-hydroxycarbofuran	2
BDMC	10	methiocarb	20

In methanol, 1mL/ampul
cat. # 32275 (ea.)

Polyaromatic Hydrocarbons (PAHs) by HPLC

Most HPLC PAH methods recommend using a C18 column with fluorescence and/or UV/VIS detection. Our Pinnacle™ II PAH columns have a highly reproducible modified alkyl phase on Restek manufactured silica, specifically developed for this application. Figure 1 shows the analysis of 16 target PAHs in less than 18 minutes, and Figure 2 shows a separation of 20 target PAHs and related compounds, in less than 6 minutes, using a 5cm column.

Figure 1 Baseline separation of 16 PAHs in less than 18 minutes on a Pinnacle™ II PAH column.

Sample:
Inj.: 5µL
Solvent: 1:9 methylene chloride:acetonitrile

Column: **Pinnacle™ II PAH** (Cat. # 9219563)
150mm x 3.2mm ID

Conditions:
Mobile phase: A: water, B: acetonitrile

Time (min.)	%B
0	40
7	60
11	100
17.9	100
18	40

Flow: 1.2 mL/min.
Temp.: ambient
Det.: UV @ 254nm

Peak List:	Conc. (µg/mL)
1. naphthalene	100
2. acenaphthylene	100
3. acenaphthene	100
4. fluorene	100
5. phenanthrene	50
6. anthracene	100
7. fluoranthene	50
8. pyrene	50
9. benzo(a)anthracene	50
10. chrysene	50
11. benzo(b)fluoranthene	50
12. benzo(k)fluoranthene	50
13. benzo(a)pyrene	50
14. dibenzo(a,h)anthracene	50
15. benzo(ghi)perylene	50
16. indeno(1,2,3-cd)pyrene	50

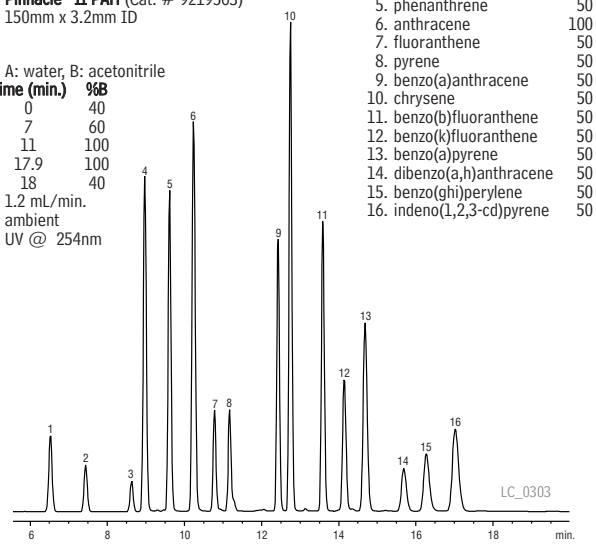


Figure 2 Fast, efficient separation of 20 target PAHs and related compounds using a 5cm Pinnacle™ II PAH column.

Sample:
Inj.: 5µL
Conc.: 5-10ppm each
Sample diluent: methylene chloride:acetonitrile (1:9 v/v)

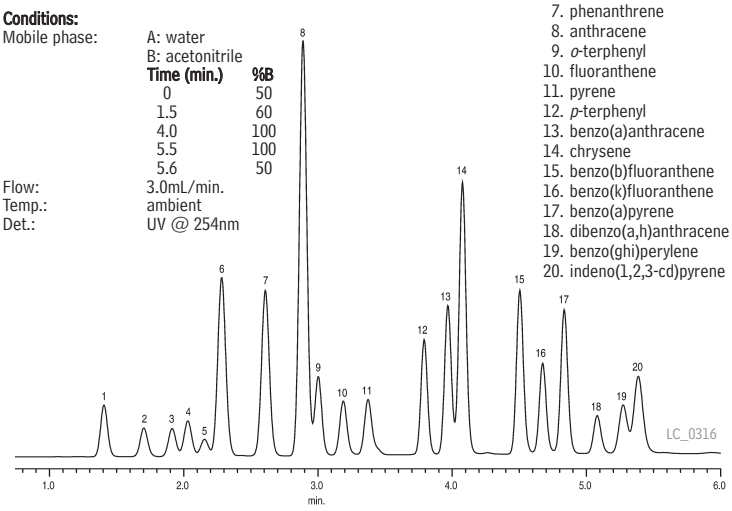
Column: **Pinnacle™ II PAH** (Cat. # 9219555)
50mm x 4.6mm ID

Conditions:
Mobile phase: A: water
B: acetonitrile

Time (min.)	%B
0	50
1.5	60
4.0	100
5.5	100
5.6	50

Flow: 3.0mL/min.
Temp.: ambient
Det.: UV @ 254nm

Peak List:
1. naphthalene
2. acenaphthylene
3. 1-methylnaphthalene
4. 2-methylnaphthalene
5. acenaphthene
6. fluorene
7. phenanthrene
8. anthracene
9. *o*-terphenyl
10. fluoranthene
11. pyrene
12. *p*-terphenyl
13. benzo(a)anthracene
14. chrysene
15. benzo(b)fluoranthene
16. benzo(k)fluoranthene
17. benzo(a)pyrene
18. dibenzo(a,h)anthracene
19. benzo(ghi)perylene
20. indeno(1,2,3-cd)pyrene



Recommended Columns

Pinnacle™ II PAH Columns

Physical Characteristics:

particle size: 5µm, spherical
pore size: 110Å
endcap: fully endcapped
pH range: 2.5 to 10
temperature limit: 80°C

5µm Column	cat. #
50 x 2.1mm	9219552
50 x 3.2mm	9219553
150 x 3.2mm	9219563
50 x 4.6mm	9219555
150 x 4.6mm	9219565
10 x 2.1mm	921950212
10 x 4.0mm	921950210
20 x 2.1mm	921950222
20 x 4.0mm	921950220

ordering note
For guard cartridges for these columns, visit our website at **www.restek.com**.

Analytical Reference Materials

EPA Method 8310 PAH Mixture (18 components)

acenaphthene
acenaphthylene
anthracene
benzo(a)anthracene
benzo(a)pyrene
benzo(b)fluoranthene
benzo(ghi)perylene
benzo(k)fluoranthene
chrysene
dibenzo(a,h)anthracene
fluoranthene
fluorene
indeno(1,2,3-cd)pyrene
1-methylnaphthalene
2-methylnaphthalene
naphthalene
phenanthrene
pyrene

500µg/mL each in acetonitrile, 1mL/ampul
cat. # 31841 (ea.)

EPA Method 8310 Quality Control Check

(18 components)

acenaphthene	100µg/mL	dibenzo(a,h)anthracene	10
acenaphthylene	100	fluoranthene	10
anthracene	100	fluorene	100
benzo(a)anthracene	10	indeno(1,2,3-cd)pyrene	10
benzo(a)pyrene	10	1-methylnaphthalene	100
benzo(b)fluoranthene	10	2-methylnaphthalene	100
benzo(ghi)perylene	10	naphthalene	100
benzo(k)fluoranthene	5	phenanthrene	100
chrysene	10	pyrene	10

In acetonitrile, 1mL/ampul
cat. # 31843 (ea.)

EPA Method 8310 Surrogate Standard

decafluorobiphenyl
1,000µg/mL in acetonitrile, 1mL/ampul
cat. # 31842 (ea.)

free literature

Environmental Flyer
HPLC analyses described in this 8-page publication include carbamates, carbonyls, explosives, paraquat/diquat, phenoxyacid herbicides, and polyaromatic hydrocarbons.

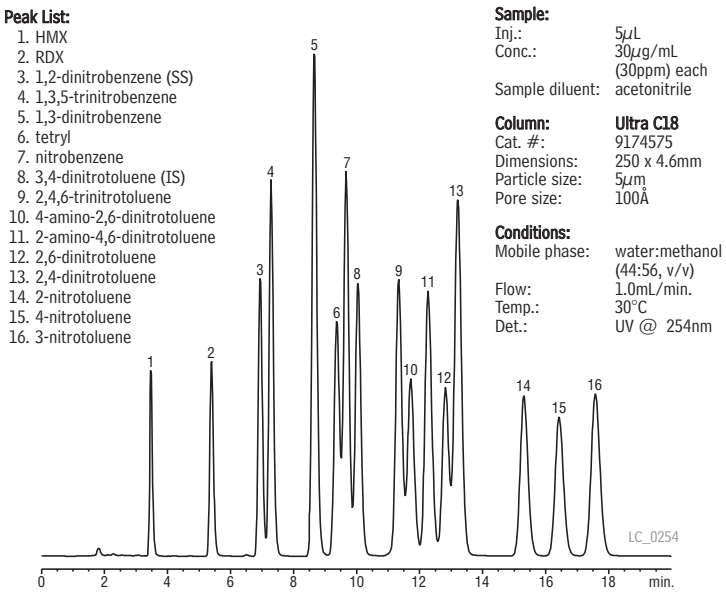
Lit. cat. # 59741A

Explosives by HPLC

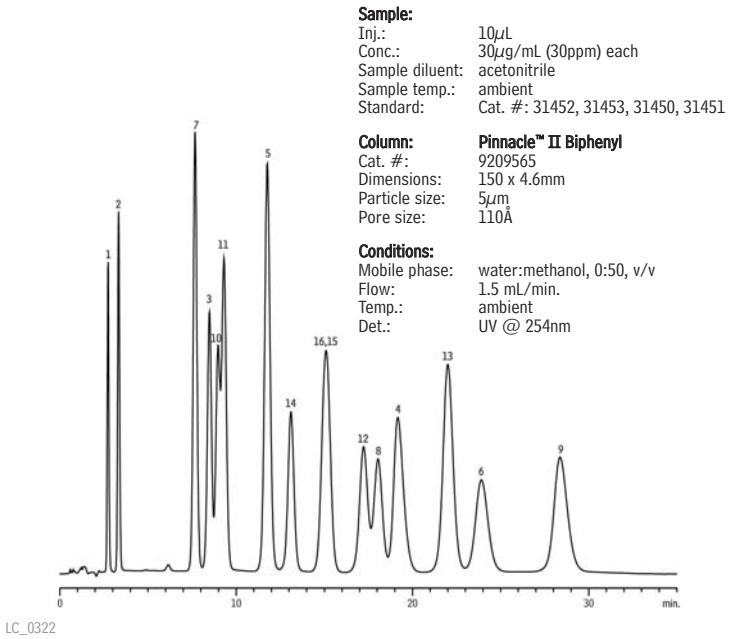
Common methods for quantifying explosives call for reversed phase HPLC with UV detection, using a primary column and a confirmation column. While cyano phases typically have been used for the confirmation column, resolution of the target explosive compounds is poor. The Pinnacle™ II Biphenyl column provides excellent resolution of Method 8330 explosives, as shown in Figure 1, and selectivity is markedly different from C18 phases, making the Pinnacle™ II Biphenyl column an ideal confirmation column. If a cyano phase must be used for confirmation, we recommend a Pinnacle™ II Cyano column.

Figure 1 An outstanding column pair for explosives analysis.

For superior performance, use an Ultra C18 primary column...



...coupled with a Pinnacle II™ Biphenyl column.



LC_0322

Recommended Columns

Ultra C18 Columns (USP L1)

Physical Characteristics:

particle size: 3µm or 5µm, spherical
pore size: 100Å
carbon load: 20%
endcap: fully endcapped
pH range: 2.5 to 7.5
temperature limit: 80°C

5µm Column, 4.6mm	cat. #
150mm	9174565
250mm	9174575

Pinnacle™ II Biphenyl Columns (USP L11)

Physical Characteristics:

particle size: 5µm, spherical
pore size: 110Å
endcap: yes
pH range: 2.5 to 7.5
temperature limit: 80°C

5µm Column, 4.6mm	cat. #
150mm	9209565
250mm	9209575

ordering note
For guard cartridges for these columns, visit our website at **www.restek.com**.

Analytical Reference Materials

Nitroaromatics and Nitramine Explosives by HPLC (14 components)

1,3-dinitrobenzene
2-amino-4,6-dinitrotoluene
4-amino-2,6-dinitrotoluene
2,4-dinitrotoluene
2,6-dinitrotoluene
HMX
nitrobenzene
2-nitrotoluene
3-nitrotoluene
4-nitrotoluene
RDX
tetryl
1,3,5-trinitrobenzene
2,4,6-trinitrotoluene

1,000µg/mL each in acetonitrile, 1mL/ampul
cat. # 33905 (ea.)

8095 Internal Standard

3,4-dinitrotoluene
1,000µg/mL in methanol, 1mL/ampul
cat. # 31452 (ea.)

8330 Surrogate

1,2-dinitrobenzene
1,000µg/mL in methanol, 1mL/ampul
cat. # 31453 (ea.)

free literature

HPLC Analysis of Trace-Level Explosives Using Pinnacle II™ C18 and Cyano Columns

Pinnacle II™ C18 reversed phase columns and Pinnacle II™ Cyano normal phase columns are effective primary and confirmation columns for analyzing explosives according to US EPA Method 8330A. Analytical conditions and example chromatograms are presented in this 2-page note. Per recommendation in the method, the same mobile phase is used for the primary and confirmation analysis.

Lit. cat. # 59361

Environmental Essentials

Direct Injection Liners - What's a drilled uniliner? See page 5.

COLUMN INSTALLS THIS END

Description	ID*/OD & Length (mm)	cat.# ea.	5-pk.
DI Liners for Agilent 5890 & 6890 GCs (For 0.25/0.32/0.53mm ID Columns)			
Drilled Uniliner®▲	4.0 ID 6.3 OD x 78.5	21054	21055
Siltek® Drilled Uniliner®▲	4.0 ID 6.3 OD x 78.5	21054-214.1	21055-214.5
Drilled Uniliner®▼	4.0 ID 6.3 OD x 78.5	20756	20771
Double Gooseneck Drilled Uniliner®▲	4.0 ID 6.3 OD x 78.5	20508	20509
Double Gooseneck Drilled Uniliner®▼	4.0 ID 6.3 OD x 78.5	20954	20989
Siltek® 1mm Drilled Uniliner®▲	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)			
Drilled Uniliner®▲	4.0 ID 6.3 OD x 78.5	21470	21471
Drilled Uniliner®▼	4.0 ID 6.3 OD x 78.5	21468	21469
DI Liners for Shimadzu 17A, 2010, and 2014 GCs (For 0.32/0.53mm ID Columns)			
Open-top Drilled Uniliner®▲	3.5 ID 5.0 OD x 95	21285	21286
Open-top Drilled Uniliner®▼	3.5 ID 5.0 OD x 95	21287	21288
Gooseneck Drilled Uniliner®▲	3.5 ID 5.0 OD x 95	21289	21290
Gooseneck Drilled Uniliner®▼	3.5 ID 5.0 OD x 95	21291	21292
DI Liners for PerkinElmer GCs (For 0.32/0.53mm ID Columns)			
Auto SYS™ Drilled Uniliner®▲	4.0 ID 6.2 OD x 92.1	20819	20822
Auto SYS™ Drilled Uniliner®▼	4.0 ID 6.2 OD x 92.1	21293	21294
Auto SYS™ Gooseneck Drilled Uniliner®▲	4.0 ID 6.2 OD x 92.1	21295	21296
Auto SYS™ Gooseneck Drilled Uniliner®▼	4.0 ID 6.2 OD x 92.1	21297	21298
Direct Injection Liners for Thermo Electron TRACE™ & Focus SSL (0.32 & 0.53mm ID columns)			
Drilled Uniliner®▲	5.0 ID 8.0 OD x 105	22411	22412
Drilled Uniliner®▼	5.0 ID 8.0 OD x 105	22413	22414

*Nominal ID at syringe needle expulsion point. ▲Hole on top. ▼Hole on bottom.

O-Rings

	Max. temp.	qty.	cat.#
A) Viton® O-Rings for Agilent GCs			
	250°C	25-pk.	20377
B) Graphite O-rings for Agilent & Varian split liners (6.35mm ID)			
	450°C	10-pk.	20296
B) Graphite O-rings for Agilent & Varian splitless liners (6.5mm ID)			
	450°C	10-pk.	20298
C) 5mm Graphite Liner Seals for Varian 1078/1079 GCs			
	450°C	10-pk.	22683
D) Viton® O-Rings for Shimadzu 17A, 2010, and 2014 GCs			
	250°C	10-pk.	21477
E) Graphite O-Rings for Shimadzu 17A, 2010, & 2014 Split Liners			
	450°C	5-pk.	20243
E) Graphite O-Rings for Shimadzu 17A, 2010, & 2014 Splitless Liners			
	450°C	5-pk.	20244
F) Silicone O-Rings for PerkinElmer Auto SYS™ GCs			
	250°C	10-pk.	20262
G) Viton® O-Rings for PerkinElmer PSS			
	250°C	10-pk.	20366
H) Inlet Liner Seals for TRACE™ PTV			
	450°C	2-pk.	21392
I) Graphite Sealing Ring for TRACE™ and Focus SSL Instruments			
	450°C	ea.	21898
I) Graphite Sealing Rings for TRACE™ and Focus SSL Instruments			
	450°C	2-pk.	21899

did you know?

We can supply all your sample extract clean-up needs.
See our catalog or website for details.

Restek Septa

- Precision molding assures consistent, accurate fit.
- Ready to use.
- Do not adhere to hot metal surfaces.
- Packaged in non-contaminating glass jars.

Septum Diameter	25-pk.	50-pk.	100-pk.
Thermolite® Septa			
5mm (3/16")	27120	27121	27122
6mm (1/4")	27123	27124	27125
7mm	27126	27127	27128
8mm	27129	27130	27131
9mm	27132	27133	27134
9.5mm (3/8")	27135	27136	27137
10mm	27138	27139	27140
11mm (7/16")	27141	27142	27143
11.5mm	27144	27145	27146
12.5mm (1/2")	27147	27148	27149
17mm	27150	27151	27152
Shimadzu Plug	27153	27154	27155
IceBlue™ Septa			
9mm		27156	27157
9.5mm (3/8")		27158	27159
10mm		27160	27161
11mm (7/16")		27162	27163
11.5mm		27164	27165
12.5mm (1/2")		27166	27167
17mm		27168	27169
Shimadzu Plug		27170	27171
BTO® Septa			
5mm CenterGuide™		27100	27101
6mm (1/4")		27102	27103
9mm CenterGuide™		27104	27105
9.5mm (3/8")		27106	27107
10mm		27108	27109
11mm (7/16") CenterGuide™		27110	27111
11.5mm CenterGuide™		27112	27113
12.5mm (1/2") CenterGuide™		27114	27115
17mm CenterGuide™		27116	27117
Shimadzu Plug		27118	27119

Thermolite® Septa

- Usable to 340°C inlet temperature.
- Excellent puncturability.

IceBlue™ Septa

- Usable to 250°C inlet temperature.
- General-purpose septa.
- Excellent puncturability.
- Ideal for SPME.

BTO® Septa

- CenterGuide™ design—requires less force for initial penetration.
- Usable to 400°C inlet temperature.
- Each batch GC-FID tested.
- Bleed and temperature optimized; ideal for demanding GC and GC/MS applications.

Dual Vespel® Ring Inlet Seals - Eliminates the need for a washer!

0.8mm ID Dual Vespel® Ring Inlet Seal	2-pk.	10-pk.
Siltek® Treated	21242	21243
Gold-Plated	21240	21241
Stainless Steel	21238	21239
1.2mm ID Dual Vespel® Ring Inlet Seal	**2-pk.**	**10-pk.**
Siltek® Treated	21248	21249
Gold-Plated	21246	21247
Stainless Steel	21244	21245

Replacement Inlet Seals with 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® Treated 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.

HANDY
septum size chart

Instrument	Septum Diameter (mm)
Agilent (HP)	
5880A, 5890, 6890, 6850, PTV	11
5700, 5880	9.5/10
On-Column Injection	5
Thermo Electron	
TRACE™ GC	17
GCQ w/TRACE™, PTV	17
8000 series	17
Finnigan (TMQ)	
GC 9001	9.5
GCQ	9.5
QCQ™	9.5
TRACE™ 2000	9.5
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

septum handling tips

- Handle septa carefully, to prevent contamination.
- Minimize bleed—use preconditioned, low-bleed septa.
- Follow septum and instrument manufacturers' recommendations.

RESTEK

Restek Trademarks:
CarboPrep, Crossbond, IceBlue, MegaMix, MXT, Pinnacle, Press-Tight, Rtx, Rxi, SeCure, SilcoCan, Silcosteel, Siltek, Sulfinert, Thermolite, TO-Can, Uniliner.

Other Trademarks:
BTO (Chromatography Research Supplies, Inc.), Freon, Vespel, Viton (E.I. du Pont de Nemours & Co., Inc.), QCQ (Finnigan Corp.), SUMMA (Moletrics), Auto SYS (Perkin-Elmer), Baygon (S.C. Johnson & Son, Inc.), TRACE (Thermo Scientific), Sevin (Union Carbide Corp.), Veriflo (Veriflo Corp.)

Lit. Cat.# 580127

Biodiesel Solutions

Innovative Products for Simple, Reliable Biodiesel Analysis

- MXT®, Rtx®, and Stabilwax® biodiesel columns—engineered specifically for high performance biodiesel analysis.
- GC accessories to simplify your lab work and increase productivity.
- Analytical reference materials—high quality standards for reliable results.

Integrated retention gaps—

The Ultimate Biodiesel Solution!

See page 5 for details

Introduction to Biodiesel

Today, as oil prices climb and pollution levels soar, there is significant worldwide interest in alternative fuels. Biodiesel is one of the most popular alternative fuels available today. It may be used in engines, either pure or blended with diesel fuel, to reduce exhaust pollutants. It can be produced easily from sunflowers, soy, rapeseed, tallow, lard, yellow grease, and other sources. Chemically, it is the product obtained when a vegetable oil or animal fat is reacted with an alcohol in the presence of a catalyst, such as sodium or potassium hydroxide, to produce fatty acid methyl esters.

Methods used to test the quality of biodiesel fuels can be categorized into three types based on the target compounds: ASTM D6584 and EN 14105 test for total glycerin, EN 14103 tests for fatty acid methyl esters (FAMEs), and EN 14110 tests for residual methanol. These methods may be performed using either fused silica or metal columns, but the column chosen must have extremely high temperature tolerance. Restek offers both fused silica and metal columns designed specifically for high temperature biodiesel analysis. These columns, the Rtx®-Biodiesel TG, MXT®-Biodiesel TG, Stabilwax®, and Rtx-1® column lines, offer outstanding performance for biodiesel testing.

Rtx®-Biodiesel TG Columns (fused silica)

Rtx®-Biodiesel TG Columns:

- Low column bleed at high temperatures.
- Alumaseal™ connector provides leak-free connection, retention gap extends column life.
- Complete resolution for all compounds from interference peaks.

Description	temp. limits	cat.#
10m, 0.32mm ID, 0.10	to 330/380°C	10292
10m, 0.32mm ID, 0.10 w/2m x 0.53mm retention gap**	to 330/380°C	10291
15m, 0.32mm ID, 0.10	to 330/380°C	10294
15m, 0.32mm ID, 0.10 w/2m x 0.53mm retention gap**	to 330/380°C	10293

**Connected with low-dead-volume Alumaseal™ connector.

Biodiesel Calibration Standards

Concentration is µg/mL in pyridine. Volume is 1mL/ampul unless otherwise noted.

Compound	Solvent	cat.#
(s)-(-)-1,2,4-butanetriol	1,000	33024
(s)-(-)-1,2,4-butanetriol (5mL)	1,000	33032
diolein(1,3-di[*cis*-octadecenoyl] glycerol)	5,000	33022
glycerin	500	33020
monolein (1-mono[*cis*-9-octadecenoyl]-rac-glycerol)	5,000	33021
monopalmitin	5,000	33026
tricaprin (1,2,3-tricaprinoyl glycerol)	8,000	33025
tricaprin (1,2,3-tricaprinoylglycerol) (5mL)	8,000	33033
triolein (1,2,3-Tri[*cis*-octadecenoyl] glycerol)	5,000	33023

Silylation Derivatization Reagents

Compound	CAS#	cat.#
MSTFA (N-methyl-N-trimethylsilytrifluoroacetamide)		
10-pk. (10x1g)	24589-78-4	35600
25g Flex Tube	24589-78-4	35601

Analyzing Total Glycerin in Biodiesel

Rtx®-Biodiesel TG Fused Silica Columns

Glycerin in biodiesel falls out of solution, causing gumming in fuel systems and malfunctioning of engine parts, which eventually leads to inferior engine performance. Total glycerin presents itself in two forms: free glycerin and bound glycerin in the form of glycerides. Derivatization is required for analysis, and both ASTM D6584 and EN 14105 use N-methyl-N-trimethylsilytrifluoroacetamide derivatization reagent.

A 10m x 0.32mm ID Rtx®-Biodiesel TG column with a 2m x 0.53mm ID retention gap is ideal for glycerin analysis. The retention gap is factory coupled using Restek's unique Alumaseal™ connector (Figure 1). This innovative connector is leak-tight and low dead volume, making it advantageous for high temperature work. The data in Figure 2 show the elution of glycerin, monoglycerides, diglycerides, and triglycerides in B100 biodiesel following ASTM Method D6584, utilizing cool on-column injection. The Rtx®-Biodiesel TG column provides good resolution and signal-to-noise ratios for mono-, di-, and triglycerides.

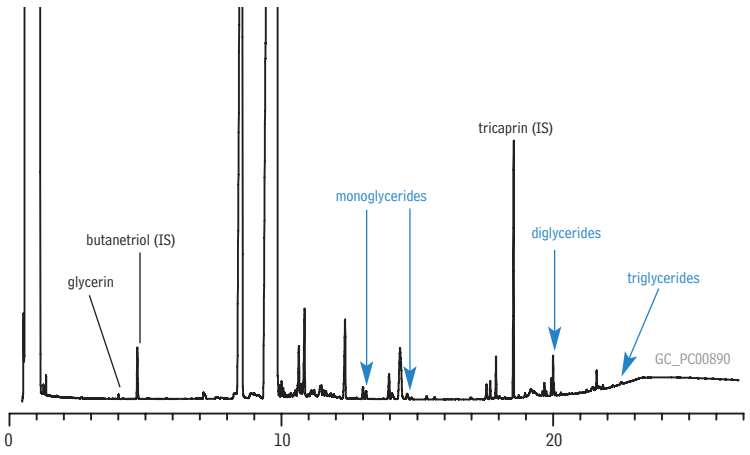
Figure 1: The Alumaseal™ connector

The Alumaseal™ connector is the best column connector for coupling fused silica and metal columns, even columns of different internal diameters. Made of aluminum, it is designed for high temperature performance. These connectors have been factory-coupled and tested using temperature programmed mass spectrometry and have shown no signs of leaks, even at 430°C.

The Alumaseal™ connector offers:

- A leak-tight connection.
- Low dead volume.
- Low thermal mass.
- High inertness.

Figure 2 The Rtx®-Biodiesel TG column meets resolution criteria and shows excellent response for determining glycerin in biodiesel.



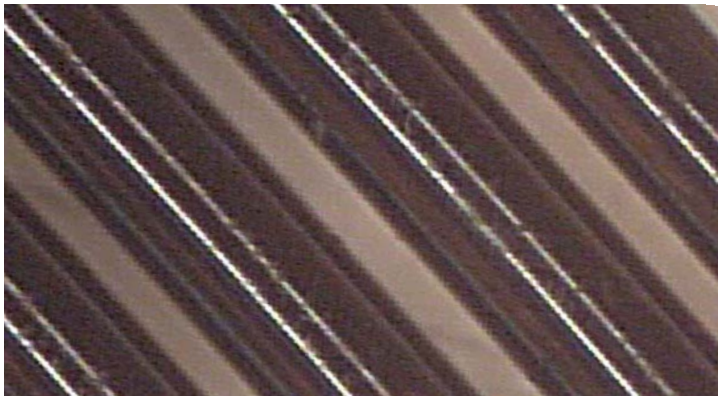
Column:	Rtx®-Biodiesel TG, 10m, 0.32mm ID, 0.10µm connected to 2m x 0.53mm Hydroguard™ tubing using Alumaseal™ connector (cat.# 10291)
Sample:	biodiesel (B100) plus monoolein, diolein, triolein, glycerin, butanetriol, tricaprin
Inj.:	1µL, cool on-column
Inj. temp.:	oven track
Carrier gas:	hydrogen, constant flow
Flow rate:	4mL/min.
Oven temp.:	50°C (hold 1 min.) to 180°C @ 15°C/min. (hold 7 min.) to 230°C @ 30°C/min. to 380°C @ 30°C/min. (hold 5 min.)
Det.:	FID
Det. temp.:	380°C

Comparing Fused Silica to Metal

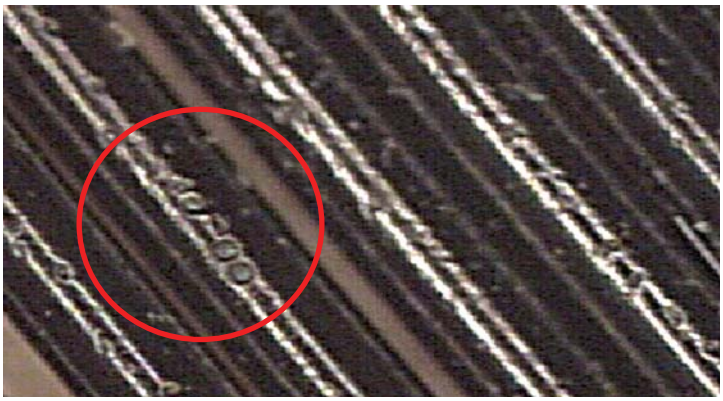
High temperature applications shorten the lifetime of fused silica columns due to deterioration of the polyimide resin used to make the columns. When fused silica columns are exposed to oven temperatures over 400°C the polyimide coating becomes brittle and the deactivation of the column is compromised. Figure 3 shows the effect of cycling a commercially available fused silica column to 430°C for 5 minutes 100 times. Although the column was labeled as stable up to 430°C, the polyimide coating shows damage. The inertness of the column also deteriorates as shown by the loss of peak symmetry for the internal standard butanetriol over multiple injections (Figure 4).

Metal MXT®-Biodiesel TG columns are a better alternative to fused silica columns. As shown in Figure 4, they clearly outperform high temperature fused silica columns under the cycling conditions required for biodiesel analysis. Metal MXT®-Biodiesel TG columns offer greater stability and longer column lifetimes compared to fused silica columns.

Figure 3 Fused silica columns, labeled as stable up to 430°C, show significant pitting and breakdown.



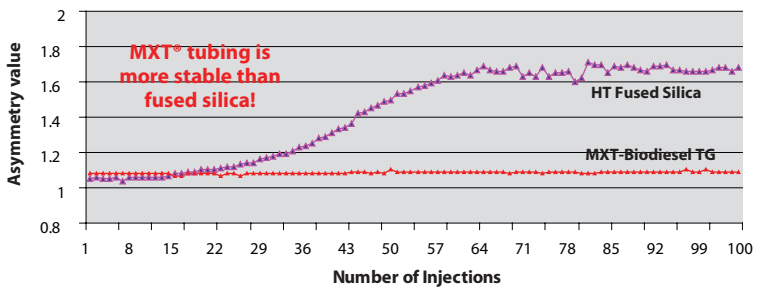
Before



After

100 temperature cycles to 430°C totaling 500 minutes at maximum temperature.

Figure 4 Stable peak shape for internal standard butanetriol on MXT®-Biodiesel TG columns gives more accurate quantification.



Metal Column Solutions: Two Options for Increased Stability and Performance

- 0.32mm MXT®-Biodiesel TG column with a 0.53mm retention gap, factory coupled with an Alumaseal™ connector
- 0.53mm MXT®-Biodiesel TG column with a built-in 0.53mm Integra-Gap™ integrated retention gap

The primary advantage of using metal MXT® columns is that they are more stable at high temperatures than fused silica columns. This means they will exhibit lower bleed, improving analytical performance, and have longer lifetimes, making them a cost-effective option. They also can be brought to high temperatures (430°C) allowing nonvolatile material to be baked off of the column, removing carryover contamination and improving cycle times.

Metal MXT®-Biodiesel TG columns are offered in the same column dimensions as their fused silica counterparts. Two different column configurations are available for cool on-column injection: 1) a 10m (or 15m) x 0.32mm ID MXT®-Biodiesel TG column factory coupled to a 2m x 0.53mm retention gap using an Alumaseal™ connector, and 2) a 14m x 0.53mm ID MXT ®-Biodiesel TG column with a built-in 2m x 0.53mm ID Integra-Gap™ integrated retention gap.

Target analytes resolve well and the solvent and triglyceride peaks show excellent symmetry on both columns (Figures 5 and 6), but the 0.53mm MXT ®-Biodiesel TG column with the Integra-Gap™ integrated retention gap eliminates the need for a connector, making connector-related leaks a thing of the past. Peak shape for butanetriol is very good, demonstrating inertness, and the resolution and response for the mono-, di- and triglycerides is excellent. The leak-proof 0.53mm MXT®-Biodiesel TG column with the Integra-Gap™ integrated retention gap is the ultimate biodiesel solution (Figure 7).

Figure 5 Derivatized B100 samples resolve well on the 15m x 0.32mm MXT®-Biodiesel TG column, which is factory coupled to a 0.53mm retention gap using an Alumaseal™ connector.

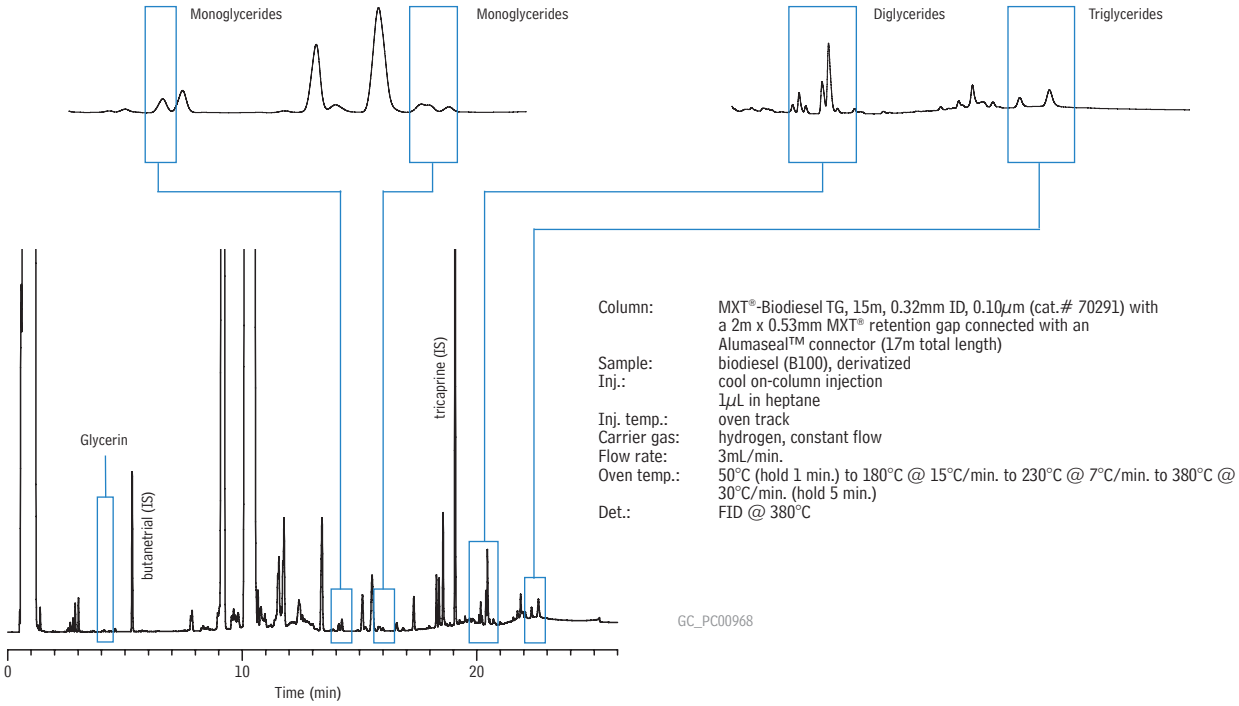
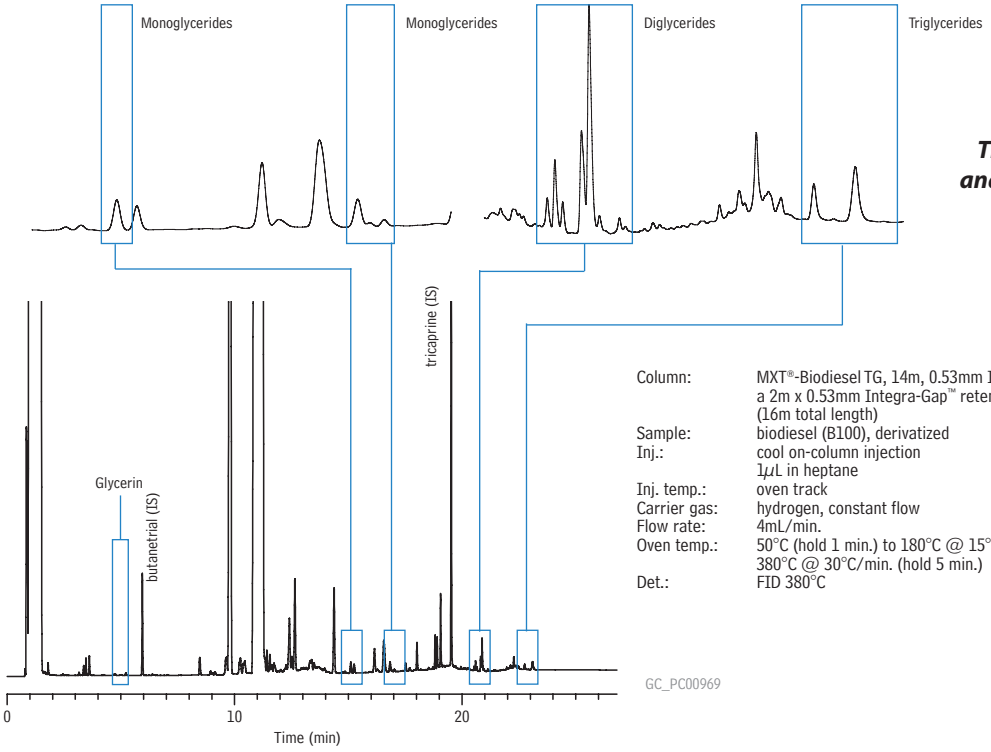


Figure 6 Excellent chromatographic quality and resolution on the 0.53mm MXT®-Biodiesel TG column, with the Integra-Gap™ integrated retention gap.

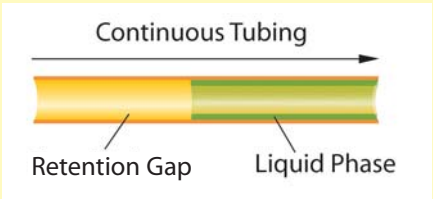


The best solution for analyzing total glycerin in biodiesel!

Column:	MXT®-Biodiesel TG, 14m, 0.53mm ID, 0.16µm (cat.# 70289) with a 2m x 0.53mm Integra-Gap™ retention gap (16m total length)
Sample:	biodiesel (B100), derivatized
Inj.:	cool on-column injection 1µL in heptane
Inj. temp.:	oven track
Carrier gas:	hydrogen, constant flow
Flow rate:	4mL/min.
Oven temp.:	50°C (hold 1 min.) to 180°C @ 15°C/min. to 230°C @ 7°C/min. to 380°C @ 30°C/min. (hold 5 min.)
Det.:	FID 380°C

Figure 7 The Ultimate Biodiesel Solution: MXT®-Biodiesel TG column with Integra-Gap™ integrated retention gap.

The 0.53mm MXT®-Biodiesel TG columns are an innovative alternative to using a 0.32mm column coupled to a 0.53mm retention gap. Restek applied the Integra-Gap™ integrated retention gap technology to the 0.53mm MXT®-Biodiesel TG columns, eliminating the column coupling. These 100% leak-proof columns feature a built-in retention gap, reducing the risk of peak broadening and tailing, and guaranteeing the user many analyses without downtime.



MXT®-Biodiesel TG Columns

- Fast analysis times and sharp glyceride peaks.
- Stable at 430°C for reliable, consistent performance.
- Integra-Gap™ built-in retention gap eliminates manual connection.

MXT®-Biodiesel TG Columns (Siltek® treated stainless steel)

Description	temp. limits	cat.#
14m, 0.53mm ID, 0.16 w/2m Integra-Gap™	-60 to 380/430°C	70289
10m, 0.32mm ID, 0.10	-60 to 380/430°C	70292
10m, 0.32mm ID, 0.10 w/2m x 0.53mm retention gap**	-60 to 380/430°C	70290
15m, 0.32mm ID, 0.10	-60 to 380/430°C	70293
15m, 0.32mm ID, 0.10 w/2m x 0.53mm retention gap**	-60 to 380/430°C	70291

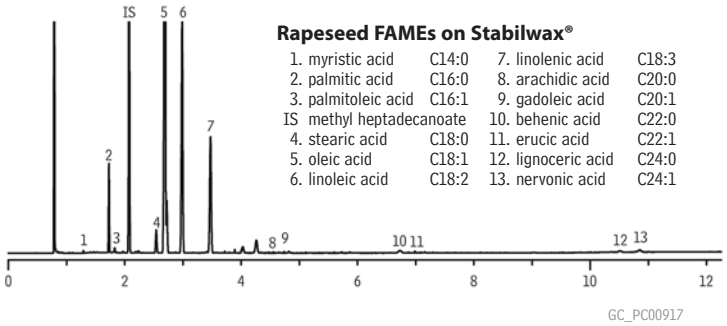
*Total column length=16 meters.
**Connected with low-dead-volume Alumaseal™ connector.

Analyzing FAMES in Biodiesel

FAMEs are the desired end product of biodiesel production and they are analyzed to determine the percent of usable fuel in the final product. A Stabilwax® fused silica GC column affords excellent peak symmetry, resolution, and reproducibility for determining the FAMEs and linolenic acid methyl ester content in B100 biodiesel fuel, following European standard method EN 14103.

As shown in Figure 8, C14:0-C24:1 FAMEs and linolenic acid methyl ester can be determined in less than 11 minutes using a 30m x 0.32mm ID x 0.25µm Stabilwax® column. Particularly notable are the stability of the baseline, excellent peak symmetry, and baseline resolution of all compounds of interest. The Stabilwax® column shows excellent peak shape for all FAMEs, even at low concentrations, which is critical for accurate quantification (Table 1).

Figure 8 Stable baselines, excellent peak symmetry, and rapid, baseline resolution of all compounds characterize FAMEs analyses on a Stabilwax® column.



Column:	Stabilwax®, 30m, 0.32mm ID, 0.25µm (cat.# 10624)
Sample:	rapeseed source of biodiesel (B100), prepared according to European Method EN 14103
Inj.:	1.0µL split (split ratio 100:1), Cyclosplitter® inlet liner (cat.# 20706)
Inj. temp.:	250°C
Carrier gas:	hydrogen, constant flow, 3mL/min.
Linear velocity:	60cm/sec.
Oven temp.:	210°C (hold 5 min.) to 230°C @ 20°C/min. (hold 5 min.)
Det.:	FID
Det. temp.:	250°C

Table I Sources of FAMEs in B100 biodiesel fuel (% m/m).

		Soy	Tallow	Rapeseed	Yellow Grease
Myristic acid	C14:0	0.21	1.7	0.11	0.68
Palmitic acid	C16:0	11.24	25.5	4.1	16.35
Palmitoleic acid	C16:1	0.2	3.27	0.27	1.23
Stearic acid	C18:0	4.04	14.41	1.8	9.32
Oleic acid	C18:1	21.93	40.34	58.57	47.8
Linoleic acid	C18:2	53.84	12.02	22.2	20.01
Linolenic acid	C18:3	7.29	0.99	13.26	2.93
Arachidic acid	C20:0	0.36	0.4	0.79	0.46
Gadoleic acid	C20:1	0.26	1.03	1.79	0.39
Behenic acid	C22:0	0.45		0.57	0.44
Erucic acid	C22:1			0.13	0.23
Lignoceric acid	C24:0	0.16	0.34	0.3	0.24
Nervonic acid	C24:1		0.17	0.54	

Stabilwax® Column (fused silica)

(Crossbond® Carbowax® polyethylene glycol)

ID	df (µm)	temp. limits	length	cat. #
0.32mm	0.25	40 to 250°C	30-Meter	10624

Analyzing Methanol in Biodiesel

Methanol is commonly used to produce biodiesel by derivatizing the fatty acids to methyl esters. The amount of residual methanol must be determined because engine performance can be negatively affected if the methanol concentration in the final product is too high. Methanol in biodiesel is quantified using a headspace method (e.g. EN 14110). We recommend an Rtx®-1 column (30m, 0.32mm ID, 3µm) for this analysis. The selectivity of the Rtx®-1 column is ideal for resolving methanol from interfering peaks in biodiesel fuels.

Conclusion

Whether testing for glycerin, FAMEs, or methanol, Restek can supply the high quality chromatography products required for biodiesel testing. We offer an array of metal and fused silica GC columns designed for high performance biodiesel analysis, including our innovative MXT®-Biodiesel TG column with an Integra-Gap™ integrated retention gap (Table II). Our columns, accessories, and analytical reference materials are designed to improve analytical quality, simplify lab work, and increase productivity. Rely on Restek for innovative solutions to your biodiesel testing needs.

Rtx®-1 Columns (fused silica)

(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	length	cat. #
0.32mm	3.00	-60 to 280/300°C	30-Meter	10184

Table II GC Column Selection Guide for Biodiesel Fuel Methods.

Fused Silica GC Columns	Description	Injection Type	ASTM D6584 Free and Total Glycerin	EN 4103 Ester and Linoleic acid methyl esters	EN 14105 Free and total glycerine and mono, di, and triglycerides	EN 14110 Methanol
Rtx-Biodiesel TG (max temp. 380°C)	15m, 0.32mm ID, 0.1µm w/ 2m x 0.53mm ID retention gap	cool on-column	10293	—	10293	—
Rtx-Biodiesel TG (max temp. 380°C)	15m, 0.32mm ID, 0.1µm	PTV**	10294	—	10294	—
Rtx-Biodiesel TG (max temp. 380°C)	10m, 0.32mm ID, 0.1µm w/ 2m x 0.53mm ID retention gap	cool on-column	10291	—	10291	—
Rtx-Biodiesel TG (max temp. 380°C)	10m, 0.32mm ID, 0.1µm	PTV**	10292	—	10292	—
Stabilwax	30m, 0.32mm ID, 0.25µm	split/splitless	—	10624	—	—
Rtx-1	30m, 0.32mm ID, 3.0µm	headspace	—	—	—	10184
Metal (MXT) GC Columns						
*MXT-Biodiesel TG (max temp. 430°C)	14m, 0.53mm ID, 0.16µm w/ 2m Integra Gap	cool on-column	70289	—	70289	—
MXT-Biodiesel TG (max temp. 430°C)	15m, 0.32mm ID, 0.1µm w/ 2m x 0.53mm ID retention gap	cool on-column	70291	—	70291	—
MXT-Biodiesel TG (max temp. 430°C)	15m, 0.32mm ID, 0.1µm	PTV**	70293	—	70293	—
MXT-Biodiesel TG (max temp. 430°C)	10m, 0.32mm ID, 0.1µm w/ 2m x 0.53mm ID retention gap	cool on-column	70290	—	70290	—
MXT-Biodiesel TG (max temp. 430°C)	10m, 0.32mm ID, 0.1µm	PTV**	70292	—	70292	—

*Recommended for total glycerin analysis.

**PTV=programmed temperature vaporizer.

GC Accessories

Thermolite® Septa

- Usable to 340°C inlet temperature.
- Preconditioned and precision molded.
- Do not adhere to hot metal surfaces.
- Packaged in precleaned glass jars.

Septum Diameter	25-pk.	50-pk.	100-pk.
9mm	27132	27133	27134
9.5mm (3/8")	27135	27136	27137
10mm	27138	27139	27140
11mm (7/16")	27141	27142	27143
11.5mm	27144	27145	27146
12.5mm (1/2")	27147	27148	27149
17mm	27150	27151	27152
Shimadzu Plug	27153	27154	27155

Parker Balston® Hydrogen Generators

- Proton Exchange Membrane (PEM) cell eliminates the need for liquid electrolytes.
- Reliably generate 99.9995% pure hydrogen, for better chromatography.
- Cost-effective, convenient, and safe alternative to high pressure cylinders.

Specifications

Purity:	99.9995% pure hydrogen	Physical Dimensions:	17.12"h x 13.46"w x 17.95"d (43.48 x 34.19 x 45.6cm)
Delivery Pressure:	10-100psig ± 1psig (69-689kPa ± 7kPa)		
Outlet Port:	1/8" compression	Shipping Weight:	40 lbs. (18kg) dry
Electrical Requirements:	100-230VAC/50-60Hz		

- Dimensions: 17.12"h x 13.46" x 17.95"
- 40 lb. dry weight

Description	Capacity	qty.	cat.#
H2PEM-100	100cc/min.	ea.	23065
H2PEM-165	165cc/min.	ea.	23066
H2PEM-260	260cc/min.	ea.	23067
H2PEM-510	510cc/min.	ea.	23068

Small, compact unit—easy to hold and operate.

Restek Electronic Leak Detector

- Reliable thermal conductivity leak detector.
- Responds to leaks in less than 2 seconds.
- Audible alarm plus LED readout.
- Auto zeros with the touch of a button.
- Built-in rechargeable 7.2-volt battery.

Leak Detector Facts

Detectable gases:	helium, nitrogen, argon, carbon dioxide
Battery:	Rechargeable Ni-MH, 7.2 volt
Operating Temperature Range:	32°-120°F (0°-48°C)
Humidity Range:	0-97%
CE Approved:	Yes

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 under any condition. The Restek Electronic Leak Detector may be used for determining trace amounts of hydrogen in a GC environment only.

Also available in money-saving 50-packs!

Capillary Ferrules—For 1/16-Inch Compression-Type Fittings

Graphite Ferrules

- Preconditioned to eliminate out-gassing.
- High-purity, high-density graphite.
- Stable to 450°C.

Vespel®/Graphite Ferrules

- 60%/40% Vespel®/graphite blend, offering the best combination of sealing and workability.
- Stable to 400°C.

Ferrule ID	Fits Column ID	qty.	Graphite	Vespel®/Graphite
0.5mm	0.32mm	10-pk.	20201	20212
0.8mm	0.45/0.53mm	10-pk.	20202	20213

tech tip

Which FID Jet Should I Use?
There are two FID jet configurations for Agilent GCs. The longer "adaptable" jet fits both 5890 and 6890 GCs, and can be used with capillary or packed columns. The shorter "dedicated" jet is for the FID in the 6890 GC that is designed only for use with capillary columns.

Replacement Jets

- Available untreated or Siltek® treated, for maximum inertness.

Capillary Adaptable FID Replacement Jet for Agilent 5890/6890/6850 GCs

0.011-Inch ID Tip	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
Standard	19244-80560	ea.	20670	3-pk.	20671
High-Performance Siltek® Treated	19244-80560	ea.	20672	3-pk.	20673

Capillary Dedicated FID Replacement Jet for Agilent 6890/6850 GCs

0.011-Inch ID	Similar to Agilent part #	qty.	cat.#	qty.	cat.#
Standard	G1531-80560	ea.	21621	3-pk.	21682
High-Performance Siltek® Treated	G1531-80560	ea.	21620	3-pk.	21683

FID Jet Removal Tool for Agilent 5890/6890/6850 FIDs

- Securely grips jet in socket for easy removal or installation.
- Unique, ergonomic handle—easy to hold.

Description	qty.	cat.#
FID Jet Removal Tool for Agilent 5890/6890/6850 FIDs	ea.	22328

Lit. Cat.# 580207