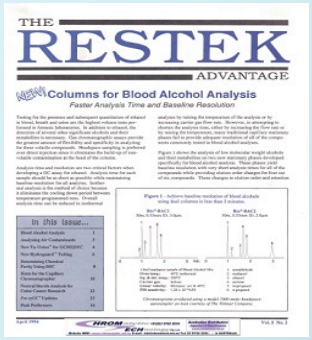
RESTEK ADVANTAGE Newsletter

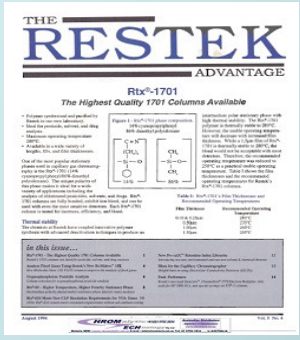
1991-1995 : Quarterly

ADV Overview

1994

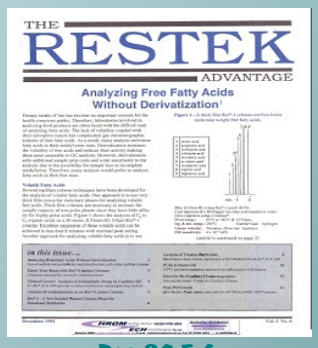


April 94 5.2



Aug 94 5.4

Oct 94 5.4



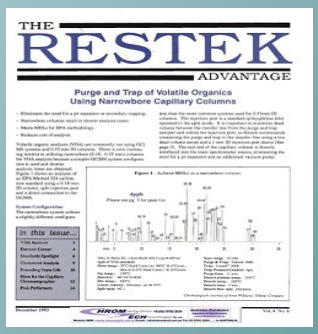
Dec 94 5.6

1993

Jan 93 4.2

Mar 93 4.2

Sept 93 4.3



Dec 93 4.4

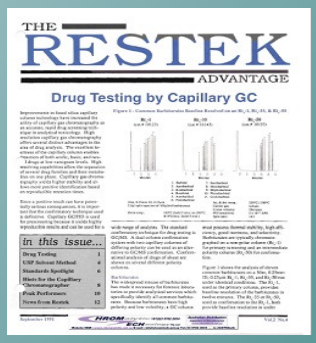
1992

Jan 92 3.1

Sept 92 3.5

Nov 92 3.6

1991



Sept 91 2.4

Nov 91 2.5

Archive Only : *some products may be obsolete / redundant and technologies upgraded !*

. . . see later Restek Updates

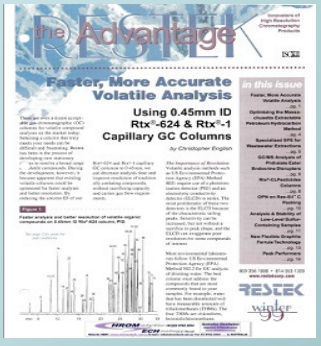
RESTEK ADVANTAGE Newsletter

1996-2000 : Quarterly

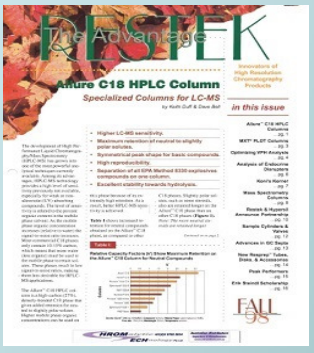
ADV Overview

2000

1999



1998



1997

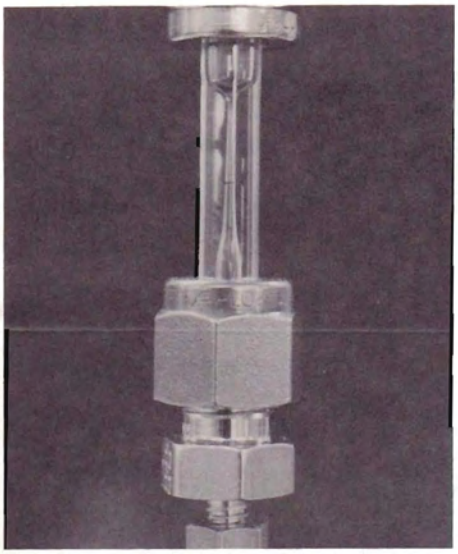
1996

Archive Only : *some products may be obsolete / redundant and technologies upgraded !*

. . . see later Restek Updates

THE RESTEK ADVANTAGE

Vu-Tight™ - A New View to Packed Column Conversion



- visually observe press-tight connection between the column end and sleeve
- fits HP, Varian, and most packed column GCs*
- converts packed column inlets for use with 0.32 & 0.53mm ID fused silica capillary columns (tubing OD must be 0.4mm or greater)
- deactivated, strong, and extremely inert
- slotted top prevents obstruction of carrier gas flow
- low cost

Convert packed column inlets for use with high resolution capillary columns using the new Vu-Tight Direct Injection Sleeve. The Vu-Tight sleeve design offers the simplicity of a straight 1/4-inch sleeve and allows visual confirmation of the seal integrity. The large buffer volume can easily be packed with glass wool for the analysis of dirty samples.

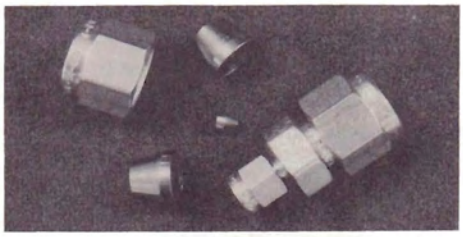
It's easy to use! Simply insert the glass inlet sleeve into the packed column injection port and tighten it with a 1/4-inch nut and graphite ferrule. Then connect the column to the outlet of the sleeve with a 1/4 to 1/16-inch SS reducing fitting. The Press-Tight®taper is positioned for easy observation of a proper seal between the column end and the direct injection sleeve. Sound easy? That's because it is! And the chromatography is exceptional. The Vu-Tight injection sleeve provides sharp solvent peaks and exceptional inertness, even with sensitive compounds like Endrin and DDT.

Vu-Tight Direct Injection Sleeves

cat.# 20342, $28 each
cat.# 20343, $115/5-pack
cat.# 20344, $480/25-pack

* Fits 1/4-inch packed column injection ports with a maximum insertion depth of 4 inches.

Vu-Tight Installation Fittings



Includes a 1/4-inch SS nut and 1/4-inch graphite ferrule for attaching the sleeve to the GC inlet and a 1/4 to 1/16-inch SS reducer plus a 1/4-inch and 0.5mm ID graphite ferrule for attaching the column to the Vu-Tight direct injection sleeve.

Vu-Tight Installation Fittings:
cat.# 20504, $30 per kit

in this issue...

Update . . .

New Uses for the Rt$_x$-200 Trifluoropropyl Phase

- 360°C thermal stability
- low bleed with FIDs, ECDs, MSDs
- selective for lone pair electron functionalities
- ideal confirmation column for many EPA analyses

Trifluoropropyl polysiloxane stationary phases have unique selectivity due to the electrophilic nature of the fluorine atom pendant on the polymer backbone. This selectivity intensifies interactions with compounds that contain groups displaying lone pair electrons such as alcohols, ketones, nitro-containing compounds and electron rich molecules such as Freons. This selectivity switches elution orders and resolves compounds that methyl, phenyl, cyano, and Carbowax®containing phases cannot.

While trifluoropropyl stationary phases have been recognized for their unique selectivity, they have also suffered from low thermal stability, high bleed, poor inertness, and incomplete cross-linking or surface bonding. Restek's new trifluoropropyl polymer, Rt$_x$-200, eliminates the standard problems associated with typical trifluoropropyl phases. Because of the complete surface deactivation and high phase purity, inertness is exceptional allowing highly active compounds to elute without tailing or adsorbing onto the column surface. Since the polymer is bonded to the surface and completely cross-linked, it can be solvent rinsed to clean the sample residue from the polymer.

ECD Bleed

The Rt$_x$-200 is synthesized with advanced polymer technology and is coated on a carefully matched surface deactivation, increasing thermal stability to over 360°C. Background is minimal even on halogen specific detectors such as ECD's. Figure 1 shows an ECD bleed profile of an Rt$_x$-200 column that was temperature programmed to 350°C and 360°C. Even at the column's maximum operating temperature, the ECD bleed is minimal.

Figure 1 - The high degree of immobilization allows the Rt$_x$-200 to be used with an ECD despite the presence of fluorine in the polymer.

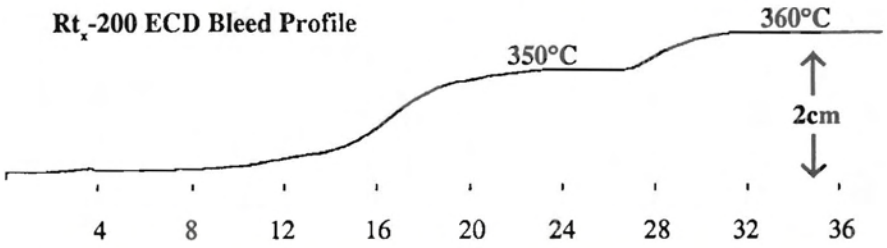
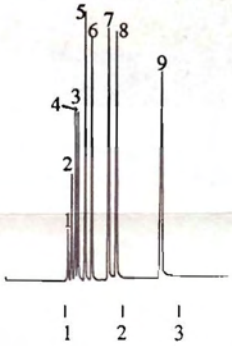


Figure 2 - Excellent peak symmetry of volatile free fatty acids is exhibited on an Rt$_x$-200 capillary column.



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

30m, 0.25mm ID, 0.25µm Rt$_x$-200 (cat.# 15023)
0.8µl split injection of a free fatty acid standard.
Concentration approximately 10 to 20ng/µl.
Oven temp.: 90°C isothermal
Inj. & det. temp.: 250°C
Carrier gas: Hydrogen
Linear velocity: 40cm/sec. (flow rate: 1.4cc/min.)
FID sensitivity: $4x10^{-11}$ AFS
Split vent: 40cc/min.

Applications

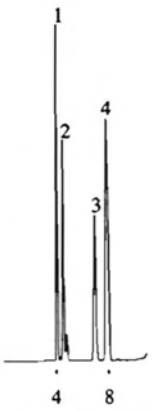
Due to improvements in thermal stability, bleed, and inertness, the Rt$_x$-200 is ideal for the analysis of a wide variety of compounds. Some new, novel applications for the Rt$_x$-200 include free fatty acids, chlorosilanes, glycols, and alkyl nitrates. We previously published applications chromatograms on Freons, polar solvents, phenols, and polynuclear aromatic hydrocarbons (The Restek Advantage, July 1991).

Free Fatty Acids

The inertness and selectivity of the Rt$_x$-200 makes it ideal for the analysis of volatile free fatty acids. Figure 2 shows the analysis of nine common fatty acids on a 30m, 0.25mm ID, 0.25µm Rt$_x$-200 column. All components are virtually

Figures 3a & b - Thick film Rt$_x$-200's are ideal for the analysis of volatile chlorosilanes.

3a

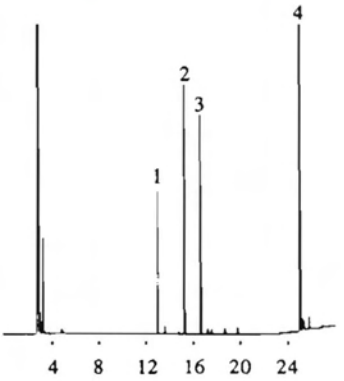


1. Dimethylchlorosilane
2. Methyldichlorosilane
3. Methyltrichlorosilane
4. Dimethyldichlorosilane

60m, 0.53mm ID, 3.0µm Rt$_x$-200 (cat.# 15088)
0.5µl split injection of methyl chlorosilanes
Concentration approximately 250ng/on-column

Oven temp.:	40°C (hold 9 min.) to 250°C @15°C/min. (hold 5 min.)
Inj. temp.:	200°C **Det. temp.:** 250°C
Carrier gas:	Hydrogen
Linear velocity:	40cm/sec. (flow rate: 5.2cc/min.)
HP FID sensitivity:	range=2, attn.=5
Split vent:	90cc/min.

3b

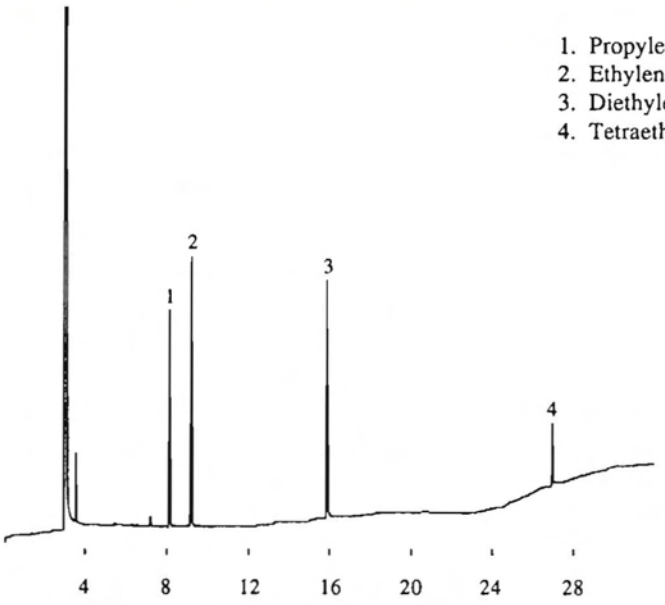


1. Phenyldichlorosilane
2. Methylphenyldichlorosilane
3. Phenylvinyldichlorosilane
4. Diphenyl dichlorosilane

60m, 0.53mm ID, 3.0µm Rt$_x$-200 (cat.# 15088)
0.2µl split injection of phenyl containing chlorosilanes
Concentration approximately 100ng/on-column

Oven temp.:	60°C to 270°C @ 15°C/min. (hold 8 min.)
Inj. temp.:	200°C **Det. temp.:** 270°C
Carrier gas:	Hydrogen
Linear velocity:	40cm/sec. (flow rate: 5cc/min.)
HP FID sensitivity:	range=2, attn.=5
Split vent:	90cc/min.

Figure 4 - Analyze glycols without tailing on an Rt$_x$-200 column.



1. Propylene glycol
2. Ethylene glycol
3. Diethylene glycol
4. Tetraethylene glycol

60m, 0.53mm ID, 3.0µm Rt$_x$-200 (cat.# 15088)
1.0µl split injection of glycols
Concentration approximately 50ng/on-column

Oven temp.:	40°C to 250°C @ 8°C/min. (hold 15 min.)
Inj. temp.:	200°C **Det. temp.:** 250°C
Carrier gas:	Hydrogen
Linear velocity:	40cm/sec. (flow rate: 5cc/min.)
FID sensitivity:	6.4 x 10^{-11}AFS
Split vent:	90cc/min.

baseline resolved in less than three minutes with excellent peak symmetry.

Chlorosilane Analysis

Analysis of chlorosilane monomers on trifluoropropyl capillary columns has been hampered by the limited film thicknesses available. With film thicknesses up to 3.0µm, the Rt$_x$-200 columns are ideal for the analysis of low molecular weight chlorosilanes. Figures 3a and 3b show the analysis of both methyl and phenyl chlorosilanes on a 60m, 0.53mm ID, 3.0µm Rt$_x$-200. The unique selectivity of the Rt$_x$-200 combined with the increased film thickness results in baseline separation of these volatile chlorosilanes.

Glycols

The excellent inertness of the Rt$_x$-200 allows active compounds to be analyzed without tailing or adsorption. Figure 4 shows the analysis of several glycols on a 60 meter, 0.53mm ID, 3.0µm Rt$_x$-200 column. Even at the 50ng level, these reactive components exhibit sharp, symmetrical peaks.

(continued on next page)

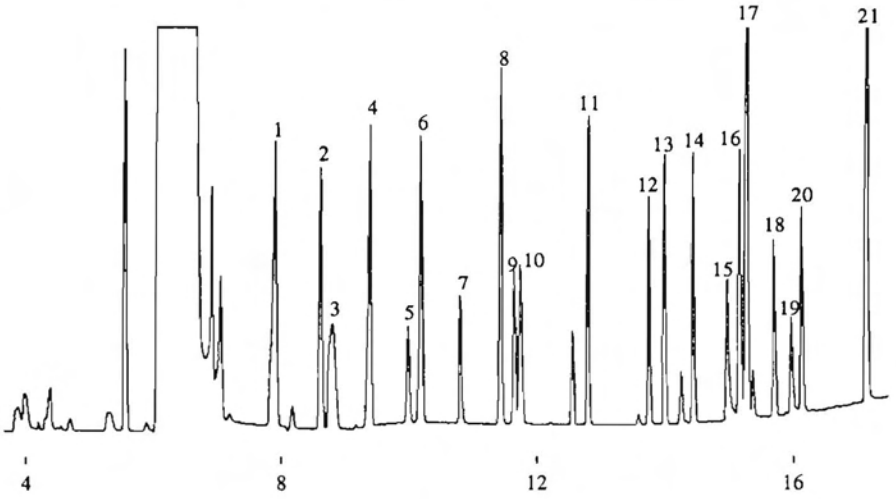
Alkyl Nitrates and Halocarbons

Figure 5 shows the analysis of a complex mixture of alkyl nitrates and halocarbons on a 60m, 0.25mm ID, 1.0µm Rt$_x$-200. The inertness and low bleed of the Rt$_x$-200 allow trace level analysis of these compounds using an Electron Capture Detector. The Rt$_x$-200 provides excellent separation in less than 40 minutes.

The Rt$_x$-200 is a highly selective stationary phase that is ideal for many types of analyses. Because of its unique polarity and high thermal stability, the Rt$_x$-200 is an excellent confirmational column. The 360°C maximum operating temperature, low bleed, and excellent inertness gives analysts an alternative to other intermediate polarity capillary columns. Available in a wide range of film thicknesses and diameters, the Rt$_x$-200 may be the solution to your difficult analytical separations.

Figure 5 - Unique selectivity of the Rt$_x$-200 resolves Alkyl Nitrates and Halocarbons.

1. ethyl nitrate
2. dibromochloromethane
3. carbon tetrachloride
4. isopropyl nitrate
5. ethylene dibromide
6. n-propyl nitrate
7. methylene bromide
8. dibromopropane
9. isobutyl nitrate
10. 2-butyl nitrate
11. n-butyl nitrate
12. 3-pentyl nitrate
13. 2-pentyl nitrate
14. isoamyl nitrate
15. ethyl-hydroxy nitrate
16. n-pentyl nitrate
17. hexachloroethane
18. 3-hexyl nitrate
19. propyl-hydroxy nitrate
20. 2-hexyl nitrate
21. dibromochloropropane



60m, 0.25mm ID, 1.0µm Rt$_x$-200 (cat.# 15056)
15pg/µl alkyl nitrate & halocarbon standard
Oven temp.: 85°C (hold 7 min.) to 225°C @ 12°C/min.
Detector: ECD

courtesy of Dr. Elliot Atlas, National Center for Atmospheric Research

length	df	0.25mm ID	0.32mm ID	0.53mm ID
15 meter	0.10	15005 $250	15006 $275	15007 $290
	0.25	15020 $250	15021 $275	15022 $290
	0.50	15035 $250	15036 $275	15037 $290
	1.00	15050 $250	15051 $275	15052 $290
	1.50		15066 $275	15067 $290
	3.00			15082 $290
30 meter	0.10	15008 $400	15009 $425	15010 $475
	0.25	15023 $400	15024 $425	15025 $475
	0.50	15038 $400	15039 $425	15040 $475
	1.00	15053 $400	15054 $425	15055 $475
	1.50		15069 $425	15070 $475
	3.00			15085 $475

length	df	0.25mm ID	0.32mm ID	0.53mm ID
60 meter	0.10	15011 $685	15012 $740	15013 $850
	0.25	15026 $685	15027 $740	15028 $850
	0.50	15041 $685	15042 $740	15043 $850
	1.00	15056 $685	15057 $740	15058 $850
	1.50		15072 $740	15073 $850
	3.00			15088 $850
105 meter	0.10	15014 $900	15015 $975	
	0.25	15029 $900	15030 $975	
	0.50	15044 $900	15045 $975	
	1.00	15059 $900	15060 $975	
	1.50		15075 $975	
	3.00			15091 $1400

length	df	0.18mm ID	length	df	0.18mm ID	length	df	0.18mm ID
10 meter	0.20	45001 $225	20 meter	0.20	45002 $350	40 meter	0.20	45003 $625
	0.40	45010 $225		0.40	45011 $350		0.40	45012 $625

Standards Spotlight

New Chemical Standards for EPA 500 Series Methods

Restek continues to expand its line of environmental chemical standards. In an effort to provide analytical laboratories with calibration standards to meet their clients requirements, we are pleased to announce the availability of several new products. These mixtures are prepared to the strict specifications required for laboratories performing superfund analyses.

As with all Restek environmental standards, a complete data package is available to comply with EPA regulations. Each data package is designed to be your audit survival package. Restek data packages have been accepted by EPA auditors across the USA.

Method 506 - Phthalate Esters Mix

bis(2-ethylhexyl)phthalate
butyl benzyl phthalate
di-n-butyl phthalate
diethyl phthalate
dimethyl phthalate
di-n-octyl phthalate
bis(2-ethylhexyl)adipate
200µg/ml each in 1ml isooctane

Cat.#	31038	$25ea.
	31038-500	$40 ea. w/data pack
	31138	$225 10pk. w/data pack

Method 524 Volatile Organics Kit

The complete kit contains all target analytes, internal standards, and surrogates recommended for use by this method. All solutions are prepared at a concentration of 2000µg/ml per each component. The kit contains one each of the following mixtures:

502.2 Calibration Mix#1
502.2 Calibration Mix #2
502.2 Calibration Mix #3
502.2 Calibration Mix #4
502.2 Calibration Mix #5
502.2 Calibration Mix #6
Fluorobenzene (Internal standard)
4-bromofluorobenzene (surrogate)
1,2-dichlorobenzene-d_4 (surrogate)

Cat.#	30052	$215ea.
	30052-500	$360 ea. w/data pack

BTEX Standard

This standard mixture can be used to calibrate GC systems for the analysis of aromatic hydrocarbons in petroleum products from leaking underground storage tanks.

Benzene
Toluene
Ethylbenzene
o-Xylene
m-Xylene
p-Xylene
200µg/ml each in 1ml purge & trap grade methanol

Cat.#	30051	$25 ea.
	30051-500	$40 ea. w/data pack
	30151	$225 10pk. w/data pack

Obtaining Copies of EPA Methods

Call or write the following government departments to request copies of the listed EPA methods.

EPA 500 Series, 600 Series (Water)

U.S. Department of Commerce
National Technical Information Service
5285 Port Royal Rd.
Springfield, VA 22161
Phone: (703) 487-4650

EPA SW-846 Methods (Office of Solid Waste)

Superintendent of Documents
U.S. Government Printing Office
Washington, DC 20402-9371
Phone: (202) 783-3238

EPA Superfund Contract Lab Program

U.S. EPA Sample Management Office
P.O. Box 818
Alexandria, VA 22313
Attn: CLP Documents Section
Phone: (703) 684-5678

Hints for the Capillary Chromatographer

Becoming Proficient at Troubleshooting Capillary Chromatography Systems

The key to good troubleshooting is to methodically, logically, and quickly pinpoint problems that arise. Good problem solving techniques are essential in a laboratory to minimize down time.

The first and most important step to becoming proficient at troubleshooting is to read the instrument manuals. Instrument manufacturer's invest a lot of time and expense in writing manuals to provide a better understanding of the GC system. They include many of the basic concepts they have learned over the years that help avoid many common pitfalls. They also provide detailed flow path diagrams and instructions for disassembling injectors, detectors, and other parts that require customer servicing. Spend some time and review the manual. Learn about the inlet and detector systems. Understand the basic pneumatics and flow paths and know where the critical seals are located to quickly identify the source of most problems that arise.

To Begin Troubleshooting

First, determine if the problem is column or system related. Frequently, analysts call our technical service department with what they believe is a column problem. However, after some basic troubleshooting questions, it often turns out to be a bad inlet sleeve or improperly set carrier gas.

To determine if the system or column is the problem, simply install a column of known performance. If the problem remains, then it is most likely a system related problem, or a problem with both the system and the column. If the problem goes away, then it could have been column related or simply that the problem was corrected during re-installation. To be certain that the problem was column related, re-install the problem column again to make sure that the problem reappears.

Routine Instrument Maintenance

Usually, a careful methodical approach to troubleshooting is not attempted until the common instrument problems are addressed. Common instrument maintenance procedures performed are:

- changing dirty or contaminated inlet sleeves
- replacing the septum
- checking the inlet seal (o-ring or ferrule) for leaks
- confirming proper column insertion distances
- leak checking all column connections and external fittings
- replacing spent purifiers
- checking for properly set flow rates and linear velocity
- inspecting gauge pressures, electrometer settings, all temperatures, signal levels, integrator settings, and any other parameters that could be suspect.

Routine Column Maintenance

While the column is out of the instrument, perform routine column maintenance. Common maintenance procedures performed are:

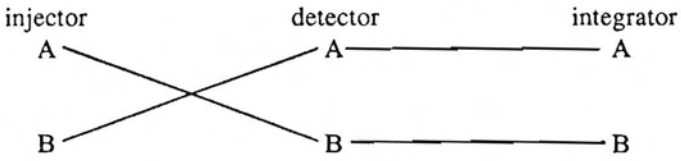
- inspecting the column for spontaneous breakage, discoloration, or contamination
- cutting two loops off of the inlet and one loop off of the detector end of the column

If Routine Maintenance Does not Work, Begin Diagramming and Documenting

Now troubleshooting gets tough. If the problem is not solved after routine maintenance, immediately begin documenting what has been done and start diagramming what should be done. This aids in communicating to others what effect changing variables have on solving the problem. Document the procedures in chronological order listing times, dates, and important instrument parameters. Label all troubleshooting chromatograms. These steps help to inform anyone else that may be working on the system of the troubleshooting procedures that have been completed.

Start with a simple instrument diagram (Figure 1), and try switching Column A to Detector B and vice versa. If the problem moves to detector B, then the problem is most likely occurring in the injector.

Figure 1 - Begin problem isolation when system is at fault.



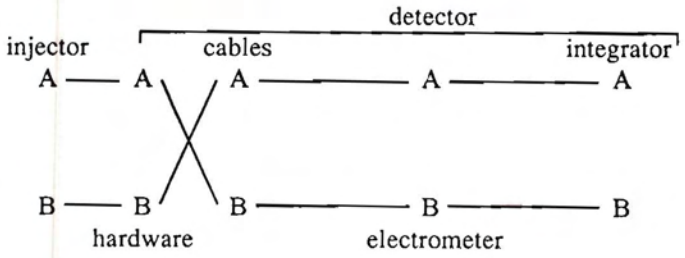
(connect the column to injector A and detector B)

Some common injector problems we have observed are:

- wrong size graphite o-ring on HP inlet sleeve
- wrong sleeve type (using splitless sleeve for split analyses)
- leaking or contaminated metal disk on HP inlet
- bad solenoid valve containing split flow
- knife edge not cutting septum in Varian systems
- wrong length sleeve used in Varian systems
- not using glass wool with fast injecting autosamplers

If the problem stays on detector A when the column outlet is switched, then suspect a detector problem. Begin isolating detector problems by switching hardware, cables, electrometers, integrators, or any suspect part in the pathway. Figure 2 shows the detector hardware being isolated. If the problem goes away from the A side when the detector base is changed, then that detector is most likely the cause and should be replaced.

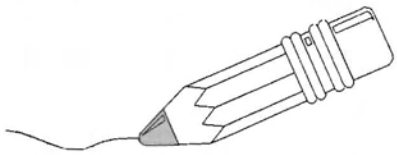
Figure 2 - Isolating Detector Problems



Some common detector problems we have observed are:

- broken or leaking jets
- column or ferrule fragment located inside the jet
- plugged jet orifice
- column installed too far into the detector
- oxidized polarizer or signal contacts
- shorted insulator on the collector assembly
- leak at the detector base
- bad needle valve or regulator
- incomplete or oxidized ground
- bad heater or heater controller
- air conditioning air currents blowing on the detector
- bad or contaminated carrier or combustion gasses
- bottled air with less than 21% O_2
- detector gasses not set properly or optimized

The complexity of a capillary GC system almost guarantees that one day you will be faced with troubleshooting a difficult problem. If you have read the manuals and follow a logical troubleshooting sequence, you can quickly isolate the cause of most problems.

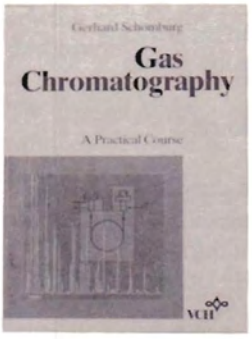


If there's a topic you'd like to see covered in Hints for the Capillary Chromatographer, write to:

Hints Topics,
c/o Restek Corporation,
110 Benner Circle,
Bellefonte, PA 16823-8812.

Useful Additions to Your Chromatographic Library

The field of capillary chromatography is complex and new discoveries are made every year. It is difficult to keep up with new innovations and expand one's knowledge of existing techniques. We often get requests from our customers for recommendations on books relating to capillary gas chromatography. We have received several books and have found a few that are noteworthy.



Gas Chromatography, A Practical Course

Gerhard Schomburg, Max-Planck Institute

This new book, written by a world-renowned and award-winning scientist, provides a practical approach to gas chromatography suitable for both the novice and for the specialist. **Gas Chromatography** includes background theory, recent GC procedures, a course in gas chromatography and forty chromatograms with detailed explanations. This book will be of special interest to research chemists in analytical, organic, environmental, clinical and biochemistry; food scientists, toxicologists, pharmacologists, students and libraries.

VCH Publishers, 1990 • Paper • 320pp.
cat.# 20459 $55

Restek Recommended!

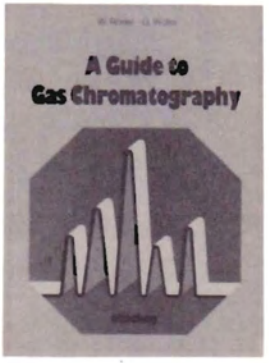
Modern Practice of Gas Chromatography, Second Edition

Edited by Robert L. Grob, Villanova University

The Second Edition of *Modern Practice of Gas Chromatography* comprehensively treats the theory, instrumentation, and applications of gas chromatography. Chapters on applications to the petroleum field, high resolution gas chromatography, and optimization in gas chromatography are featured, and coverage includes packed columns and packed column selection, techniques and instrumentation such as qualitative and quantitative analysis by gas chromatography, and more.

John Wiley & Sons, Inc., 1985 • 897pp.
cat.# 20464 $95

Guide to Gas Chromatography



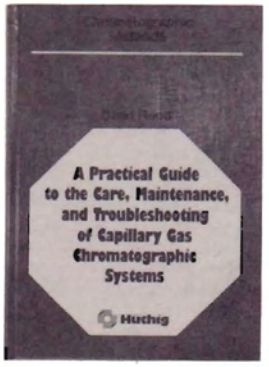
Wolfgang Rödel, Gerhard Wölm

This book provides a general understanding of the principles, methods, and applications of gas chromatography. Priority was given to practical aspects only and theory is confined to explanations and formulas necessary for understanding techniques.

Huethig Publishing, Ltd., 1987 • 304pp.
cat.# 20455 $53

New! Hot off the Press!

A Guide to the Care, Maintenance and Troubleshooting of Capillary Gas Chromatographic Systems



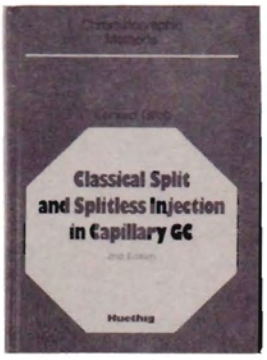
Dean Rood

This guide places strong emphasis on avoiding problems. It is organized by the nature of chromatographic problem encountered. Each possible explanation for the problem refers back to the text of the book which outlines procedures to diagnose and repair the problem, but more importantly, procedures to prevent or minimize the frequency and severity of the problem. A comprehensive, step-by-step flow chart is included to aid in pinpointing the problem area. The text is written so that it can be used as a guide in the proper operation and maintenance of a capillary gas chromatograph to obtain maximum performance with minimal difficulties and effort. Particular care was taken to keep the explanations on a practical level so that intimate knowledge of

chromatography and chemistry is not required to fully benefit from the information presented.

Huethig Publishing, Ltd., Xii + 192pp.
cat.# 20450 $47

A must for those using splitless injections!



Classical Split and Splitless Injection in Capillary GC

Konrad Grob

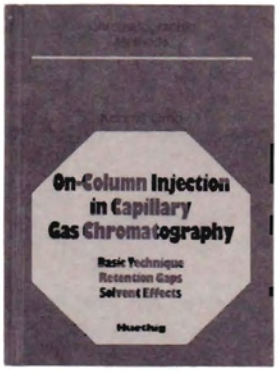
The classical techniques of split and splitless injection are still by far the most common methods of sample introduction, and yet, despite their age, many of the fundamental problems encountered have hardly been described before. This book probably represents the very first comprehensive, single-volume treatment of all aspects of split and splitless injection. The volume is divided into three major parts: Split injections; Splitless injection; Problems Relating to the Hot Syringe Needle in Vaporizing Injection. A brief further section is also devoted to a more recent development: Programmed Temperature Vaporizing (PTV) Injection.

Huethig Publishing, Ltd., 1988 • 324pp.
cat.# 20451 $78

On-Column Injection in Capillary Gas Chromatography

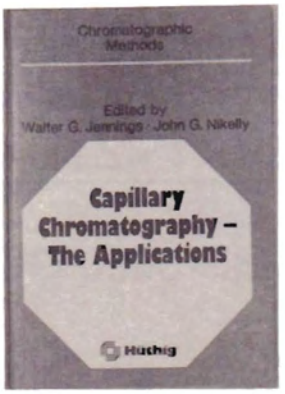
Basic Technique; Retention Gaps; Solvent Effects

Konrad Grob



On-column injection techniques have become an attractive mode of sample introduction due to the minimizing if detrimental adorption and non-linearity problems associated with split/splitless techniques. Grob's text is a *must read* treatise for the novice as well as the experienced GC practioner desiring to gain the on-column advantages. Basic technique is explained clearly with excellent schematics. Topics include: sample introduction, injector design, syringes, solvent and temperature effects. Full chapters are spent discussing solvent effects and retention gaps. This is a real how and why guide to on-column injection.

Huethig Publishing, Ltd., 1987 • xx + 591pp.
cat.# 20453 $110



Capillary Chromatography

The Applications

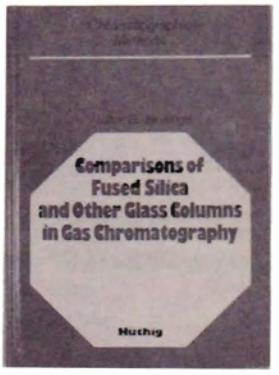
edited by W. Jennings and J. Nikelly

This symposium-based book describes several newly developed applications of fused silica capillary columns mainly in gas chromatography. The volume details experimental conditions and applications in the analysis of organic pollutants in ambient air, pesticides in environmental samples, and components of microbial products and pharmaceuticals. Besides the applications, there are sections on equivalency of different stationary phases and computerized modeling of new phases.

Huethig Publishing, Ltd., 1991 • Hardcover • 156pp.
cat.# 20452 $60.75

Comparisons of Fused Silica and other Glass Columns in Gas Chromatography

W.G. Jennings



This book deals with the various types of glass columns used in gas chromatography. It includes a detailed account of the construction materials, a description of the pretreatment of the capillary, methods for evaluating inertness and testing uncoated capillaries and coated columns. In addition, the physical characteristics of glass columns are considered and the advantages conferred by column flexibility are presented.

Huethig Publishing, Ltd., 1981 • 88pp.
cat.# 20456 $25

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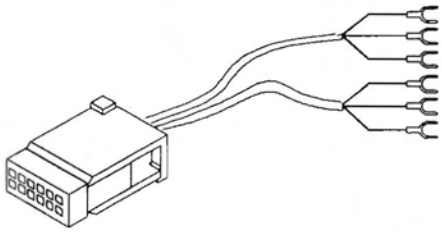

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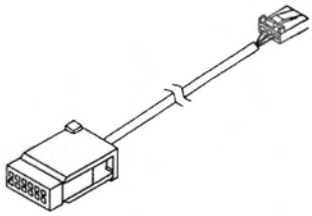

Connect an HP-5890 GC to an HP integrator (for second inet integrator). Replaces HP part number 34900-60610.

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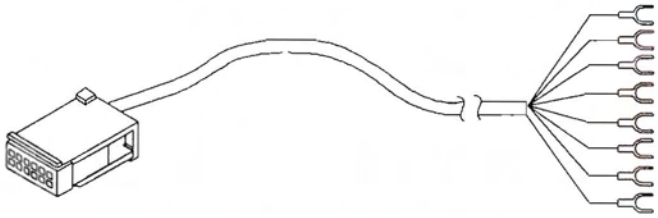
cat.# 20651, $65/2-pack



Connect an HP-5890 GC to remote start another piece of equipment or to start the HP-5890 GC from that piece of equipment. Replaces HP part number 05890-61080.

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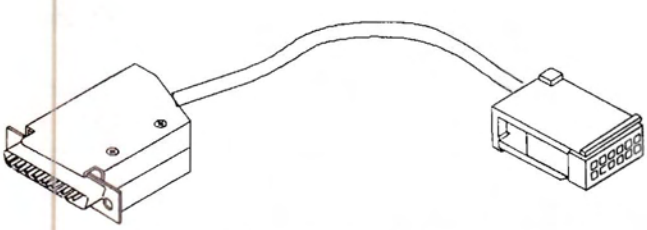

Connect an HP-5890 GC to an HP-3396 integrator to enable remote starts (non-inet connection from GC to integrator). Replaces HP part number 03394-60560.

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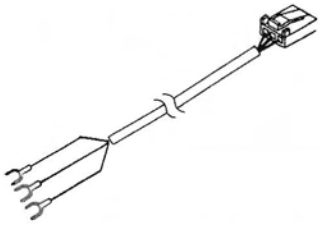

Connect an HP-3396 integrator to another non-HP type of GC. Replaces HP part number 35900-60630.

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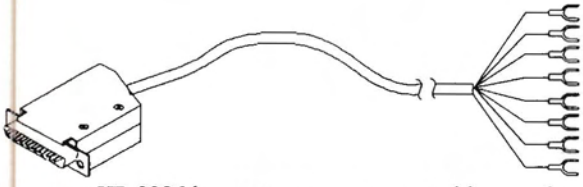

Connect an HP-3396 integrator to remote start either another piece of equipment or to start the HP-3396 integrator from that other piece of equipment. Replaces HP part number 03394-60540.

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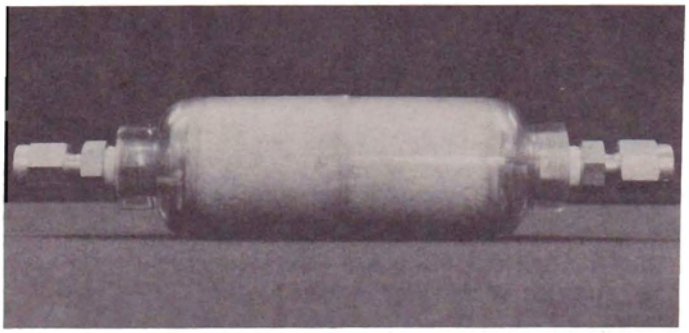
New! Indicating Hydrocarbon Trap for Compressed Air



Many laboratories now run Flame Ionization Detectors (FID) from oil-lubricated air compressors or from house air lines. Although most of these system have extensive filtration devices to remove oil vapors or mist from the air stream, there is no way to determine if these filters are doing their job or when to change them. The only indication that the filters are not working is when oil contamination reaches the detector and creates massive baseline disturbances, ghost peaks, and clogged jets. By then it is too late and only laborious solvent rinsing of the gas lines and detector fittings will restore the stability of the FID.

The Restek Wizards have come up with a novel solution to this problem. Our new Indicating Hydrocarbon Trap changes from pink to red as it absorbs oil vapors. This trap gives advance warning should the filters on the compressed air lines ever fail. In addition to changing color, the trap reduces oil vapor concentration down to five parts per million to prevent gross contamination of the instrument lines.
Available with 1/8″ or 1/4″ tube compression fittings, these traps are a must for any lab using oil lubricated air compressors as a gas source for their FIDs.

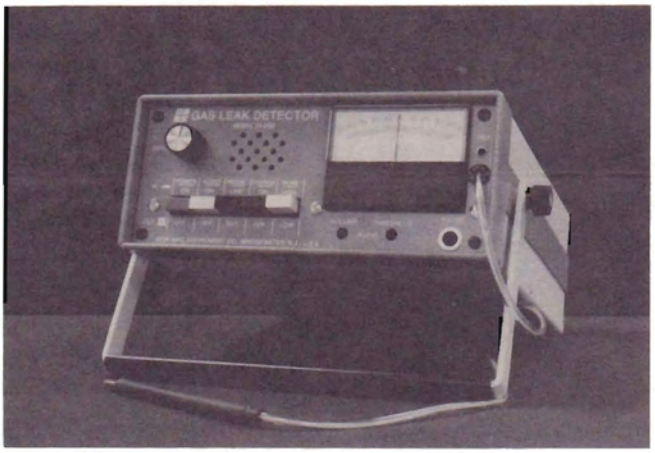
1/4″ Indicating Hydrocarbon Trap: cat.# 20636, $55
1/8″ Indicating Hydrocarbon Trap: cat.# 20637, $55

Gow-Mac Leak Detector

- identifies minute leaks that are undetectable by liquid leak detectors
- contamination and residue-free leak detection
- essential for capillary chromatographers
- prolongs column lifetime and stabilizes sensitive detectors by detecting all points of O_2 influx

Leaks in a gas chromatographic system increase detector noise, cause baseline instability, shorten column lifetime, waste expensive carrier gas, and increase the error of analyses. A Gow-Mac thermal conductivity leak detector is not a luxury, it is a must for all capillary chromatographers. In fact, a Gow-mac leak detector is so sensitive that it detects the instantaneous, minute leak caused while a syringe penetrates the septum during an injection.

The Gow-Mac is a portable unit that operates on line voltage or an internal, rechargeable, leak/acid gel battery. It also incorporates an audible alarm as well as a visual readout device. This unit is set for 115V/60Hz operating voltage, but is internally switchable to 230B/60Hz.
cat.# 20130, $995 each



Limited Time Offer!

Order a Gow-Mac leak detector before December 31, 1991, and receive a coupon with the Gow-Mac for a FREE Restek Wizard Sweatshirt.

Product Changes

Varian SPI Inlet Sleeves: Our 0.8mm ID Varian SPI sleeves (cat.#'s 20778, 20779, & 20780) were originally designed for the direct injection mode only. We have redesigned this SPI sleeve to allow both direct and on-column injections. We recommend that the on-column mode be used with clean samples and the direct injection mode be used for samples containing non-volatile residues.

Less Fragile, More Inert Silica Wool: We have changed the deactivation procedure and enhanced the flexibility of our silica wool (cat.# 20790). These improvements make it easier to stuff the wool into the sleeves and maintain inertness with active compounds.

News from Restek

Restek Licenses Fused Silica Technology from HP

After several years of negotiations, Restek and Hewlett-Packard have finally signed a licensing agreement. Effective August 5, this license allows Restek to manufacture and distribute fused silica capillary columns under HP's U.S. Patent 4,293,415 and several other foreign patents. This license covers all fused silica capillary columns previously manufactured by Restek and is in effect until the patent expires in 1998.

The Wizards Celebrate Six Innovative Years

October 1991 marked Restek's sixth anniversary. From our start in a business incubator in 1985, we have grown to over sixty people dedicated to supplying innovative products and services. A major expansion of our facilities is currently underway. We would like to take this opportunity to thank our loyal customers who have supported us and helped us grow so rapidly.

Truth in Advertising

Recently, we have noticed a rash of advertisements by some of our competitors that can deceive the inexperienced analyst. An ad by one of our competitors misleadingly compares our 105 meter Rt_x-502.2 column to their 75 meter column. They state that their columns costs less and produces faster analysis times. But, they fail to mention that their column actually costs more per meter and that their column produces seven pairs of co-eluting peaks while our column only exhibits two co-eluting pairs. Every lab should be concerned with reducing cost and analysis time, but not at the expense of producing reliable data.

There has also been a wave of ads by several companies offering "EPA Certified Standards". While it may be true that a few standards that these companies supply do have EPA approval, the reality is that the vast majority of standards that they offer have no EPA approval and may not even be quality controlled.

Restek is committed to satisfying the needs of our customers with products that are innovative and offer honest benefits. We would never stoop to overstating the merits of our products just to make a sale. Honesty is always the best policy.

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Restek capillary columns are manufactured under U.S patent 4,293,415, licensed by Hewlett-Packard Company

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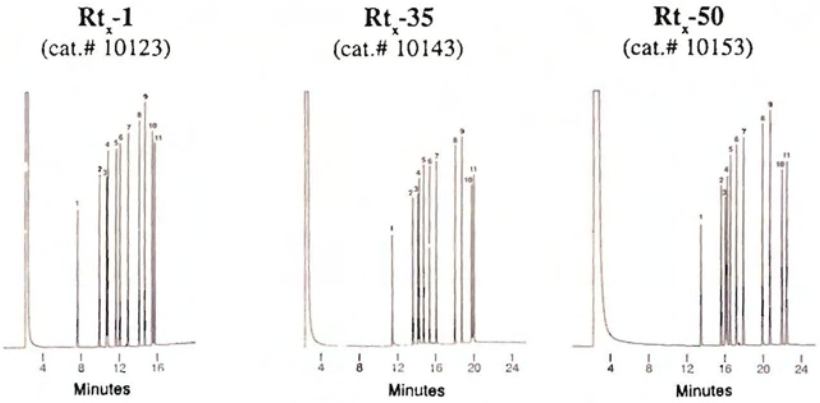
Drug Testing by Capillary GC

Improvements in fused silica capillary column technology have increased the utility of capillary gas chromatography as an accurate, rapid drug screening technique in analytical toxicology. High resolution capillary gas chromatography offers several distinct advantages in the area of drug analysis. The excellent inertness of the capillary column enables detection of both acidic, basic, and neutral drugs at low nanogram levels. High resolving capabilities allow the separation of several drug families and their metabolites on one phase. Capillary gas chromatography yields higher stability and allows more positive identification based on reproducible retention times.

Since a positive result can have potentially serious consequences, it is important that the confirmatory technique used is definitive. Capillary GC/FID is used for prescreening because it yields highly reproducible results and can be used for a wide range of analytes. The standard confirmatory technique for drug testing is GC/MS. A dual column confirmation system with two capillary columns of differing polarity can be used as an alternative to GC/MS confirmation. Confirmational analysis of drugs of abuse are shown on several different polarity columns.

Figure 1 - Common Barbiturates Baseline Resolved on an Rt_x-1, Rt_x-35, & Rt_x-50



1. Barbital
2. Aprobarbital
3. Butabarbital
4. Butalbital
5. Amobarbital
6. Pentobarbital
7. Secobarbital
8. Hexobarbital
9. Mephobarbital
10. Phenobarbital
11. Cyclobarbital.

30m, 0.25mm ID, 0.25µm
2.0µl split injection of 100µl/ml barbiturates

Oven temp.: 150°C (hold 5 min.) to 290°C @ 8°C/min. (hold 5 min.)

Inj. & det. temp.: 250°C / 300°C
Carrier gas: helium
Linear velocity: 20cm/sec.
FID sensitivity: 2×10^{-11} AFS
Split ratio: 10:1

in this issue...

Barbiturates

The widespread misuse of barbiturates has made it necessary for forensic laboratories to provide analytical services which specifically identify all common barbiturates. Because barbiturates have high polarity and low volatility, a GC column must possess thermal stability, high efficiency, good inertness, and selectivity. Barbiturates are typically chromatographed on a non-polar column (Rt_x-1) for primary screening and an intermediate polarity column (Rt_x-50) for confirmation.

Figure 1 shows the analysis of eleven common barbiturates on a 30m, 0.25mm ID, 0.25µm Rt_x-1, Rt_x-35, and Rt_x-50 run under identical conditions. The Rt_x-1, used as the primary column, provides baseline resolution of the barbiturates in twelve minutes. The Rt_x-35 or Rt_x-50, used as confirmation to the Rt_x-1, both provide baseline resolution in under

twenty-three minutes. The dual column systems offer baseline resolution, minimal analysis times, and immediate confirmation.

Opiates

The opiate drug class primarily refers to morphine and codeine, but is loosely used to describe a group of narcotic analgesics and their semi-synthetic derivatives. Opiate quantitation is typically performed on a non-polar column (Rt_x-1) or an intermediately polar column (Rt_x-50).

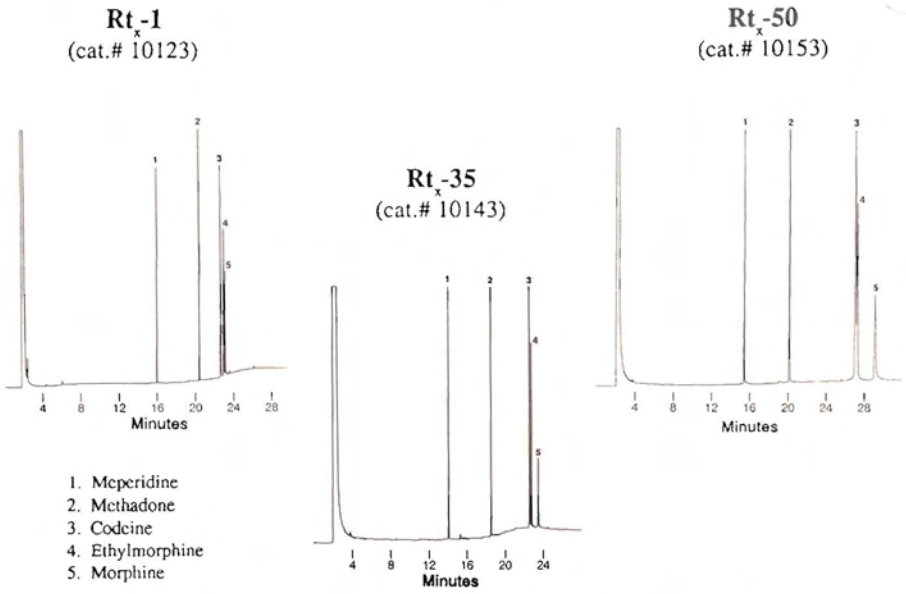
Figure 2 shows the simultaneous analysis of five opiates on a 30m, 0.25mm ID, 0.25µm Rt_x-1, Rt_x-35, and Rt_x-50. The Rt_x-1 and Rt_x-35 give baseline resolution between the critical components codeine, ethylmorphine, and morphine in under twenty-four minutes and show excellent peak symmetry. The Rt_x-50 can also be used as confirmation to the Rt_x-1, however, resolution between codeine and ethylmorphine is only seventy-five percent and the total analysis time is twenty-nine minutes.

Depressants

Depressants are a group of structurally similar compounds used as sedatives and abused for their euphoric properties. They are all weak bases and are typically analyzed on an intermediately polar column (Rt_x-50) by FID.

Figure 3 shows the simultaneous analysis of five depressants on a 30m, 0.25mm ID, 0.25µm Rt_x-1 and Rt_x-35 at 260°C isothermal. Both columns give baseline resolution in 12.5 minutes and 30.5 minutes, respectively. Because the depressants are highly polar, they are retained longer on the Rt_x-35 than on the Rt_x-1. The Rt_x-50 can also be used as a confirmation column to the Rt_x-1, but not simultaneously. Total analysis time of the depressants on the Rt_x-50 at 260°C isothermal is forty-five minutes and peak shape is poor. If the depressants are run at 290°C, analysis time decreases to fifteen minutes and peak symmetry is restored. This occurs because the higher temperature decreases the k of the depressants and allows them to elute faster from the Rt_x-50.

Figure 2 - Confirmational Analysis of Opiates & Derivatives on an Rt_x-1, Rt_x-35, & Rt_x-50



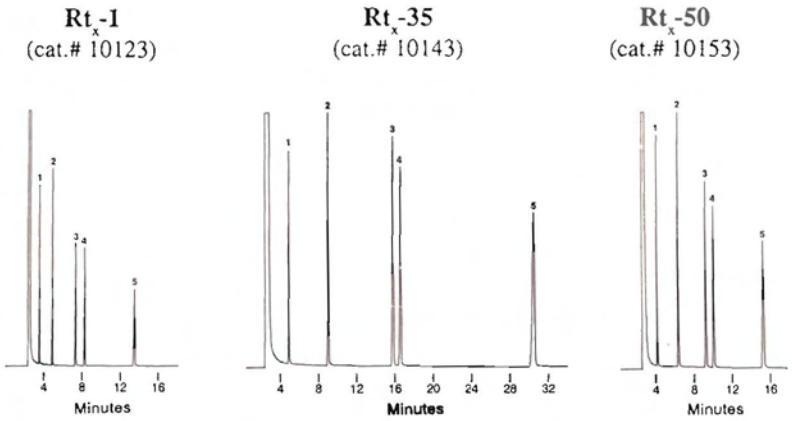
1. Meperidine
2. Methadone
3. Codeine
4. Ethylmorphine
5. Morphine

30m, 0.25mm ID, 0.25µm
2.0µl split injection of 0.1mg/ml opiate standard

Oven temp.: 100°C to 300°C @ 10°C/min. (hold 15 min.)

Inj. & det. temp.: 250°C / 300°C
Carrier gas: helium
Linear velocity: 20cm/sec.
FID sensitivity: 2 x 10^{11} AFS
Split ratio: 10:1

Figure 3 - Simultaneous Confirmational Analysis of Depressants on an Rt_x-1, Rt_x-35, & Rt_x-50



1. Glutethimide
2. Methaqualone
3. Diazepam
4. Chlorpromazine
5. Flurazepam

30m, 0.25mm ID, 0.25µm
2.0µl split injection of an 0.2mg/ml depressant standard

Oven temp.: 260°C isothermal (Rtx-1 & Rtx-35)
290°C isothermal (Rtx-50)

Inj. & det. temp.: 260°C / 300°C
Carrier gas: helium
Linear velocity: 20cm/sec.
FID sensitivity: 2 x 10^{11} AFS
Split ratio: 10:1

Base/Neutral and Acid/Amphetamine Drug Screen

Capillary GC gives forensic chemists the ability and confidence to perform drug analysis accurately. Capillary column inertness enables detection of acidic, basic, and neutral drugs at low nanogram levels and the high resolving capabilities allow the separation of several drug families and their metabolites on a single stationary phase.

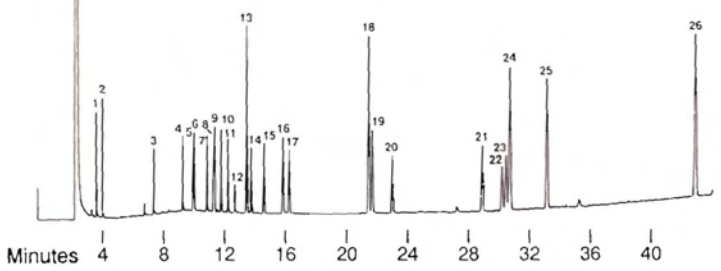
Figure 4 shows the analysis of the barbiturate, opiate, depressant, and stimulant mixes on the Rt_x-1 by GC/FID. The Rt_x-1 resolves all sample components in under forty-three minutes. Resolution between Methylphenidate and Pentobarbital is sixty percent. All other components are baseline resolved.

Another example of the excellent inertness and high resolving capabilities of capillary columns is shown in the analysis of a Tox-Clean drug standard mix. Figure 5 shows the analysis of barbiturates, depressants, tricyclic anti-depressants, opiates, and stimulants on the Rt_x-1. The Rt_x-1 yields resolution of a twenty component drug mix in under twenty-five minutes. Resolution between Imipramine and Oxycodone is seventy percent, all other drug components are baseline resolved. As seen from the chromatogram, all of the drug compounds have excellent peak symmetry on the Rt_x-1.

Detection and identification of unknown substances in biological materials is a challenging task for a forensic toxicologist. Forensic laboratories depend on methods that provide accurate drug identifications, yield rapid analysis times, and permit expanded detection limits of more drugs at lower concentrations. Capillary GC can provide accurate, rapid drug screening in analytical toxicology. Capillary columns have the necessary inertness to quantitate acidic, basic, and neutral drugs, have the separation efficiency to resolve several drug families in a single run, and have the high thermal stability to analyze highly polar, low volatility drugs and eliminate stationary phase bleed on sensitive detectors. ■

Figure 4 - Rt_x-1 Resolves Acidic, Basic, & Neutral Drugs

1. Amphetamine
2. Methamphetamine
3. Barbital
4. Aprobarbital
5. Butabarbital
6. Butalbital
7. Amobarbital
8. Methylphenidate
9. Pentobarbital
10. Meperidine
11. Secobarbital
12. Caffeine
13. Glutethimide
14. Hexobarbital
15. Mephobarbital
16. Phenobarbital
17. Cyclobarbital
18. Methaqualone
19. Methadone
20. Cocaine
21. Codeine
22. Ethyl morphine
23. Morphine
24. Diazepam
25. Chlorpromazine
26. Flurazepam

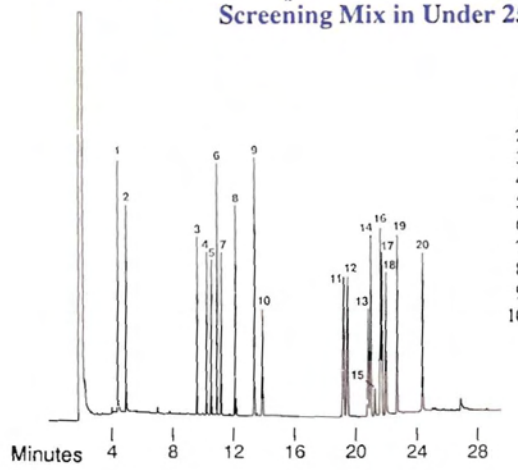


30m, 0.25mm ID, 0.25µm Rtx-1 (cat.# 10123)
2.0µl split injection of a 0.15mg/ml DOA standard

Oven temp..	150°C (hold 4 min.) to 200°C @ 10°C/min. (hold 2 min.) to 300°C @ 2°C/min. (hold 5 min.)
Inj. & det. temp..	250°C / 300°C
Carrier gas:	helium
Linear velocity:	20cm/sec.
FID sensitivity:	2 x 10^{-11} AFS
Split ratio:	10:1

Figure 5 - Rt_x-1 Resolves Twenty Component Drug Screening Mix in Under 25 Minutes

1. Amphetamine
2. Methamphetamine
3. Butabarbital
4. Amobarbital
5. Pentobarbital
6. Meperidine
7. Secobarbital
8. Glutethimide
9. Phencyclidine
10. Phenobarbital
11. Methaqualone
12. Methadone
13. Cocaine
14. Doxepin
15. Amitriptyline
16. Imipramine
17. Oxycodone
18. Desipramine
19. Pentazocine
20. Codeine



30m, 0.25mm ID, 0.25µm Rtx-1 (cat.# 10123)
2.0µl split injection of a 50µg/ml drug mix.

Oven temp.:	110°C (hold 2 min.) to 170°C @ 20°C/min. to 210°C @ 8°C/min. (hold 10 min.) to 260°C @ 15°C/min. (hold 10 min.)
Inj. & det. temp.:	250°C / 300°C
Carrier gas:	helium
Linear velocity:	20cm/sec.
FID sensitivity:	2 x 10^{-11} AFS
Split ratio:	10:1

Phase & Composition	length	ID	df	cat.#	price
Rtx-1	30m	0.25mm	0.25µm	10123	$370
Rtx-35	30m	0.25mm	0.25µm	10423	$370
Rtx-50	30m	0.25mm	0.25µm	10523	$370

Improving the Analysis of Residual Solvents in Pharmaceutical Products

The pharmaceutical industry employs a vast number of different solvents during its manufacturing process of drug products. Some of these solvents pose a potential health hazard if they remain in the finished product. To ensure the safety of the general public, methodology and guidelines for acceptable levels of these toxic solvents were established by the United States Pharmacopeia (USP).

The guidelines were first published in Chemical Tests and Assays, General Chapter ⟨467⟩ "Organic Volatile Impurities," pp. 2395-2397; Method I, from the third supplement to Volume XXII of the USP 1990. The six solvents specified in this method are listed in Table 1. The method outlines recommendations for sample preparation and final analysis. It also outlines several different sample preparation procedures to cover the wide variety of sample matrices. Most of these procedures involve the extraction or dissolution of the pharmaceutical product in water followed by capillary gas chromatography combined with Flame Ionization Detection (FID).

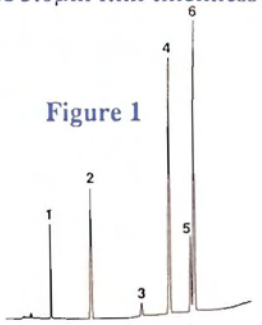
Table 1 - Six Solvents Found in Pharmaceutical Products

Compound	Concentration
Ethylene oxide	10ppm
Methylene chloride	100ppm
Chloroform	50ppm
Benzene	100ppm
Trichloroethylene	100ppm
1,4-dioxane	100ppm

The capillary column recommended in the USP method is a 30m, 0.53mm ID fused silica capillary column coated with a 5.0µm chemically cross-linked 5% phenylmethylsilicone stationary phase. Figure 1 shows the analysis of these six solvents on a 30m, 0.53mm ID, 5.0µm Rt_x-5 column. This column produces baseline resolution of all the components except for trichloroethylene and 1,4-dioxane which are approximately 95% resolved.

A 3.0µm film thickness promotes better resolution than a 5.0µm.

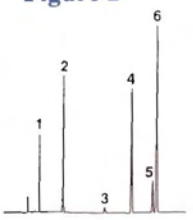
Figure 1



1. Ethylene oxide
2. Methylene chloride
3. Chloroform
4. Benzene
5. Trichloroethylene
6. 1,4-dioxane

30m, 0.53mm ID, 5.0µm Rt_x-5 (cat.# 10279)

Figure 2



30m, 0.53mm ID, 3.0µm Rt_x-5 (cat.# 10285)

1.0µl direct injection. Concentration 100ppm.
Oven temp.: 35°C (hold 5 min.) to 175°C @ 8°C/min. to 260°C @ 35°C/min. (hold 16 min.)
Inj. & det. temp.: 260°C
Carrier gas: helium
Linear velocity: 35cm/sec.
FID sensitivity: 2 x 10^{11} AFS

Understanding the various column parameters is important in selecting the proper column for a particular analysis. Column length effects both analysis time and separation of sample components. Generally, 30-meter columns are chosen as a compromise between fast analysis time and adequate resolution. Column diameter effects sample capacity and also dictates the type of instrumentation that is required. The 0.53mm ID columns exhibit excellent sample capacity and are easily adapted to packed or capillary column instruments. Film thickness also effects sample capacity as well as analysis time. In general, thin films are used when the majority of the sample contains high molecular weight components and thick films are used if the sample contains low molecular weight species.

Since this analysis is comprised primarily of six relatively low boiling solvents, a 30-meter column was an ideal choice. Because this method will be used in many different labs with different types of instrumentation, a 0.53mm ID column was chosen for its versatility. Very thick film columns, such as the 5.0µm column recommended in this method, are normally used for compounds that boil very close to room temperature. Several of these solvents in this method have boiling points above 80°C. These solvents are retained too long by the thick film resulting in longer analysis times and some loss in resolution. We have determined that reducing the film thickness to 3.0µm improves resolution of trichloroethylene and 1,4-dioxane and shortens the analysis time by approximately two minutes. Figure 2 shows the analysis of these six solvents on a 30m, 0.53mm ID, 3.0µm Rt_x-5 column.

Confirmational Analyses

It is not unusual, in many cases, for different sample components to have similar GC retention times. This can lead to misidentification and false positive results. In order to improve the accuracy of the analysis and avoid misidentifications, the use of a confirmational column is recommended. Several different columns were evaluated as potential confirmational columns. The two columns that exhibit the best potential as confirmational columns are either a 30m, 0.53mm ID, 3.0µm Rt_x-502.2 or a 30m, 0.53mm ID, 3.0µm Rt_x-1701. Both columns produce baseline separation of all

Standards Spotlight

Chemical Standards for 500 Series Methods

Restek is pleased to announce the continued expansion of our high quality environmental standards. These mixtures are manufactured in strict compliance with guidelines established by the EPA in their Contract Laboratory Program (CLP). Although these mixtures are NOT intended for use with the CLP program, the requirements specified in the 3/90 Statement of Work are the most stringent published by the EPA in any existing protocol.

501 Trihalomethane Mix
bromodichloromethane
dibromochloromethane
chloroform
bromoform
200µg/ml ea. in 1ml purge & trap grade methanol
Cat.# 30036 $25 ea.
30036-500 $40 ea. w/data pack
30136 $225 10pk. w/data pack

DW-VOC Mix #1
These compounds are currently REGULATED under the Safe Drinking Water Act. Although these materials are found in several additional EPA methods, laboratories may choose to monitor these compounds only.

Benzene	carbon tetrachloride
1,4-dichlorobenzene	1,2-dichloroethane
1,1-dichloroethene	1,1,1-trichloroethane
trichloroethene	vinyl chloride

200µg/ml ea. in 1ml purge & trap grade methanol
Cat.# 30037 $25ea.
30037-500 $45 ea. w/data pack
30137 $225 10pk. w/data pack

DW-VOC Mix #2
Regulations to monitor these compounds were promulgated by the EPA in the Federal Register, Volume 56, No. 20, January 30, 1991.

1,2-dichlorobenzene	cis-1,2-dichloroethene
trans-1,2-dichloroethene	1,2-dichloropropane
chlorobenzene	styrene
tetrachloroethene	toluene
ethylbenzene	o-Xylene
m-Xylene	p-Xylene

200µg/ml ea. in 1ml purge & trap grade methanol
Cat.# 30038 $25 ea.
30038-500 $55 ea. w/data pack
30138 $225 10pk. w/data pack

DW-VOC Kit
Contains 1ml each of the following regulated and promulgated volatile compound mixtures:
30036 501 Trihalomethane mix
30037 DW-VOC Mix #1
30038 DW-VOC Mix #2
Cat.# 30039 $60ea.
30039-500 $115ea. w/data pack

504 EDB/DBCP Mix
1,2-dibromoethane
1,2-dibromo-3-chloropropane
200µg/ml each in 1ml purge & trap grade methanol
Cat.# 30034 $25 ea.
30034-500 $40 ea. w/data pack
30134 $225 10pk. w/data pack

Additional Surrogates & Internal Standards for EPA Methods

α,α,α-trifluorotoluene
2000µg/ml in 1ml purge & trap grade methanol
Cat.# 30048 $25ea.
30048-500 $35 ea. w/data pack
30148 $225 10pk. w/data pack

1,2-dichlorobenzene-d4
2000µg/ml in 1ml purge & trap grade methanol
Cat.# 30049 $25 ea.
30049-500 $35 ea. w/data pack
30149 $225 10pk. w/data pack

Using Method 3500 listed in SW-846?

EPA Method 3500, listed in SW-846, requires the use of an acid matrix spike solution at a concentration of 200µg/ml. In the past, laboratories following this procedure could use our CLP acid matrix spike mix (cat.# 31005) to meet this requirement. In March, the EPA modified the 3/90 Superfund Statement of Work requiring acid matrix spikes at 150µg/ml. Restek changed the concentration of the CLP acid matrix spike mix (cat.# 31005) to 1500µg/ml to follow the new protocol. However, we are now offering an acid matrix spike mix prepared specifically for laboratories following the SW-846 procedures. This mixture is available from stock, complete with certificate of analysis and data packages.

Acid Matrix Spike Mix
Phenol
Pentachlorophenol
4-chloro-3-methylphenol
2-chlorophenol
4-nitrophenol
2000µg/ml ea. in methanol, 1ml per ampule
Cat.# 31014 $25 ea.
31014-500 $45 ea. w/data pack
31114 $225 10pk. w/data pack

Get Reliable GPC Calibrations!

Over the past several months we have had numerous customers request a CLP GPC Calibration Standard. In response to these requests, we are pleased to announce the availability of a GPC calibration standard for the CLP program. This mixture is offered as a qualitative standard that is used to verify resolution criteria of the GPC column and determine dump/collect times for sample clean-up. This mix is available in two convenient package sizes. Because this is a qualitative standard, a certificate of analysis and data packages are not available.

CLP GPC Calibration Mix
Dissolved in methylene chloride at the concentrations listed.

Corn oil	250mg/ml
Bis(2-ethylhexyl)phthalate	10mg/ml
Methoxychlor	2.0mg/ml
Perylene	0.2mg/ml
Sulfur	0.8mg/ml

1ml per ampule. Yields 10ml working solution.
Cat.# 32019 $15 ea.
32119 $135 10pk.
5ml per ampule. Yields 50ml working solution.
Cat.# 32023 $30 ea.
32123 $270 10pk.

Custom package sizes available upon request.

used to increase each column's resolving power and sample handling capacity. To minimize analysis time, high carrier gas flow rates using hydrogen as the carrier gas were employed. Hydrogen was chosen as the carrier gas to minimize the loss in column efficiency when run with high flow rates.* Results showed that all four columns have partial or complete co-elutions of some compounds. There are two co-elutions on the Rt_x-1 column (methyl silicone). Methanol and acetaldehyde are completely unresolved and acetone and isopropanol are only 40% resolved. The remaining compounds elute very close to one another because of the low polarity of the methyl silicone stationary phase. The Stabilwax column (polyethylene glycol) shows better selectivity than the Rt_x-1 column because of the increased stationary phase polarity, but ethanol and isopropanol co-elute with one another. The Rt_x-200 column (trifluoropropylmethyl silicone) had excellent resolution for all of the alcohols due to its selectivity for compounds with lone pair electrons. The only co-elution was with ethanol and acetaldehyde. When compared to the other phases, the Rt_x-1701 column (cyanopropylphenyl methyl silicone) had the best overall resolution. There were no complete co-elutions, with acetone and isopropanol being approximately 40% resolved.

In choosing two columns for use in a dual column system for blood alcohol analysis, overall resolution and co-elutions with ethanol are the primary considerations. Although ethanol is completely resolved on the Rt_x-1 column, it was excluded because of the two co-eluting pairs and low resolving power. The Stabilwax column was dropped from consideration because ethanol co-eluted with isopropanol, a potential contaminant from skin preparation prior to collecting blood samples. Based upon the chromatography for the two remaining phases, the Rt_x-1701 column would be the choice for the screening column. It provides the best resolution of all compounds. The Rt_x-200 would then be the choice for the confirmational column. Because of the unique selectivity of the trifluoropropyl phase, the elution order for acetone and acetaldehyde relative to the other alcohols is dramatically altered. This shift in retention time and elution order provides the mechanism for confirmation of identity of any volatile intoxicants present. Acetone and isopropanol, which had been unresolved on the Rt_x-1701, are now completely resolved from one another. The co-elution of ethanol and acetaldehyde on the Rt_x-200 is a minor problem since clinically significant concentrations of acetaldehyde are rarely encountered.

". . . the Rt_x-1701 column would be the choice for the screening column. . . . The Rt_x-200 would then be the choice for the confirmational column."

The Rt_x-1701 and the Rt_x-200 were then installed into an instrument containing a packed column injection port and two FIDs. Installation of both columns into the injection port was achieved by using a "Y" Press-Tight®connector and a 10cm length of 0.53mm ID deactivated guard column tubing. A 1mm ID Uniliner®was used to adapt the packed column port for use with wide bore capillary columns. Because both columns were of the same length and internal diameter, carrier gas flow and samples to be analyzed were split evenly between the two columns. The detector ends of each column were then installed in separate FIDs. All other analytical parameters were the same. The chromatography achieved with this dual column system was identical to that obtained when using a single column.

Rt_x-1701 and Rt_x-200 columns can be used in a dual column configuration that provides rapid detection and quantitation for ethanol and associated volatile compounds in biological samples.

Product Listing

Phase & Composition	length	ID (mm)	df	cat.#	price
Rt_x-1701	30m	0.53	3.0	12085	$445
Rt_x-200	30m	0.53	3.0	15085	$475
Guard Column	5m	0.53	---	10045	$60
6-pack	5m	0.53	---	10045-600	$300
"Y" Press-Tight connector	---	---	---	20405	$55
3-pack	---	---	---	20406	$145

* longer analysis times and some loss of resolution may occur with helium as the carrier gas

Correction

In the last issue of The Restek Advantage (November 1991, Vol. 2 No. 5), there were some peak misidentifications in Figure 5 on page 4. Corrections are shown below:

As shown in newsletter		Correction
Peak#	Name	
3	Carbon tetrachloride	Tetrachloroethylene
6	n-Propyl nitrate	Bromoform
7	Methylene bromide	n-Propyl nitrate

Why Order Restek Standards?

- High concentration for maximum value
- Prepared for latest EPA protocols
- Complete data package available
- Packaged for laboratory convenience
- In stock for immediate delivery
- Bulk quantities available
- Custom orders welcomed
- Restek's 100% Satisfaction Guarantee

Restek manufactures its own line of high quality chemical standards for laboratories performing environmental analyses.

Most chemical standards are produced at concentrations from 1000µg/ml to 2000µg/ml to insure that an adequate volume of working solution can be prepared from a single ampule. Also, many individual mixtures can be combined to achieve the working calibration levels required by EPA protocols.

Our line of standards for the EPA Con-tract Laboratory Program (CLP) meet or exceed the quality specifications in the latest statement of work. The newest surrogates and target compounds that the EPA requires are included. Complete volatile, semi-volatile, and pesticide kits are available. A data pack that conforms to the EPA's documentation requirements for commercially obtained standards can be purchased with each mixture. These data packs have been accepted by EPA auditors in many regions as adequate documentation for commercially produced standards.

The difference between Restek chemical standards and those produced by other manufacturers

Restek provides all of the production and QA testing documentation for every lot of chemical standards purchased. Restek promotes a total quality program that begins before the raw materials arrive at our facility. Mixtures are designed for customer convenience and stability over long periods of time. Only the highest purity raw materials are purchased from reputable firms. These raw materials are extensively quality tested prior to their use in any mixture. Every compound is analyzed for purity and identity by melting point/refractive index, GC/FID, and GC/MS using high resolution fused silica capillary columns. Additionally, pesticides are analyzed by GC/ECD and the volatile gases are analyzed by GC/ELCD. Most compounds are 98% pure or greater. All raw materials and finished products are stored at reduced temperatures to increase shelf-life.

Our analytical balance calibrations are verified at six mass levels daily using ASTM class 1 weights. Two chemists independently prepare identical mixtures for each product. These mixtures are then analyzed in triplicate using high resolution fused silica capillary columns. The results from both lots are statistically compared to each other using a comparison of means and the Student's *t*-test. The criteria are established at the 95% confidence level with two degrees of freedom. A certificate of analysis is provided with each ampule. This certificate lists each component's exact gravimetric composition, and shows a typical chromatogram from that product.

Restek's Data Packs

The data packs provide customers with the necessary information for EPA audit documentation. The latest Superfund statement of work requires laboratories to have quality, detailed documentation on file for commercially purchased standards. These data packs include the following: melting point measurements for solids and refractive index for liquids, GC/FID purity analyses, GC/MS identity confirmation, copies of all lot sheets showing the exact gravimetric weights, copies of the final mixture's actual QA chromatograms, plus the statistical comparison data. For laboratories not performing Superfund analyses, the data pack can be used by their internal QA departments to review the quality of the standards used.

User Friendly Packaging

Restek's chemical standards are packaged with the laboratory's convenience in mind. First, every ampule is deactivated before being filled and sealed. There is no chance that active analytes can adsorb to the glass walls. We package every ampule in a flex square that contains THE AMPULE CRACKER™ to safely open the ampule, a deactivated vial to place unused portions of the mixture in, and an extra label. Several mixtures are offered in varying concentration and package sizes for different size laboratories.

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Custom Standards

Need something special? Call us at 800-356-1688 for a custom mixture quotation.

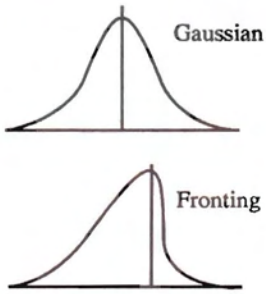
Hints for the Capillary Chromatographer

Sample Capacity and Column Overload

What is column overload?

Column overload occurs when the amount of sample injected exceeds the column's capacity for that component. Overload is normally observed as a fronting, non-gaussian peak shape (Figure 1). A column's capacity is a function of several parameters including the column's internal diameter (ID), its film thickness (df), the solubility of the compound in the column's stationary phase, and capacity factor (*k*).

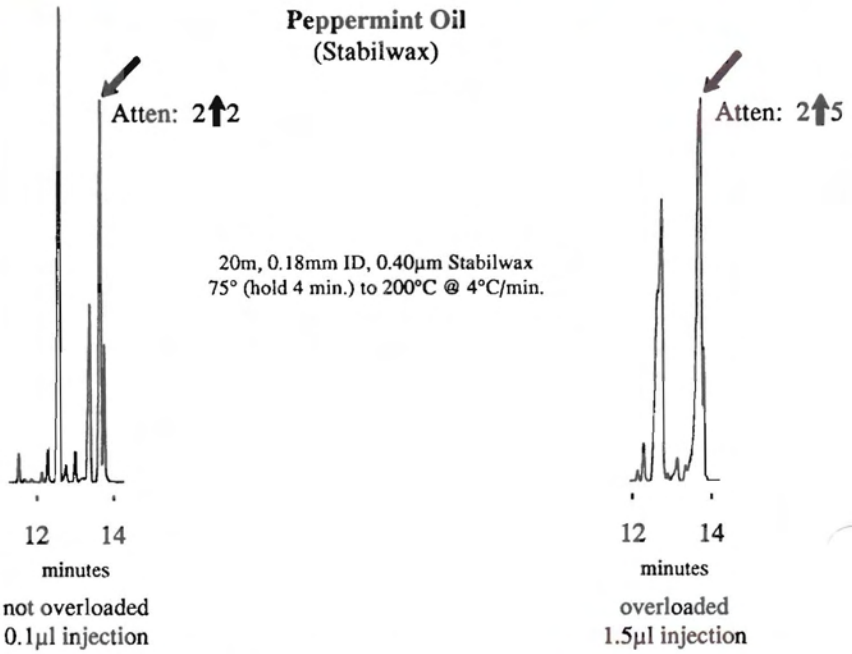
Figure 1 - Normal Gaussian vs. Overloaded Fronting Peak Shapes



Why is it important not to exceed a column's capacity?

Capillary columns have much lower sample capacities than packed columns, therefore, it is extremely important to optimize the amount of sample injected. When sample capacity is exceeded, peak symmetry is lost and resolution is affected. Because the peak shape will be much broader, resolution between two closely eluting peaks can be lost. Figure 2 shows the loss of peak symmetry and resolution in the analysis of peppermint oil on a Stabilwax column. In the first chromatogram, 0.1µl of neat peppermint oil was injected. At these low concentrations, very good resolution between the menthyl acetate, neo-menthol,

Figure 2 - Minimize the amount of sample injected to maximize resolution.



β-caryophyllene, and terpinene-4-ol is obtained. In the second chromatogram, 1.5µl of neat peppermint oil was injected. Because the sample concentration exceeded the column's capacity, a significant loss in resolution occurred.

How can overload be prevented?

Two choices are available to prevent overload:

- ▼ reduce the sample concentration reaching the column
- ▼ choose a column and run conditions that will allow greater sample capacity

To reduce the sample concentration reaching the column, the sample components can be diluted by increasing the split ratio, diluting with additional solvent, or by introducing a smaller amount.

How does column ID affect sample capacity?

As the column ID increases so does sample capacity. Table 1 shows typical column capacity for several different diameter columns. Figure 3 compares sample capacity on 0.25 and 0.53mm ID columns. Four hydrocarbons (heptane, octane, nonane, and decane) were analyzed at a concentration of 1000ng on

Table 1

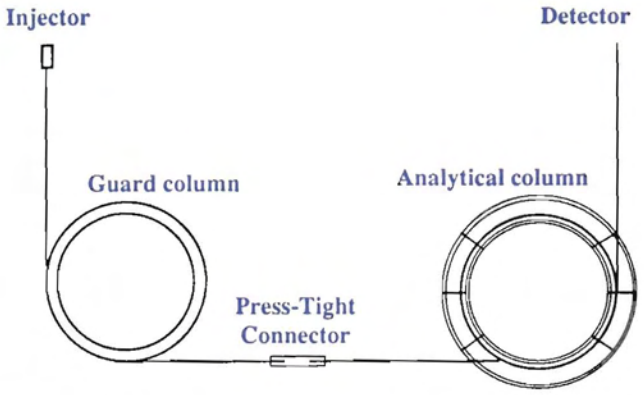
Column ID	0.18mm	0.25mm	0.32mm	0.53mm
Sample Capacity	<50ng	50-100ng	400-500ng	1000-2000ng

Hints for the Capillary Chromatographer

The Benefits of Guard Columns for Capillary Gas Chromatography

The use of guard columns has been commonplace in high performance liquid chromatography for many years. Their use as safeguards to protect the analytical column from highly retentive compounds and particles is well understood. It has only been in the past few years that the benefits of guard columns have been associated with capillary gas chromatography. Although guard columns prolong the life of capillary columns and protect them from sample contamination, they are not widely used in many laboratories. Understanding the basics of guard columns helps to dispel confusion and apprehension about their use.

Figure 1 - A Guard Column Connected to an Analytical Column.



What is a guard column?

A guard column for capillary chromatography is a short length of deactivated, uncoated fused silica tubing that is placed between the injection port and the analytical column. Figure 1 shows a diagram of a guard column connected to an analytical column.

What advantages do guard columns offer?

Prolong column lifetime.

A guard column protects and prolongs the lifetime of an analytical column in several ways. It traps non-volatile residues and prevents them from collecting at the head of the analytical column. These non-volatile residues may be very high molecular weight organic compounds, inorganic salts, or particulate materials. If these contaminants enter the analytical column, they can cause adsorption of active compounds and loss of resolution. When this contamination begins to affect sample analysis, a small section of the analytical column must be removed to restore proper performance. However, each time a section of the column is removed, retention times change, some resolution is lost, and column length is decreased, eventually resulting in a useless analytical column. By trapping this contamination in the guard column, the analytical column remains the same length and stays cleaner longer.

Decrease maintenance requirements.

Since there is no stationary phase on the guard column, the amount of time the sample spends in it is minimized. This reduces the interaction between sample components and the contamination from non-volatile residue. Therefore, guard columns allow more injections to be made before residue interferes with analytical results.

Improve resolution.

Many analysts are reluctant to use guard columns because they believe that they will lose resolution. In fact, guard columns aid in focussing the components by decreasing aerosol formation and actually increasing separation efficiency. The guard column acts as a retention gap to help focus the sample at the head of the column. When a sample is injected, it first exists as vapor and aerosol. Without a guard column the vapor begins to partition in and out of the column's stationary phase. The aerosol portion of the sample does not partition in the phase and moves out ahead of the vaporized sample. This results in broader, less efficient peaks and, in extreme cases, can cause split peaks. Since a guard column is not coated with stationary phase, there is no interaction with the vaporized sample or the aerosol. They move along together in a tighter band. The aerosol vaporizes in the guard column so that when the sample reaches the coated column it is completely vaporized. This produces sharper, more efficient peaks, as shown in Figure 2. Table 1 shows the results of analyzing 2,6-dimethylphenol on a 30 meter, 0.53mm ID, 1.0µm Stabilwax®column with and without a guard column. The efficiency of the 2,6-dimethylphenol peak was measured in each case and the results show a 3.1% increase in efficiency with the guard column.

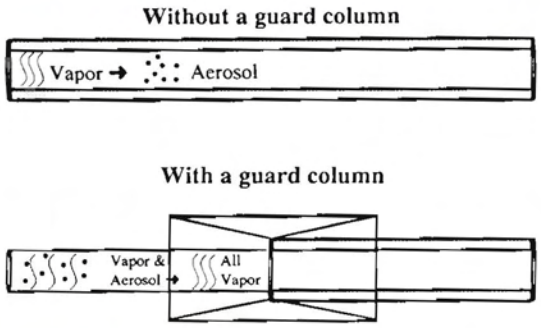
Table 1 - Column Efficiency Data
(1µl split injection of 2,6-dimethylphenol)

30m, 0.53mm ID, 1.0µm Stabilwax

Without guard column	With guard column
Total plates = 51500	Total plates = 53100
Plates/meter = 1716	Plates/meter = 1770

3.1% increase in plates

Figure 2 - Guard columns increase separation efficiency because the aerosol completely vaporizes before it reaches the coated column.



How is the guard column connected to the analytical column?

A Press-Tight®Connector is the simplest and most common way to connect a guard column to the analytical column. These connectors do not require ferrules and work on the principle of radial compression. Once heated, the polyimide on the outside of the tubing bonds to the inside of the connector, making a permanent, leak-free seal. There are several key steps to ensure a leak-free seal. First, cut the column ends squarely with a device designed for cutting fused silica tubing, such as a sapphire scribe or a ceramic scoring wafer. (Pointed scoring devices are not recommended.) Second, clean and lubricate the tubing by wiping the ends with a tissue moistened with methanol or deionized water. Next, firmly insert the tubing into the connector taper and check for leaks. If no leaks are found, bond the tubing to the connector by heating it to 200°C.

When should a guard column be replaced?

As the guard column becomes contaminated with non-volatile residue, the performance of the entire chromatographic system will begin to deteriorate. This is normally exhibited as a drastic decrease in the response of active compounds. Figure 3 shows the analysis of phenols on an Rt_x-5 with a guard column. The response of all of the phenols is excellent. Figure 3 also shows the same analysis after repeated injections of a sample containing significant quantities of non-volatile residue. The reduction in the response of 2,4-dinitrophenol and pentachlorophenol indicate that the guard column is contaminated and must be replaced.

How often must a guard column be replaced?

The life expectancy of a guard column depends on the length of the guard column, the amount of non-volatile residue in the samples, and the number of samples run. When analyzing dirty samples, the guard column becomes contaminated quickly. Normally, contamination deposits in the first .5 to .8 meters of the guard column. If a short guard column (1-meter or less) is used, it must be completely replaced when it becomes overly contaminated. If a longer guard column (5-meters) is used, the contaminated section can be removed without re-connecting the analytical column.

How long should a guard column be?

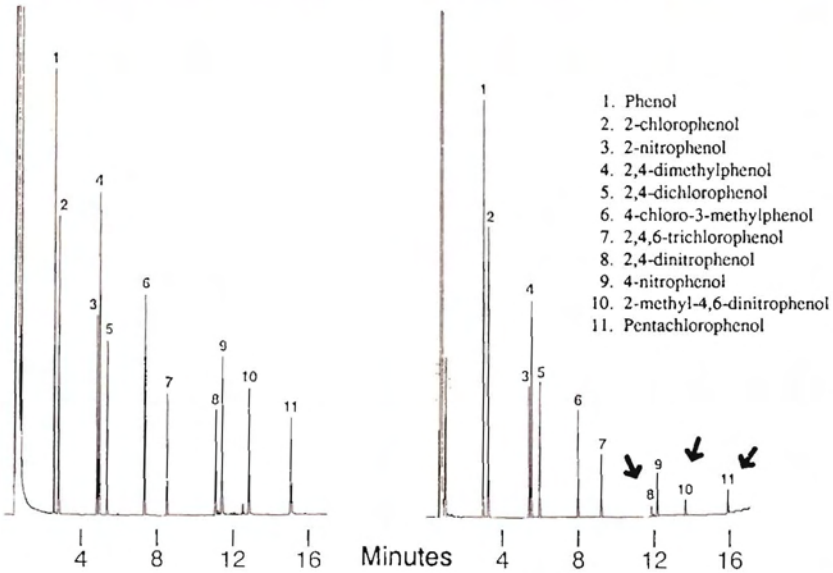
A guard column should be long enough to keep non-volatile residue from entering the column, but short enough so that the analysis time is not significantly increased. Five meter guard columns are more cost effective, reduce the frustrations of making the connection between the guard column and analytical column, and are preferred by most analysts over 1-meter guard columns. If a very long guard column (>10-meters) is used, the residence time of the sample components increases, resulting in longer analysis times.

Guard columns help prolong the life expectancy of capillary columns and are an excellent and economical alternative to column replacement. Analysts working with dirty samples find that the use of guard columns significantly reduces column replacement costs and time lost in troubleshooting column contamination problems. ■

Figure 3

Phenols show excellent response on an Rt_x-5 with a newly installed guard column.

Reduced response factors show guard column contamination.

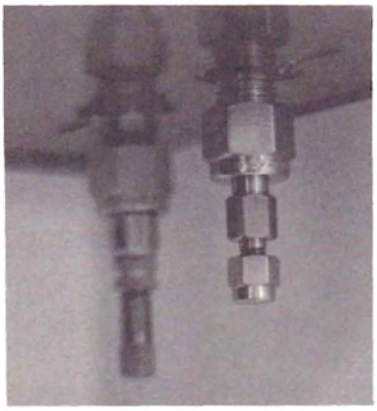


15m, 0.32mm ID, 1.0µm Rtx-5 (cat.# 10251)
0.2µl split injection of phenols.

Oven temp.: 80°C to 290°C @ 8°C/min.
Inj. & det. temp.: 310°C

Carrier gas: hydrogen
Linear velocity: 45cm/sec.
FID sensitivity: 8×10^{-11} AFS
Split ratio: 9:1

HP FID/NPD Detector Adaptor Fitting



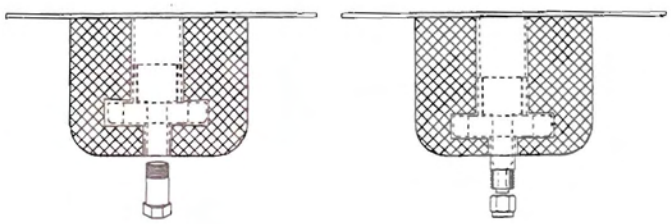
After wrestling with HP's detector adaptor fitting, Restek's chemists decided to engineer an improved version. First we replaced the awkward column fitting with a 1/16" Swagelok® type nut that uses standard graphite or Vespel®/graphite ferrules. Then we shortened the fitting to make it more compact and improved the wrench pad to prevent it from turning when installing a capillary column. The end result? An easy-to-use, sturdy stainless steel fitting that makes capillary installation easy. All the parts needed for installation (1/4"SS nut, 1/4" Vespel ferrule, 1/16"SS nut, and 0.4mm ID graphite ferrule) are included. Replaces HP Part numbers 19244-80610 and 5921-21170.

cat.# 20884, $55 per kit

HP Capillary Inlet Adaptor Fitting

HP fitting is hard to see when inserting capillary column

Restek's fitting makes column insertion easy



HP 5890 capillary inlet fittings are frustrating because it is difficult to see where the column is inserted. Restek's chemists engineered a simplified HP 5890 capillary inlet fitting that uses 1/16" Swagelok®-type nuts and standard graphite or Vespel®/graphite ferrules. We carefully machined the threads and matched the stainless steel types to eliminate seizing onto the injector body. We also paid careful attention to the fitting depth to keep inlet insertion distances exactly the same and designed it to use the same type of inlet disc. The end result? An easy-to-use, sturdy stainless steel fitting that makes capillary column installation easy. All the parts needed for installation (the inlet disc, washer, 1/16" SS nut, and 0.4mm ID graphite ferrule) are included. Replaces HP part numbers 18740-20800, 05921-21170, and 18740-20880.

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- Produces square, even cuts
- Eliminates distortion of the tubing
- Replaceable cutting wheel

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estek 1/16" Tubing Cutter: cat.# 20192, $75 each
Replacement Wheels: cat.# 20185, $10/3-pack

Standard Capillary Ferrules

Restek has graphite and 60% Vespel®/40% graphite ferrules to use with capillary columns. The graphite ferrules are made from highly-compressed ribbon that will not crack or split under torque. The 60% Vespel/40% graphite ferrules are designed to seal easily with minimal torque and are re-useable. Both are stable to 400°C.

Ferrule ID	Fits Column ID	Graphite			Vespel/graphite		
		cat.#	price	quan.	cat.#	price	quan.
0.4mm	0.25	20200	$25	10-pk	20211	$30	10-pk
0.5mm	0.32	20201	$25	10-pk	20212	$30	10-pk
0.8mm	0.53	20202	$25	10-pk	20213	$30	10-pk
0.4mm	0.25	20227	$100	50-pk	20229	$120	50-pk
0.5mm	0.32	---	---	---	20231	$120	50-pk
0.8mm	0.53	20224	$100	50-pk	20230	$120	50-pk

News from Restek

Jazz it up with the Restek Wizards

Don't miss the Restek Wizards at Pittsburgh Conference 1992 in New Orleans. We will be presenting nine technical papers and four posters at this year's show (please check insert for a complete listing of times and topics).

We are anticipating a terrific conference and are looking forward to seeing everyone there. Please visit our booth (#2612) and bring along the coupon insert to receive a free wizard fanny pack. Eighteen of our technical support chemists will be there to serve your chromatographic needs.

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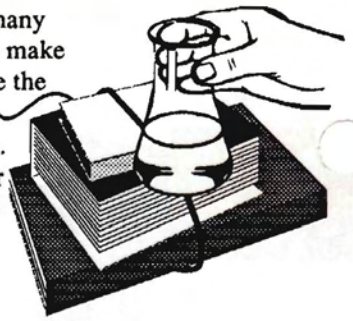
With shrinking budgets and research funds drying up, many colleges and universities have found it more difficult to make ends meet. This often means not being able to purchase the equipment necessary to educate the chromatographers of tomorrow or to complete important research projects. Restek is working to stretch your budgets a little further with the development of a new Educational Discount Program. This program is available to any accredited college or university. For more information contact Restek's Marketing Department at 800-356-1688.



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Industrial Solvent Analysis Simplified

Retention index table helps analysts choose the most appropriate capillary column for a given solvent matrix

Manufacturers generate residual solvents during the manufacturing process of their product. Today it is beneficial to accurately and quickly monitor these components. Capillary GC has become more popular for solvent analysis than traditional packed column GC since it provides better resolution, faster analysis times, and greater inertness. With the growing number of solvents in common use, it has become increasingly difficult to select the most appropriate capillary column polarity. By having a table of retention indices, column selection can be made with the confidence that all components will be separated on the stationary phase chosen. Columns of different polarity can be used for confirmational analysis to improve identification and quantitation.

We analyzed a wide range of solvents and determined their elution orders and retention indices on three different polarity capillary columns. The stationary phases evaluated were a non-polar, 100% dimethyl polysiloxane (Rt_x-1), an intermediate polarity, methyl trifluoropropyl polysiloxane (Rt_x-200), and a polar, polyethylene glycol (Stabilwax). The solvents analyzed also spanned a wide range of polarity. Various alcohols, ketones, aromatic hydrocarbons, and halogenated compounds were included to cover a range of typical industrial solvents. Seven saturated hydrocarbons ranging from pentane to nonadecane were added as retention index reference standards.

Figure 1 - Equation for calculating retention indices under temperature programmed conditions

$$Ia=100N + 100n(t_{Ra}-t_{RN})/(t_{R(N+n)}-t_{RN})$$

where: N = the carbon number of the lower hydrocarbon
n = the difference in carbon numbers that bracket the component of interest
t_{Ra} = the retention times of the component of interest
t_{RN} = the retention time of the saturated hydrocarbon eluting before the component of interest
$t_{R(N+n)}$ = the retention time of the saturated hydrocarbons eluting after the component of interest

As an example, N-methyl-2-pyrrolidone has a retention index of 1004 on an Rtx-1 column, meaning that it elutes approximately halfway between nonane (nC9) and undecane (nC11).

Pages 2 and 3 list the absolute retention times and retention indices of 120 common industrial solvents. The absolute retention times show the elution order of the solvents under the run conditions used in this study. Also included are retention indices for each solvent, on all three columns, in the event other run conditions are chosen.

The retention index system, as defined by Kováts (1), is a measure of relative retention times referenced to a series of saturated hydrocarbons. Normally, retention indices are determined under isothermal conditions. However, since the vast majority of analyses are performed under temperature programmed conditions, a modified retention index equation as defined by Van den Dool and Kratz (2) was chosen for this study. Figure 1 shows this equation and an example of how the equation was applied to this study.

in this issue...

Solvent	BP	MW	Rtx-1 Retention Index	Rtx-1 Retention Time	Rtx-200 Retention Index	Rtx-200 Retention Time	Stabilwax Retention Index	Stabilwax Retention Time
1,1,2-trichloroethane	115	132	747	15.331	867	14.632	1287	20.908
1,2,3,4-tetrahydronaphthalene	207	132	1168	32.49	1274	28.803	1576	29.571
1,2,4-trimethylbenzene	168	120	993	26.033	1083	22.723	1312	21.745
1,2-dichlorobenzene	175	146	1031	27.499	1174	25.667	1482	26.934
1,2-dichloropropane	96	112	675	12.164	802	12.053	1059	13.031
1,2-xylene	145	106	886	21.728	991	19.296	1212	18.335
1,3-xylene	139	106	863	20.685	954	17.943	1166	16.72
1,4-dichlorobutane	163	126	881	21.52	1074	22.389	1366	23.404
1,4-xylene	137	106	865	20.775	952	17.86	1160	16.501
1-heptanol	176	116	954	24.481	1078	22.509	1472	26.642
1-nonanol	211.5	144	1156	32.096	1288	29.254	1677	32.314
2,2,2-trifluoroethanol	74	100	----	4.028	534	4.302	1199	17.862
2,2-dimethyl-1-pentanol	121	116	869	20.971	986	19.136	1341	22.633
2,4-dimethyl-2-pentanol	133.1	116	780	16.871	895	15.74	1146	16.03
2,4-dimethyl-3-pentanone	124.5	114	782	16.928	969	18.505	1005	11.18
2,6-dimethyl-4-heptanone	169	142	961	24.759	1163	25.317	1194	17.686
2-bromopentane	117	150	551	7.142	879	15.105	980	10.32
2-butanol	98	74	571	7.942	691	7.81	1028	11.976
2-buten-1-ol	121.2	72	641	10.8	765	10.573	1235	19.107
2-chloropropane	36	79	----	4.925	557	4.818	695	3.589
2-chloro-2-methyl-butane	84	106	649	11.091	754	10.138	794	5.472
2-decanol	211	158	1189	33.2	1318	30.094	1631	31.069
2-decanone	210	156	1177	32.783	1429	33.132	1518	27.966
2-dodecanone	246.5	184	1382	39.339	1642	38.557	1730	33.686
2-ethyl-1-butanol	146	102	827	19.02	941	17.465	1323	22.068
2-ethyl-1-hexanol	184.5	130	1016	26.926	1136	24.467	1502	27.531
2-heptanol	161	116	884	21.643	1008	19.923	1329	22.269
2-heptanone	150	114	870	21.005	1101	23.358	1205	18.066
2-methoxyethanol (methyl Cellosolve™)	124	76	600	9.133	768	10.687	1197	17.788
2-methyl-1-pentanol	148	102	820	18.679	934	17.206	1316	21.861
2-methyl-3-buten-2-ol	97.5	86	582	8.407	704	8.177	1046	12.568
2-methyl-3-pentanol	127	102	759	15.885	864	14.505	1171	16.889
2-nonanol	193.5	144	1086	29.643	1214	26.911	1529	28.278
2-nonanone	195.3	142	1073	29.162	1319	30.122	1415	24.901
2-octanol	177.5	130	988	25.812	1108	23.602	1432	25.4
2-octanone	173	128	974	25.278	1213	26.899	1308	21.626
2-phenoxyethanol	245	138	1199	33.53	1447	33.606	----	44.134
2-propen-1-ol/allyl alcohol	97	58	521	5.941	608	5.955	1126	15.347
2-(2-butoxyethoxy)ethanol	231	162	1167	32.438	1373	31.596	1821	35.907
3-buten-1-ol	113	72	606	9.36	735	9.385	1186	17.411
3-heptanone	150	114	867	20.869	1077	22.481	1175	17.023
3-hexanol	135	102	783	16.976	891	15.571	1212	18.33
3-methyl-1-butanol/isoamyl alcohol	130	88	719	14.026	833	13.274	1227	18.826
3-methyl-2-buten-1-ol	140	86	758	15.86	889	15.514	1337	22.492
3-methyl-3-pentanol	122.4	102	740	15.006	852	14.04	1125	15.305
3-pentanone	102	86	670	11.975	875	14.963	984	10.455
adiponitrile	295	108	1099	30.16	1837	42.933	----	47.357
allyl acetate	103.5	100	676	12.205	846	13.814	1041	12.418
α- methyl styrene	169	118	977	25.382	1075	22.419	1362	23.271
α-methylbenzyl alcohol	204	122	1043	27.964	1219	27.061	1848	36.552
α-pinene	155.5	136	944	24.093	965	18.364	1026	11.911
benzene	80	78	642	10.843	733	9.308	949	9.246
benzonitrile	188	103	958	24.654	1274	28.8	1655	31.716
benzyl acetate	206	150	1141	31.573	1388	32.024	1769	34.624
benzyl alcohol	205	108	1013	26.811	1135	24.452	----	38.202
butyl ether	142.5	130	877	21.311	912	16.378	966	9.825
chloroacetonitrile	126.5	75	622	10.011	865	14.545	1435	25.489
chlorobenzene	132	112	838	19.524	947	17.665	1242	19.372
chloroform	61.5	118	586	8.567	650	6.89	1032	12.091
cumene/isopropylbenzene	154	120	920	23.155	1004	19.787	1202	17.973

Solvent	BP	MW	Rtx-1 Retention Index	Rtx-1 Retention Time	Rtx-200 Retention Index	Rtx-200 Retention Time	Stabilwax Retention Index	Stabilwax Retention Time
cyclohexane	81	84	654	11.32	661	7.143	716	3.922
cyclopentane	50	70	550	7.093	547	4.592	602	3.223
cyclopentanol	139.5	86	767	16.264	885	15.355	1323	22.077
cyclopentanone	131	84	763	16.07	1030	20.759	1214	18.405
decahydronaphthalene	191	138	1118	30.81	1097	23.242	1180	17.219
dibromomethane	98	174	674	12.115	756	10.244	1192	17.624
dicyclohexylamine	256	181	1440	41.026	1510	35.307	1691	32.702
diethyl phthalate	298	222	1570	44.651	----	46.167	----	48.912
diethylbenzene	182	134	1048	28.16	1123	24.049	1333	22.389
dimethyl phthalate	283.7	194	1428	40.685	1853	43.258	----	47.498
dimethyl sulfoxide	189	78	785	17.088	1248	27.973	1606	30.379
dimethylacetamide	164	87	833	19.318	1223	27.19	1438	25.595
di-iso-propylbenzene	203	162	1152	31.944	1217	27.019	1385	23.967
ethyl acetate	77	88	583	8.46	757	10.267	893	7.418
ethyl decanoate/ethyl caprate	245	200	1378	39.209	1555	36.416	1454	26.084
ethyl propionate	99	102	692	12.867	845	13.773	963	9.746
ethyl trichloroacetate	168	190	970	25.124	1141	24.626	1384	23.946
ethylbenzene	136	106	856	20.348	939	17.396	1150	16.162
furfuryl alcohol/2-furanmethanol	170	98	831	19.186	1001	19.668	1685	32.54
heptadecane	302	240	1700	48.082	1700	39.989	1700	32.956
heptane	98	100	700	13.172	700	8.008	700	3.61
hexanol	156	102	852	20.184	974	18.673	1367	23.419
hexyl acetate	169	144	996	26.138	1175	25.705	1290	21.03
hexylene glycol/2-methyl-2,4-pentanediol	197	118	888	21.83	1136	24.459	1643	31.403
isopropyl acetate	85	102	638	10.67	806	12.2	907	7.804
iso-amyl alcohol	146	130	719	14.026	1038	21.052	1227	18.826
methoxyethoxyethanol	194	120	906	22.626	1128	24.23	1613	30.576
methyl hexanoate	151	130	905	22.584	1069	22.205	1209	18.21
methyl isoamyl ketone/5-methyl-2-hexanone	144	114	840	19.604	1077	22.472	1160	16.525
methylcyclohexane	101	98	725	14.329	735	9.416	746	4.524
methyldecanoate	224	186	1306	37.08	1482	34.582	1617	30.682
m-chlorotoluene	162	126	949	24.302	1071	22.279	1348	22.85
m-cresol	203	108	1048	28.169	1209	26.766	----	42.798
nitrobenzene	210.5	123	1067	28.935	1400	32.329	1799	35.357
nonadecane	330	268	1900	52.766	1900	44.272	1900	37.816
nonanal	198	142	1087	29.69	1310	29.884	1422	25.108
nonane	151	128	900	22.388	900	15.945	900	7.566
N-methyl-2-pyrrolidone	202	99	1004	26.473	1455	33.828	1720	33.43
octane	127	114	803	17.93	799	11.935	764	4.88
o-chlorotoluene	159	126	948	24.272	1057	21.758	1339	22.554
o-cresol	191.5	108	1030	27.49	1180	25.844	----	40.877
pentadecane	270	212	1500	42.798	1500	35.059	1500	27.484
pentane	36	72	500	5.083	500	3.55	500	2.823
propyl acetate	102	102	696	12.991	864	14.503	979	10.288
pyridine	115.5	79	721	14.143	897	15.814	1205	18.081
p-cresol	202	108	1048	28.173	1205	26.637	----	42.592
p-cymene	178	134	1024	27.251	1092	23.034	1296	21.242
p-methoxyphenol	243	124	1184	33.023	1413	32.699	----	49.278
sec-butanol	98	74	571	7.942	691	7.81	1028	11.976
s-tetrachloroethane	147	166	885	21.704	1012	20.078	1535	28.441
tetrachloroethylene	121	164	808	18.146	854	14.104	1032	12.117
toluene	111	92	760	15.917	845	13.775	1056	12.92
trans-1,2-dichloroethylene	48	96	540	6.711	583	5.406	849	6.56
tributyl phosphate	305	266	1617	45.895	----	47.04	----	43.282
trichloroethylene	87	130	685	12.575	747	9.89	1003	11.093
trichlorotrifluoroethane/freon113	47.6	186	519	5.859	550	4.668	585	3.158
tridecane	234	184	1300	36.912	1300	29.617	1300	21.375
triethylbenzene	215	162	1219	34.194	1276	28.868	1488	27.103
undecanal	235	170	1292	36.657	1529	35.782	1636	31.215
undecane	196	156	1100	30.208	1100	23.34	1100	14.436

Solvent Analysis (cont. from page 1)

Figure 2 shows chromatograms from each of the three columns evaluated in this study. The solvents are numbered in their elution order on the Rt_x-1 column. By comparing the numbering sequence of the peaks on the Rt_x-200 and Stabilwax columns, the effects of stationary phase polarity on the elution order of these solvents is very evident. Chromatogram A shows that allyl alcohol, peak 2, elutes very quickly on a non-polar Rt_x-1 column and is not completely separated from the tail of the methylene chloride peak. Chromatogram B shows that on a more polar Rt_x-200 column, the allyl alcohol is completely separated from the methylene chloride. Chromatogram C shows that the allyl alcohol elutes more than 10 minutes later and is easily separated from the methylene chloride peak.

With the growing number of industrial solvents in common use, a reliable analytical approach for choosing the correct polarity capillary column is essential. The data presented is the first part of an extensive study to determine the retention indices of a wide range of solvents on a broad range of capillary column polarities. The retention indices for over 150 additional solvents will be determined on these same three polarity columns and retention indices for all 270 solvents are being determined on several other polarity columns. Our ultimate goal is to provide the most comprehensive database available on solvent retention indices.

References

(1) Kováts, E., Giddings, J.C. and Keller, R.A., Advances in Chromatography, Volume 1, Chapter 7. New York: Marcel Dekker (1965).

(2) Van Den Dool, H. and Kratz, P.D. Journal of Chromatography, Volume 11, pp. 463-471, (1963).

Figure 2 - By changing polarity, difficult solvents can be resolved.

Peak Identification

1. Pentane
2. 2-propen-1-ol (allyl alcohol)
3. Isopropyl acetate
4. Benzene
5. Heptane
6. Cyclopentanone
7. 2-ethyl-1-butanol
8. 1,4-dichlorobutane
9. Nonane
10. Methyl hexanoate
11. 1,2,4-trimethylbenzene
12. 2-ethyl-1-hexanol
13. Undecane
14. Decahydronaphthalene
15. 2-decanol
16. 2-phenoxyethanol
17. Tridecane
18. Pentadecane
19. Heptadecane

Run Parameters

Instrumentation: HP 5890 GC with HP 5971A Mass Selective Detector and FID with HP 7673 Autosampler
effluent splitter: FID/MSD

Columns: 60m, 0.53mm ID, 3.0µm Rt_x-1 (cat.# 10188)
60m, 0.53mm ID, 3.0µm Rt_x-200 (cat.# 15088)
60m, 0.53mm ID, 1.0µm Stabilwax (cat.# 10658)

Conditions: 1.0µl split injection
using a Restek Cyclosplitter®Sleeve (cat.# 20706)

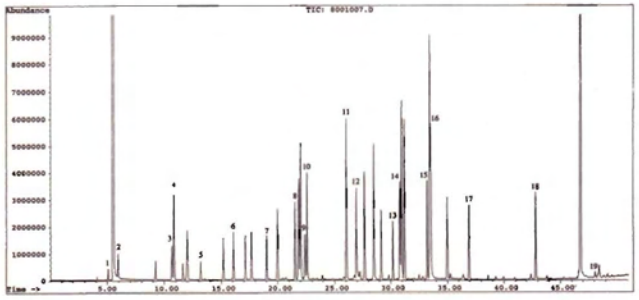
Split ratio: 50:1

Inj. Temp.: 275°C
Det. Temp.: 285°C

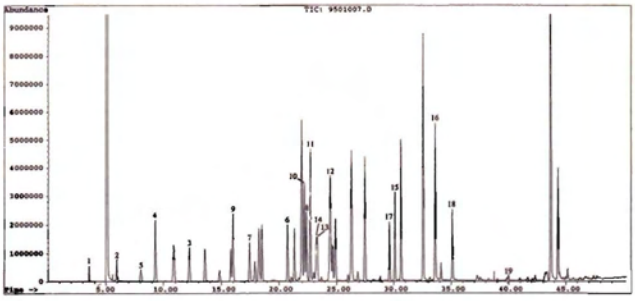
Carrier gas: Helium at approximately 40cm/sec.

Temp. program: 40°C (hold 5 min) to 285°C (250°C for Stabilwax) @ 5°C/min.

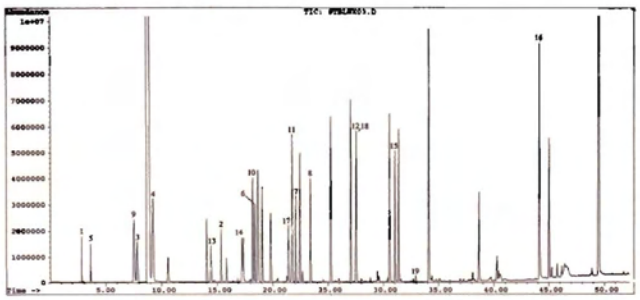
A (Rt_x-1)



B (Rt_x-200)



C (Stabilwax)



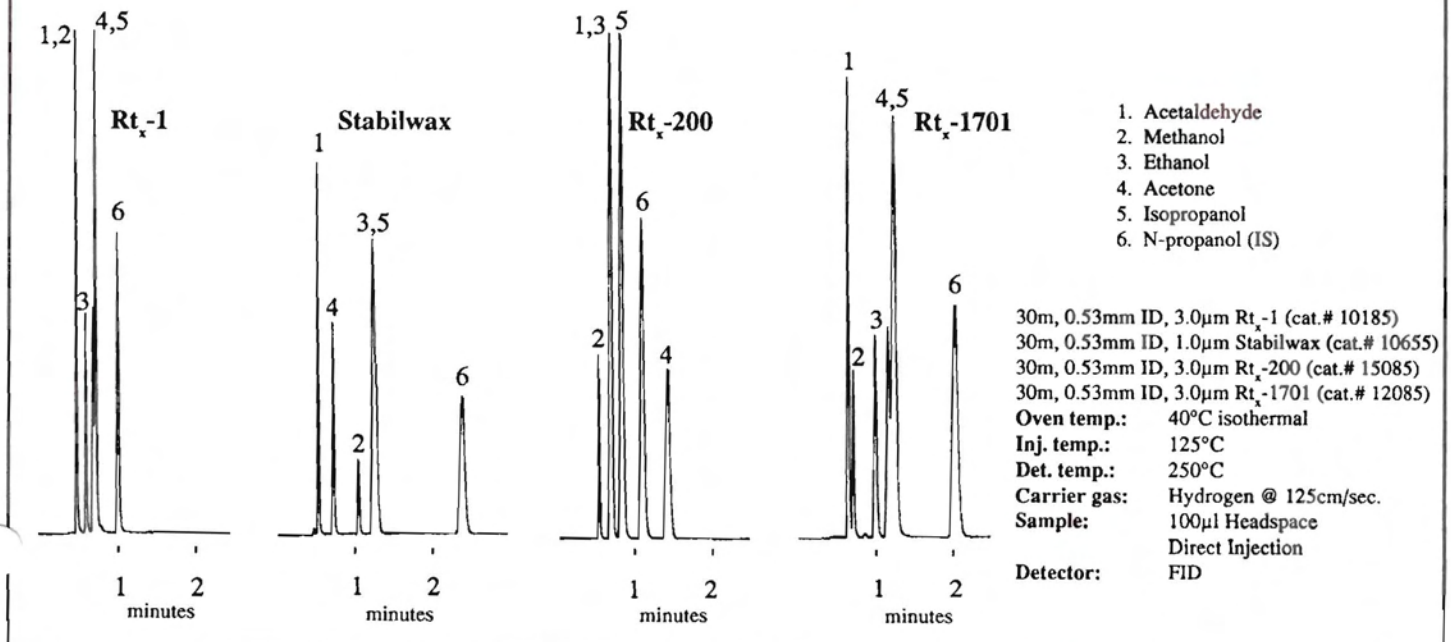
Clinical Corner

Dual Column Analysis for Blood Alcohols

Testing for the presence of ethanol in biological specimens is the highest volume test run in forensic laboratories today. Because of the potential for intoxication by ingestion of other volatile compounds, blood alcohol analysis usually includes screening and quantitation of ethanol plus methanol and isopropanol and two metabolites of these alcohols, acetone and acetaldehyde. Requirements for this analysis include positive or unique identification of all analytes with a short turnaround time. Routine testing for ethanol has been performed for more than 50 years and can be accomplished by a variety of techniques. However, each technique has its own drawbacks. Chemical tests such as oxidation by dichromate or permanganate solutions with subsequent detection by UV spectroscopy are not specific for ethanol and are rarely used any longer. Enzyme assays for ethanol can be highly specific, have sufficient sensitivity and have short turnaround times. However, they do not yield any additional information about the presence or absence of other potential intoxicants. Today, the most widely used technique for blood alcohol analysis is gas chromatography. Gas chromatographic methods have the ability to give excellent resolution for all of the alcohols and their metabolites with short turnaround times. Mass specific detectors allows positive identification of the volatile components present in biological specimens. Unfortunately, most laboratories cannot afford to dedicate a GC/MS system to a high volume test like blood alcohol analysis and must use a non-specific detector like an FID. Confirmation of the identity of these compounds can still be achieved on an FID by using a dual column system that changes the elution order and retention time of the analytes.

The first chromatographic methods for ethanol testing were established using packed columns made with primarily two types of liquid phases, a non-polar methyl silicone phase and a polar polyethylene glycol phase. Choices for capillary columns for blood alcohol analysis have followed the same trends and focus on the same types of liquid phases. Below, we show that a combination of intermediate polarity columns can effectively be used as screening and confirmational columns for ethanol and some closely related compounds. Four different stationary phases were evaluated for use in blood alcohol testing. The most commonly used stationary phases, methyl silicone and polyethylene glycol, and two intermediate polarity stationary phases, trifluoropropylmethyl silicone and cyanopropylphenylmethyl silicone, were run under identical analytical parameters (Figure 1). Thicker film columns were

Figure 1 - The Rt$_x$-1701 proved to be the best screening column while the Rt$_x$-200 is the best confirmational column for blood alcohol analysis.



used to increase each column's resolving power and sample handling capacity. To minimize analysis time, high carrier gas flow rates using hydrogen as the carrier gas were employed. Hydrogen was chosen as the carrier gas to minimize the loss in column efficiency when run with high flow rates.* Results showed that all four columns have partial or complete co-elutions of some compounds. There are two co-elutions on the Rt_x-1 column (methyl silicone). Methanol and acetaldehyde are completely unresolved and acetone and isopropanol are only 40% resolved. The remaining compounds elute very close to one another because of the low polarity of the methyl silicone stationary phase. The Stabilwax column (polyethylene glycol) shows better selectivity than the Rt_x-1 column because of the increased stationary phase polarity, but ethanol and isopropanol co-elute with one another. The Rt_x-200 column (trifluoropropylmethyl silicone) had excellent resolution for all of the alcohols due to its selectivity for compounds with lone pair electrons. The only co-elution was with ethanol and acetaldehyde. When compared to the other phases, the Rt_x-1701 column (cyanopropylphenyl methyl silicone) had the best overall resolution. There were no complete co-elutions, with acetone and isopropanol being approximately 40% resolved.

In choosing two columns for use in a dual column system for blood alcohol analysis, overall resolution and co-elutions with ethanol are the primary considerations. Although ethanol is completely resolved on the Rt_x-1 column, it was excluded because of the two co-eluting pairs and low resolving power. The Stabilwax column was dropped from consideration because ethanol co-eluted with isopropanol, a potential contaminant from skin preparation prior to collecting blood samples. Based upon the chromatography for the two remaining phases, the Rt_x-1701 column would be the choice for the screening column. It provides the best resolution of all compounds. The Rt_x-200 would then be the choice for the confirmational column. Because of the unique selectivity of the trifluoropropyl phase, the elution order for acetone and acetaldehyde relative to the other alcohols is dramatically altered. This shift in retention time and elution order provides the mechanism for confirmation of identity of any volatile intoxicants present. Acetone and isopropanol, which had been unresolved on the Rt_x-1701, are now completely resolved from one another. The co-elution of ethanol and acetaldehyde on the Rt_x-200 is a minor problem since clinically significant concentrations of acetaldehyde are rarely encountered.

". . . the Rt_x-1701 column would be the choice for the screening column. . . . The Rt_x-200 would then be the choice for the confirmational column."

The Rt_x-1701 and the Rt_x-200 were then installed into an instrument containing a packed column injection port and two FIDs. Installation of both columns into the injection port was achieved by using a "Y" Press-Tight®connector and a 10cm length of 0.53mm ID deactivated guard column tubing. A 1mm ID Uniliner®was used to adapt the packed column port for use with wide bore capillary columns. Because both columns were of the same length and internal diameter, carrier gas flow and samples to be analyzed were split evenly between the two columns. The detector ends of each column were then installed in separate FIDs. All other analytical parameters were the same. The chromatography achieved with this dual column system was identical to that obtained when using a single column.

Rt_x-1701 and Rt_x-200 columns can be used in a dual column configuration that provides rapid detection and quantitation for ethanol and associated volatile compounds in biological samples.

Product Listing

Phase & Composition	length	ID (mm)	df	cat.#	price
Rt_x-1701	30m	0.53	3.0	12085	$445
Rt_x-200	30m	0.53	3.0	15085	$475
Guard Column	5m	0.53	---	10045	$60
6-pack	5m	0.53	---	10045-600	$300
"Y" Press-Tight connector	---	---	---	20405	$55
3-pack	---	---	---	20406	$145

* longer analysis times and some loss of resolution may occur with helium as the carrier gas

In the last issue of The Restek Advantage (November 1991, Vol. 2 No. 5), there were some peak misidentifications in Figure 5 on page 4. Corrections are shown below:

As shown in newsletter		Correction
Peak#	Name	
3	Carbon tetrachloride	Tetrachloroethylene
6	n-Propyl nitrate	Bromoform
7	Methylene bromide	n-Propyl nitrate

Standards Spotlight

New Chemical Standards for EPA 505 Series Methods

- only source of complete method 505 analytes
- available with complete data pack documentation
- in stock for immediate delivery

Restek continues to expand its line of environmental chemical standards. In an effort to provide analytical laboratories with calibration standards to meet our clients requirements, we are pleased to announce the availability of this new product.

Restek is the only source of a calibration mixture for EPA Method 505 which contains every analyte specified in the protocol. We have had several rare compounds synthesized to be able to provide this calibration standard.

As with all Restek environmental standards, a complete data pack is available to comply with EPA regulations. Restek data packs have been accepted by EPA auditors across the USA. Why take a chance? Order your "audit survival pack" when you purchase our standards.

Method 505 - Organohalide Pesticide Mix

Hexachlorocyclopentadiene	Hexachlorobenzene
Simazine	Atrazine
γ-BHC (Lindane)	Alachlor
Heptachlor	Heptachlor epoxide
Aldrin	Dieldrin
Endrin	Methoxychlor
α-chlordane	γ-chlordane
cis-Nonachlor	trans-Nonachlor

200µg/ml each in 1ml methanol

Cat.# 32024 $30ea.
32024-500 $60 ea. w/data pack
32124 $270 10pk. w/data pack

Recommended columns for this method:

30m, 0.32mm ID, 1.0µm Rt_x-1 (cat.# 10154)
30m, 0.32mm ID, 1.0µm Rt_x-50 (cat.# 10554)

Product Modification - DMSO to be Used for USP 467 Standard

The solvent used for USP 467 Calibration Mixture (part no. 36000, 36100) advertised in The Restek Advantage, September 1991, has been changed from water to Dimethyl sulfoxide (DMSO). Several articles in recent issues of the Pharmacopeial Forum (1,2,3) have addressed the difficulty in obtaining consistent results with this method and the difficulty in preparing accurate calibration standards. Dimethyl sulfoxide was chosen as the replacement solvent for two reasons. First, all of the compounds being analyzed in USP 467 are soluble in DMSO. Second, DMSO is miscible with water and will act as a carrier for non-polar analytes that have poor solubility in water. This change should pose no difficulties to analysts using USP Method 467. This mixture can be easily diluted water or other solvents specified in the USP monograph.

USP 467 Calibration Mix

Ethylene oxide	10mg/ml
Benzene	100
Chloroform	50
1,4-dioxane	100
Methylene chloride	100
Trichloroethylene	100

At concentrations listed in 1ml dimethyl sulfoxide.
Packaged 1ml per ampul.

Cat.# 36000 $20ea.
36100 $180 10pk.

Recommended columns for this method:

Phase & Composition	length	ID (mm)	df	cat.#	price
Rtx-5	30m	0.53	5.0µm	10279	$455
Rtx-5	30m	0.53	3.0µm	10285	$445
Rtx-502.2	30m	0.53	3.0µm	10908	$515
Rtx-1701	30m	0.53	3.0µm	12085	$445

References

(1) Chen, T.K., Moekel, W., Surprenant, H.L., Proposed Changes to Method I for Organic Volatile Impurities <467>. Pharmacopeial Forum 1991; 17(1): 1475-479.

(2) Bergren, M.S., D.W., Comments on USP General Chapter Organic Volatile Impurities <467>, and Associated Monograph Proposals. Pharmacopeial Forum 1991; 17(3): 1963-1968.

(3)Krasowski, J.A., Dinh, H., O'Hanlon, T.J., Lindauer, R.F., Comments on Organic Volatile Impurities, Method I, <467>. Pharmacopeial Forum 1991; 17(3): 1969-1972.

Restek is committed to providing the highest quality chemical standards!

We are constantly monitoring method modifications proposed by the governmental regulating agencies. Should additional method improvements be introduced, chemical standard mixtures will be modified to meet customer requirements.

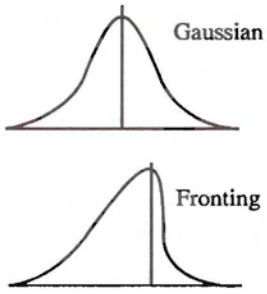
Hints for the Capillary Chromatographer

Sample Capacity and Column Overload

What is column overload?

Column overload occurs when the amount of sample injected exceeds the column's capacity for that component. Overload is normally observed as a fronting, non-gaussian peak shape (Figure 1). A column's capacity is a function of several parameters including the column's internal diameter (ID), its film thickness (df), the solubility of the compound in the column's stationary phase, and capacity factor (k).

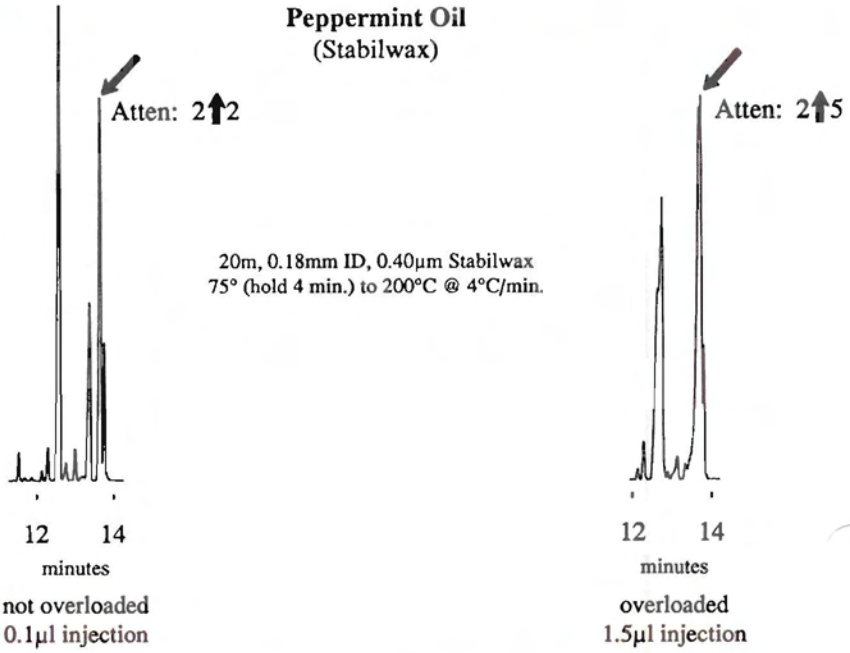
Figure 1 - Normal Gaussian vs. Overloaded Fronting Peak Shapes



Why is it important not to exceed a column's capacity?

Capillary columns have much lower sample capacities than packed columns, therefore, it is extremely important to optimize the amount of sample injected. When sample capacity is exceeded, peak symmetry is lost and resolution is affected. Because the peak shape will be much broader, resolution between two closely eluting peaks can be lost. Figure 2 shows the loss of peak symmetry and resolution in the analysis of peppermint oil on a Stabilwax column. In the first chromatogram, 0.1µl of neat peppermint oil was injected. At these low concentrations, very good resolution between the menthyl acetate, neo-menthol,

Figure 2 - Minimize the amount of sample injected to maximize resolution.



β-caryophyllene, and terpinene-4-ol is obtained. In the second chromatogram, 1.5µl of neat peppermint oil was injected. Because the sample concentration exceeded the column's capacity, a significant loss in resolution occurred.

How can overload be prevented?

Two choices are available to prevent overload:

- ▼ reduce the sample concentration reaching the column
- ▼ choose a column and run conditions that will allow greater sample capacity

To reduce the sample concentration reaching the column, the sample components can be diluted by increasing the split ratio, diluting with additional solvent, or by introducing a smaller amount.

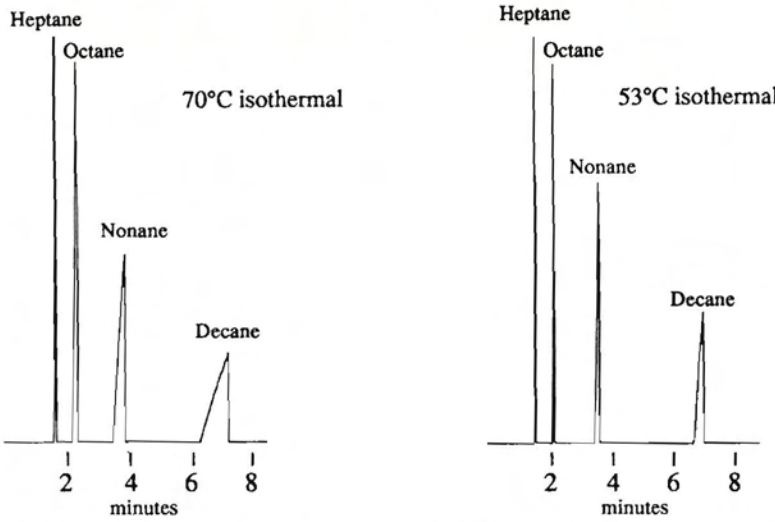
How does column ID affect sample capacity?

As the column ID increases so does sample capacity. Table 1 shows typical column capacity for several different diameter columns. Figure 3 compares sample capacity on 0.25 and 0.53mm ID columns. Four hydrocarbons (heptane, octane, nonane, and decane) were analyzed at a concentration of 1000ng on

Table 1

Column ID	0.18mm	0.25mm	0.32mm	0.53mm
Sample Capacity	<50ng	50-100ng	400-500ng	1000-2000ng

Figure 3 - Increase sample capacity by increasing column ID.



15m, 0.25mm ID, 0.25µm Rt_x-1 (cat.#10120)

15m, 0.53mm ID, 0.25µm Rt_x-1 (cat.# 10122)

both a 0.25mm ID and 0.53mm ID column. The 0.25mm ID column exhibits overload and severe peak fronting for nonane and decane. In comparison, the 0.53mm ID column shows symmetrical peak shapes for nonane and only slight overload for decane. This illustrates the effect of increasing sample capacity by increasing column ID.

How does column film thickness affect sample capacity?

Increasing the column's stationary phase film thickness also increases sample capacity. Figure 4 shows this effect. Again, we show the same series of hydrocarbons at the 1000ng concentrations on 30 meter, 0.25mm ID, 0.25µm and 1.0µm Rt_x-1 columns. On the 0.25µm column, the nonane peak shows some overload and the decane peak shows severe fronting. By increasing the film thickness to 1.0µm, the peak symmetry of nonane is restored and the decane peak shows only slight fronting.

How does solubility affect sample capacity?

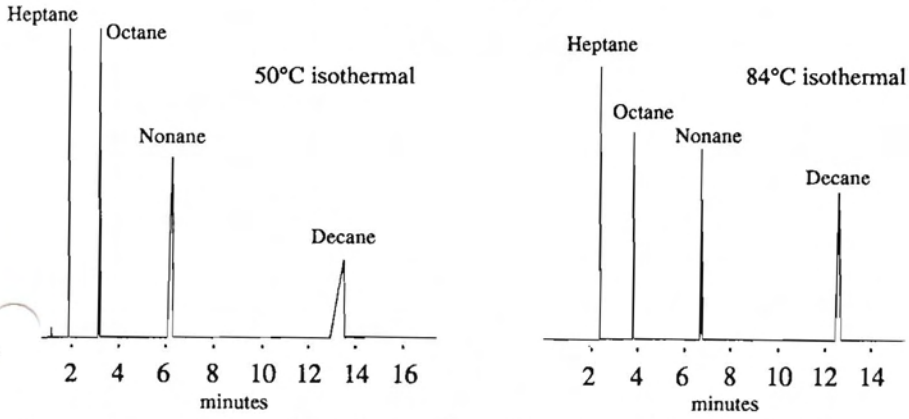
The solubility of a sample component in the column's stationary phase also has an effect on sample capacity. The more soluble a component is in the stationary phase, the greater the column capacity for the solute. For example, a polar compound will have greater solubility in a polar stationary phase than in a non-polar stationary phase. For environmental analysts, this phenomenon is very common when analyzing acid and base-neutral extracts on a non-polar, 5% diphenyl stationary phase. Benzoic acid, a polar compound, always exhibits very poor peak symmetry, demonstrating overload on this non-polar stationary phase. Even though it is at the same concentration, it is less soluble in this phase than the other priority pollutants and exceeds the column's capacity at a much lower concentration.

How does component retention affect sample capacity?

Sample capacity is also affected by how long the component remains in the stationary phase. The capacity factor or *k* gives us an indication of how long the component remains on the stationary phase. The longer a sample component remains in the stationary phase, the greater the chance for overload. The capacity for a component can be increased by selecting run conditions that will create lower *k* values by causing the component to elute faster from the column (faster flow rates or faster temperature programming).

When choosing a column, the analyst must keep in mind the range of component concentrations. By optimizing column ID and film thickness, and by matching the solubilities of sample components with the stationary phase, samples can be analyzed without overload. Also, by optimizing run conditions, the *k* value for components can be minimized resulting in better sample capacity.

Figure 4 - Increasing the stationary phase film thickness increases column sample capacity.



30m, 0.25mm ID, 0.25µm Rt_x-1 (cat.#10123)

30m, 0.25mm ID, 1.0µm Rt_x-1 (cat.# 10153)

If there's a topic you'd like to see covered in Hints for the Capillary Chromatographer, write to:

Hints Topics,
c/o Restek Corporation,
110 Benner Circle,
Bellefonte, PA 16823-8812.

Peak Performers

Restek's Digital Flow Calibrator

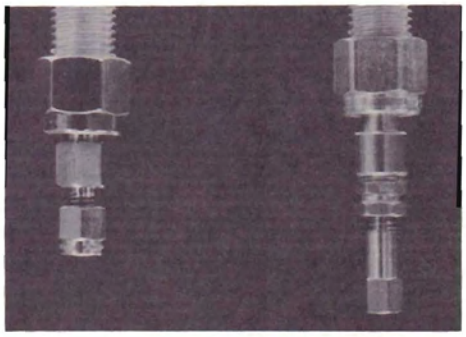
- calibrated against NIST Standards
- large LED display for easy readout
- use with all chromatography gasses
- battery operated for portability

Restek's Digital Flow Calibrator is designed to measure and calibrate gas flows used in capillary chromatography. The flow calibrator is capable of measuring flow rates of .5-500mls/min. accurately, regardless of the gas type. It is an excellent tool for measuring the split vent flow and detector gas flows. This battery operated flow calibrator is easy to operate and is capable of displaying the split ratio.

Digital Flow Calibrator:
cat.# 20123, $495 each

HP 5890 FID/NPD Detector Adaptor Fitting

The new HP 5890 FID/NPD Detector Adaptor fitting has been further improved to ensure a leak-tight column seal. A standard Swagelok®-type nut is incorporated to allow use with standard graphite or Vespel®graphite ferrules. The new adaptor replaces the awkward HP column fitting. We not only shortened the adaptor fitting to make it more compact, but we also included a wrench pad to prevent the fitting from turning when installing a column. The kit includes everything needed for installation: the adaptor fitting, 1/4" SS nut, 1/4" Vespel/graphite ferrule, 1/16" SS nut, and 0.4mm ID graphite ferrule. (Replaces HP part numbers 19244-80610 and 05921-21170.)

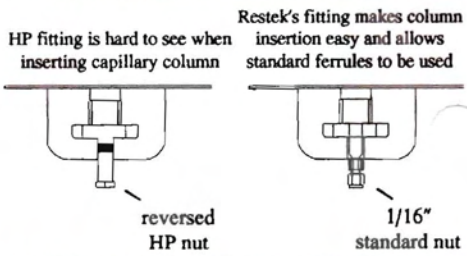


Restek's FID/NPD Detector Adaptor Fitting is shorter and more compact then HP's fitting, making capillary column installation easy.

HP 5890 FID/NPD Detector Adaptor Fitting:
cat.# 20884, $55/kit

HP 5890 Capillary Inlet Adaptor Fitting

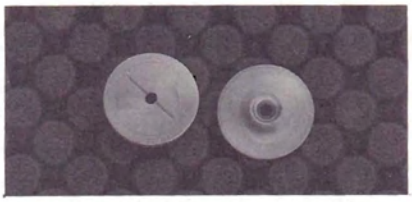
The HP 5890 capillary inlet adaptor fitting has been improved to ensure a leak tight seal with the capillary column. Restek chemists have engineered a simple HP 5890 GC capillary fitting that incorporates a standard 1/16" Swagelok®-type nut and standard graphite or Vespel®graphite ferrules.



We carefully machined the threads and matched the stainless steel types to eliminate seizing onto the injector body. We also paid careful attention to the fitting depth to keep the insertion distance exactly the same. The replacement inlet adaptor kit simplifies column installation due to this easy-to-use design. The kit includes everything needed for installation: the adaptor fitting, an inlet seal and washer, 1/16" SS nut, and 0.4mm ID graphite ferrule. (Replaces HP part numbers 18740-20800, 05921-21170, and 18740-20880.)

HP 5890 Capillary Inlet Adaptor Fitting:
cat.# 20633, $60/kit

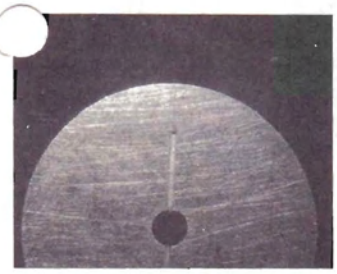
Modified Inlet Seals for HP 5890 GCs



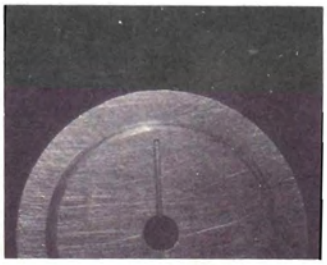
The inlet seal at the base of the HP 5890 GC injection port comes into contact with the sample and must be changed frequently to prevent adsorption of active compounds. In addition, septa fragments and sample residue accumulate on the disk surface requiring replacement.

The seal occurs by deforming the disk against the injection port base upon tightening, forming a micro ring. Originally, the disks were manufactured from 303 stainless steel which did not deform well upon tightening, resulting in a small leak. The new disk design uses 203EZ stainless steel which is softer and deforms easier, making a completely leak-tight seal.

Photomicrograph of HP inlet disks



new disk before installation

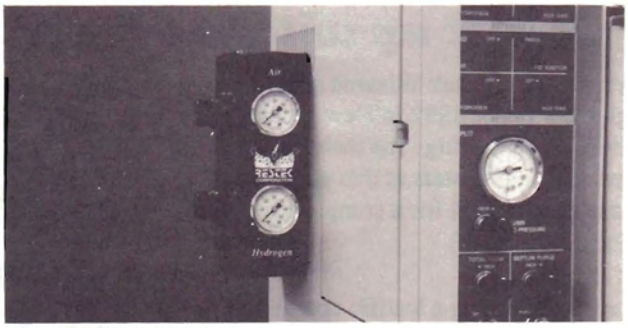


203EZ stainless forms a deep groove to enhance seal

This new disk design increases column lifetime because oxygen cannot permeate into the carrier gas. Detector noise is also reduced with high sensitivity detectors such as ECDs or MSDs. (Replace HP part number 18740-20880.)

Replacement Seals for HP 5890 Split/Splitless Inlets:
cat.# 20390, $30/2-pack
cat.# 20391, $135/10-pack

Control HP 5890 FID Gases With a Gauge Pack



- conveniently mounts on the side of the HP 5890 GC
- standard 1/8" inlet and outlet fittings
- up to 250psi inlet pressures, 0-60psi outlet pressures
- front mounted gauges for easy visibility
- side mounted regulator knobs for easy control

HP 5890 GCs do not include the pressure regulators necessary to adjust the air and hydrogen flows to the Flame Ionization Detector (FID). Since most labs operate their gas supply lines at a higher pressure necessary for the proper operation of the FID, chromatographers must supply their own regulators to adjust flows for optimum detector performance.

Restek's new FID Gauge Pack simplifies GC installation by incorporating pressure regulators and gauges for both air and hydrogen in a single enclosure. The unit conveniently mounts on the side of the GC, and 1/8" bulkhead fittings allow easy hook-up to instrument and supply lines.

HP FID Gauge Pack:
cat.# 20129, $175

Success Starts with Restek's 502.2 Column when Analyzing Volatile Organic Compounds!

Restek's Rt_x-502.2 capillary column separates early eluting gases without cryofocusing or subambient cooling. This column is compatible with all purge & trap and GC systems and is the recommended column in EPA Method 502.2.

Free offer:

For a limited time, receive a 502.2 Standards Kit (containing all sixty compounds and three internal standards) absolutely FREE with the purchase of a 105-meter Rt_x-502.2 column.

105-meter Rt_x-502.2 column with FREE 502.2 Standards Kit: cat.# 10910-250, $1200

**Offer expires March 31, 1992. Offer not valid in conjunction with any other offer.*

News from Restek

The Wizards are Awarded a Patent

On June 9, 1992, Restek was awarded a U.S. Patent for their Cyclosplitter® sleeve design. Issued under Patent #5,119,669, this innovative inlet sleeve provides increased vaporization surface, increased mixing, and is easy to clean. If you would like more information about Restek's deactivated inlet sleeves, please call us at 1-800-356-1688 and request a copy of our "Operating Hints for Split/Splitless Injectors".

Restek Honored

The October issue of Inc. Magazine contains the 1992 Inc. 500, which ranks this country's top 500 fastest-growing privately held companies. Restek is ranked (#246) on the eleventh annual list.

The rankings are based on the percentage increase in sales from 1987 to 1991. During this period, Restek's sales growth was 1137%.

Phone: (814)353-1300
FAX: (814)353-1309
Orders: (800)356-1688

We Want Your Opinion

In July, Restek installed a voice mail system to help expedite phone call transfer and response. With the large volume of in-coming calls, we felt an automated system would allow us to better serve our customers.

Your opinions about our services are important to us and we would like your feedback on our automated phone system. If you have any comments or suggestions, please call Dwayne White at 1-800-356-1688, ext. 131.

Come Visit Us at EAS

November 16 through 20, the Restek wizards will be at the Eastern Analytical Symposium (EAS) in Somerset, NJ (Booth #235). In addition to our product exhibit, we will be presenting papers on "Highly Efficient and Inert Stainless Steel Capillary GC Columns: A Durable, High Temperature Alternative to Fused Silica," and "Characterization of a Novel Stationary Phase for Toxicological Analyses".

Please visit our booth and presentations. The wizards look forward to meeting you and discussing new, innovative products for chromatographic analysis.

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THE RESTEK ADVANTAGE

new!

Rtx™-624, for the Analysis of Volatile Organic Pollutants

- Similar polarity to DB™-624
- Available in lengths up to 105 meters
- Thermally stable to 270°C
- Guaranteed low bleed at maximum temperature

EPA Methods 502.2 and 524.2 were developed for the analysis of sixty volatile drinking water contaminants. These contaminants range from dichlorodifluoromethane (boiling point = -30°C) to 1,2,3-trichlorobenzene (boiling point = 221°C). The volatile pllutants monitored in EPA Methods 502.2 and 524.2 ·e listed in Table 1.

A purge & trap apparatus is used to concentrate the volatile organics from the water matrix. Wide bore (0.53mm ID) capillary columns have been commonly used for the analysis of volatile organic compounds because their flow rates are more compatible with the desorb flows of purge & trap systems then narrow bore capillary columns.

Several different polarity columns are currently being used for the analysis of EPA Method 502.2 and 524.2 compounds. One column, Restek's Rtx™-502.2, has unique selectivity for the volatile organic pollutants and is cited in EPA Method 502.2.

in this issue...

The Rtx™-502.2 stationary phase is based on a diphenyl/dimethyl polysiloxane which provides low bleed and thermal stability up to 280°C.

Some analysts prefer a different polarity column for analyzing volatile organic pollutants. In response to many requests from environmental laboratories, Restek has developed the Rtx™-624 column for the analysis of volatile organics. The Rtx™-624 exhibits similar polarity to a DB™-624, and is based on a cyanopropyl-phenyl/dimethyl polysiloxane. It also exhibits low bleed and thermal stability to 270°C.

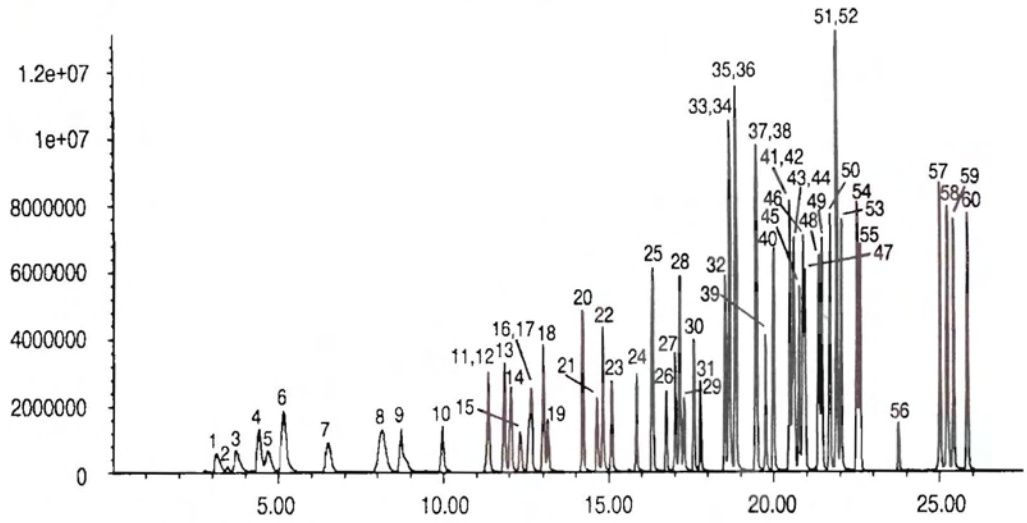
Table I - EPA Method 502.2 Volatile Organic Compounds

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

A 75-meter Rtx™-624 column resolves most of the volatile organic compounds listed in EPA Method 502.2

Figure 1 (on page 2) shows the purge & trap analysis of EPA Method 502.2 compounds on a 75 meter, 0.53mm ID, 3.0µm Rtx™-624 column. This column shows good separation of the early eluting gases without sub-ambient cooling of the GC oven. Under these temperature program conditions the analysis time is only 26 minutes.

Figure 1 - A fast temperature program rate results in reduced analysis time on the Rtx™-624 column.



75m, 0.53mm ID, 3.0µm Rtx™-624 (cat.# 10974)
20ppm of 502.2 Standards Calibration Mix 1-6 (502.2 Volatile Organics Kit, #30060)

Oven temp.:	35°C (hold 8 min.) to 220°C @ 10°C/min. (hold 3 min.)		
Detector:	MS	**Detector temp.:**	250°C
Scan range:	45-300		
Purge & Trap:	#3 trap - Tenax, Silica Gel, Charcoal		
Purge:	11 min.		
Desorb Preheat:	220°C, 2 min. desorb at 225°C		
Desorb Flow Rate:	10ml/min.		

(peak list is on page 1)

Although there are eight pairs of compounds that exhibit coelution, many of these coelutions can be overcome by the detection systems used for volatile organic analysis. When using mass spectrometer detection the ions used for quantitation are different for each set of coeluting compounds. The only exception is m-xylene and p-xylene, which are normally reported as total xylenes.

When using a Photoionization detector (PID) in tandem with an electrolytic conductivity detector (ELCD), the selective response for many of the coeluting compounds can be used to quantify each compound. Because a PID will only respond to compounds that contain aromatic rings or have double bonds, and an ELCD will only respond to compounds that contain halogen groups, several coelutions can be resolved with this dual detection system. For example, peaks 33 and 34 can be resolved since 1,1,1,2-tetrachloroethane will only respond on the ELCD and ethylbenzene will only respond on the PID. The same holds true for peaks 44 and 45. The 1,2,3-trichloropropane responds only on the ELCD and the propylbenzene responds only on the PID.

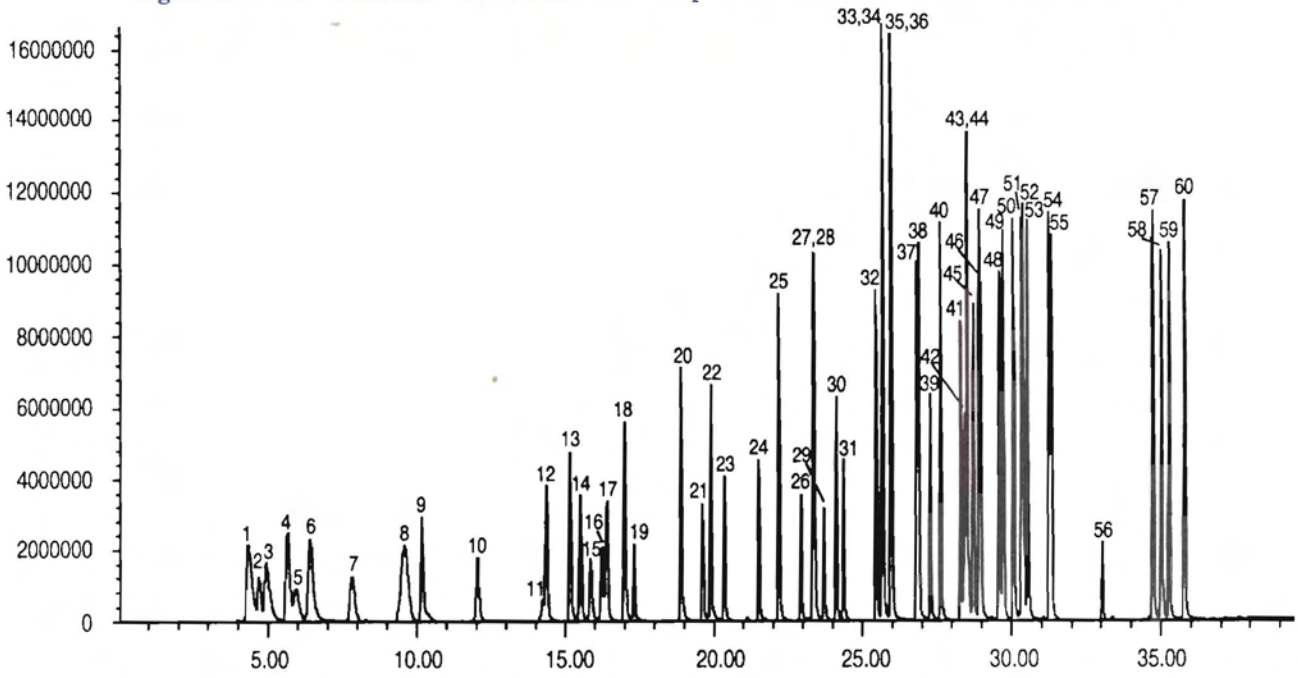
In some cases, the coeluting compounds will both respond on one of the detectors, but only one component responds on the other detector. For example, peaks 46 & 47 (4-chlorotoluene and 1,3,5-trimethylbenzene) both respond on the PID, but only 4-chlorotoluene will respond on the ELCD. In this case the 4-chlorotoluene is quantified using the ELCD response. The 1,3,5-trimethylbenzene can be quantified by first individually determining the response of both compounds on the PID and then by using a ratio of their responses, calculating a response factor for the 1,3,5-trimethylbenzene.

Rtx™-624 Columns are available in a wide range of lengths and diameters.

If additional separation is required, longer length Rtx™-624 columns are also available. Figure 2 shows the analysis of EPA Method 502.2 on a 105 meter Rtx™-624 column. Under optimized conditions, this longer column does increase the overall analysis time, but the number of coeluting compounds is reduced. In addition, the separation of the early eluting gases is improved with the 105 meter column.

The Rtx™-624 column is also offered in 30 and 60 meter lengths which can be used when preforming less complex analyses such as EPA Methods 601, 602, or 624 or utilizing GC/MS. Shorter length columns can also be used if the ini starting temperature of the analysis can be lowered to 10°C by subambient cooling the GC oven with liquid nitrogen or CO_2.

Figure 2 - A 105-meter Rtx™-624 column offers improved resolution over the 75-meter version.



105m, 0.53mm ID, 3.0µm Rtx™-624 (cat.# 10975)
20ppm of 502.2 Standards Calibration Mix 1-6 (502.2 Calibration Standards Kit, #30060)
Oven temp.: 35°C (hold 12 min.) to 220°C @ 7°C/min. (hold 3 min.)

(All other conditions are the same as listed in Figure 1.)

For analysts using cryofocusing techniques, the Rtx™-624 column is also available in 0.25 and 0.32mm IDs. These smaller diameter columns can be directly interfaced to mass spectrometer vacuum systems (0.25mm ID columns are recommended for bench-top mass spectrometers with limited vacuum pumping capacity.) However, the lower flows of these narrower diameter columns require cryofocusing interfaces for connection to purge & trap systems.

The Rtx™-624 column is now available as an alternative to other columns being used for the analysis of volatile organic pollutants. It offers polarity similar to the DB™-624 column. Available in a wide range of lengths and diameters, the Rtx™-624 column can be used with the different methods and techniques for analyzing volatile organics. ■

Rtx™-624 Ordering Information

0.25mm ID, 1.4µm columns				0.32mm ID 1.8µm columns			
30-meter		60-meter		30-meter		60-meter	
cat.#	price	cat.#	price	cat.#	price	cat.#	price
10968	$400	10969	$665	10970	$425	10972	$725
0.53mm ID, 3.0µm columns							
30-meter		60-meter		75-meter		105-meter	
cat.#	price	cat.#	price	cat.#	price	cat.#	price
10971	$445	10973	$795	10974	$900	10975	$1200

Special Offer only $995! (until 12/31/92)

MXT™-624 Ordering Information

MXT™-624 columns are available as an unbreakable alternative to fused silica Rtx™-624 columns.

0.53mm ID, 3.0µm columns							
30-meter		60-meter		75-meter		105-meter	
cat.#	price	cat.#	price	cat.#	price	cat.#	price
70971	$445	70973	$795	70974	$900	70975	$1200

only $995! (until 12/31/92)

New Columns for Simulated Distillation of Petroleum Fractions

According to ASTM Test Method D-2887

Simulated Distillation (Sim Dist) is an analysis which determines the boiling range distribution of petroleum samples using gas chromatography with temperature programming. Different Sim Dist methods are employed depending upon the boiling range of hydrocarbons in the product to be analyzed. ASTM Test Method D-2887 is most commonly used because its scope specifies petroleum products with a final boiling point less than 538°C (excluding gasoline). This boiling range includes samples such as jet fuel, kerosene, diesel, and gas oil. Although this technique has been in use for over 25 years using mostly packed columns, ASTM D-2887 is currently in the revision process to permit the use of 0.53mm capillary columns (1,2). Capillary columns with crossbonded stationary phases offer several advantages compared to packed columns including: lower column bleed, shorter conditioning times, shorter analysis times, and longer column lifetimes (3). Although the analysis is, in principle, very simple there are some important column and instrument parameters which must be optimized to meet the criteria for column resolution, bleed, and peak skewing specified in ASTM Method D-2887 (4).

It is possible to calculate boiling range distribution from GC data since an apolar stationary phase operated under temperature programmed conditions will elute hydrocarbons in order of increasing boiling points. A calibration of the chromatographic system is performed by injecting a mixture of n-alkanes to cover the hydrocarbon range of the samples. Figure 1 shows the complete analysis of the Simulated Distillation Calibration Mixture in under 23 minutes using the Rtx™-2887 column. A computer program constructs a calibration curve from the hydrocarbon retention times and their atmospheric boiling points, and then uses this curve to calculate the boiling range distribution of the petroleum fractions. Sample area is integrated into area "slices" vs. retention time, and then the boiling point for each cumulative Area % is determined by the computer program. An example analysis of the ASTM Reference Gas Oil #1 appears in Figure 2. Note that it is not desirable to resolve all the components in a sample when performing Sim Dist, since a typical laboratory distillation used for petroleum analysis generates a limited number of theoretical plates.

It is important that the column and chromatographic condition are set up according to the procedure specified in the ASTM standard, otherwise a laboratory's results will not be comparable with results obtained by other labs. The Rtx™-2887 and MXT™-2887 column dimensions, stationary phase, and stationary phase film thickness are optimized to meet the requirements specified in the current revision of the ASTM test

Figure 1 - Calibration Analysis of C5 to C44 Standard (baseline compensated).

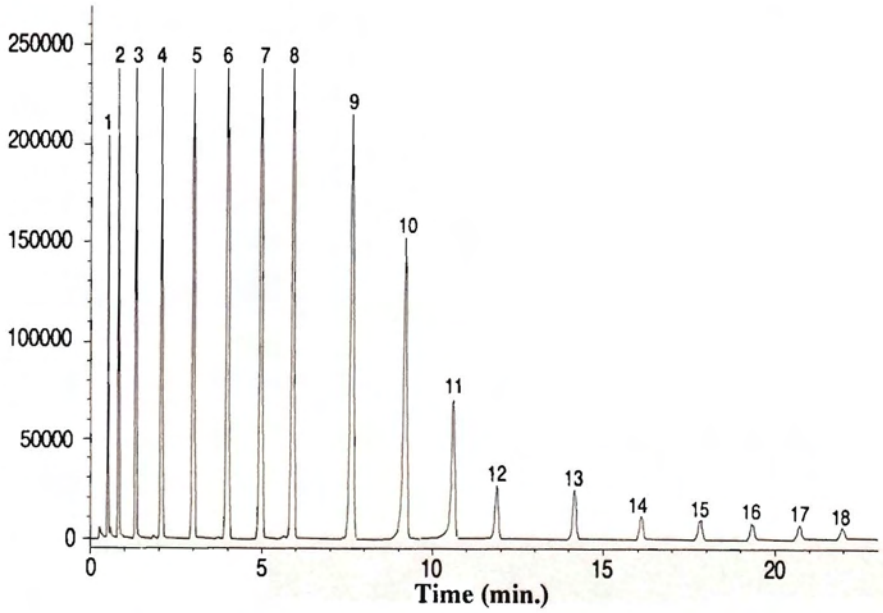
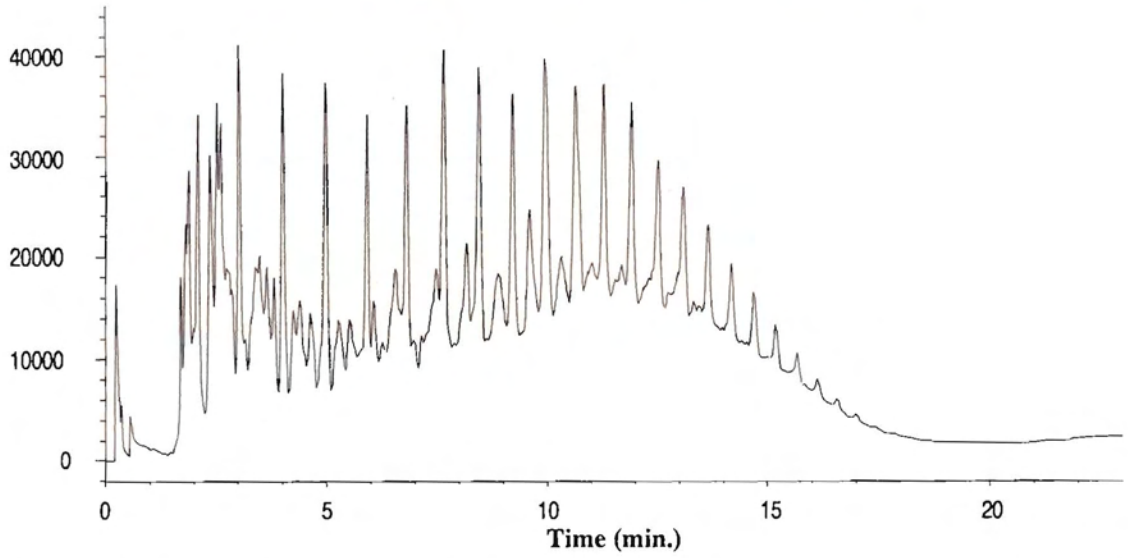


Figure 2 - ASTM Reference Gas Oil #1 (baseline compensated).



method. For example, the resolution for C16/C18 is 8.7, which is within the specified range of 3 to 10 and the skewing factor for heptane is 0.92 which must be greater than 0.5 and less than 2. The Crossbond™ methyl silicone stationary phase has increased stability compared to packed columns resulting in longer column lifetimes and shorter conditioning times when installing a new column. Each column is individually tested with a hydrocarbon mixture to guarantee a stable baseline with low bleed and reproducible retention times. This test assures that the fused silica Rtx™-2887 and the fused silica lined stainless steel MXT™-2887 will meet the performance requirements specified in ASTM Test Method D-2887. ■

References

1. Green L.E., Schumauch L.J., Worman J.C., *Anal. Chem.*, Vol. 36, 1964 p.1512.
2. Green L.E., *Hydrocarbon Processing*, May 1976 p.506
3. Workman D.S., Noel F., Watt M.R. in press
4. ASTM Test Method D-2887, *1992 Annual Book of ASTM Standards*, Volume 5.02

Peak List and Run Conditions for Figures 1 & 2.

COMPOUNDS			
1	pentane	10	hexadecane
2	hexane	11	octadecane
3	heptane	12	eicosane
4	octane	13	tetracosane
5	nonane	14	octacosane
6	decane	15	dotricontane
7	undecane	16	hexatricontane
8	dodecane	17	tetracontane
9	tetradecane	18	tetratetracontane

10m, 0.53mm ID, 2.65µm Rtx™-2887 (cat.# 10199)
1.0µl direct injection of 0.1 to 0.01 wt % hydrocarbon standard in carbon disulfide

Oven temp.: 35°C to 360°C @ 15°C/min. (hold 5 min.)
Inj. & det. temp.: 360°C
Carrier gas: nitrogen
Linear velocity: 112cm/sec (15ml/min.)

Ordering Information

MXT™-2887	10m, 0.53mm ID, 2.65µm	cat.# 70199, $175
Rtx™-2887	10m, 0.53mm ID, 2.65µm	cat.# 10199, $175

MXT™ Columns Now Available in Eight Different Polarities

The Largest Selection of Stainless Steel Capillary Columns

1. Have you ever had a fused silica capillary column break during your analysis? ☐ Yes ☐ No
2. Are you doing process control analysis? ☐ Yes ☐ No
3. Do you perform field analysis requiring a rugged, inert analytical column for your portable GC? ☐ Yes ☐ No
4. Do you have a GC oven that can only accommodate smaller diameter columns? ☐ Yes ☐ No

If you answered yes to any of the questions above, let Restek's MXT™ columns solve your problems.

What are MXT™ columns?

Restek first introduced fused silica lined, thin wall stainless steel capillary columns, the MXT™ series, in March 1992. MXT™ columns are made by depositing a uniform, micron layer of flexible fused silica on the inner surface of stainless steel. The surface is then deactivated and made inert by the same process used to treat our Crossbond® fused silica columns. A special coating and bonding process allows us to make columns in a wide variety of polarities.

MXT™ columns offer many advantages

MXT™ columns were developed to increase the utility of capillary chromatography. They offer combined benefits of fused silica and stainless steel capillary columns such as:

- High degree of inertness to active sample components that is comparable to fused silica
- Extreme flexibility with no risk of spontaneous breakage
- No loss in tubing strength with continual heating
- Rapid, uniform heat transfer
- Rugged, not affected by abrasions or scratches
- Easy to cut and connect in existing GC inlets and detectors
- Smaller coil diameter, 4″ for portable GCs
- Comparable pricing to fused silica

MXT columns are easy to use

MXT™ columns are made with thin-walled, stainless steel tubing. Both the 0.28mm ID and 0.53mm ID columns can be installed using conventional 0.8mm graphite ferrules. MXT™ columns can be installed directly into most instruments without any modification or pre-column adaptor. The inside diameter of the 0.53mm ID column is large enough to allow a standard 26 gauge needle to be inserted for on-column injections.

MXT™ columns are easily cut using a small file that is included with each column. The technique is similar to that used to cut fused silica tubing, except that more deliberate pressure is required. Once the tubing is scored, it snaps cleanly with properly applied force. This fused silica lined stainless steel tubing should be handled similarly to polyimide coated fused silica tubing. Sharp kinks or 90 degree bends must be avoided.

Extended line of MXT™ columns

MXT™ columns offer similar inertness and efficiency as fused silica columns, Restek now offers MXT™ columns in a wide range of polarities, lengths, and film thicknesses.

If the idea of an unbreakable capillary column sounds like the solution to your analytical problems, try an MXT column. Like all Restek products, MXT™s are backed by a 100% satisfaction guarantee. ■

MXT™-1
(100% dimethyl polysiloxane)
Applications
Sim Dist, waxes, fuel oils, pharmaceutical, solvents

			0.28mm ID columns						0.53mm ID columns					
			15-meter		30-meter		60-meter		15-meter		30-meter		60-meter	
df (µm)	max. temp.		cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price
0.10	400°C		70106	$235	70109	$400	-----	-----	-------	------	-------	------	-------	------
0.15	400°C		-------	-----	-------	-----	-----	-----	70107	$240	70110	$445	-------	------
0.25	360°C		70121	$235	70124	$400	70127	$715	70122	$240	70125	$445	70128	$795
0.50	330°C		70136	$235	70139	$400	70142	$715	70137	$240	70140	$445	70143	$795
1.00	325°C		70151	$235	70154	$400	70157	$715	70152	$240	70155	$445	70158	$795
1.50	320°C		-------	-------	-------	------	-------	------	70167	$240	70170	$445	70173	$795
3.00	300°C		70181	$235	70184	$400	70187	$715	70182	$240	70185	$445	70188	$795
5.00	275°C		-------	------	-------	------	-------	------	70177	$255	70179	$455	70183	$820
7.00	250°C		-------	------	-------	------	-------	------	70191	$255	70192	$455	70193	$820

MXT™-5
(95% dimethyl/5% diphenyl polysiloxane)
Applications
semi-volatiles, pesticides, PCBs, environmental samples, essential oils

		0.28mm ID columns						0.53mm ID columns					
		15-meter		30-meter		60-meter		15-meter		30-meter		60-meter	
df (µm)	max. temp.	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price
0.25	360°C	70221	$235	70224	$400	70227	$715	70222	$240	70225	$445	70228	$795
0.50	330°C	70236	$235	70239	$400	70242	$715	70237	$240	70240	$445	70243	$795
1.0	325°C	70251	$235	70254	$400	70257	$715	70252	$240	70255	$445	70258	$795
1.5	320°C	-------	------	-------	------	-------	------	70267	$240	70270	$445	70273	$795
3.0	300°C	70281	$235	70284	$400	70287	$715	70282	$240	70285	$445	70288	$795
5.0	275°C	-------	-------	-------	------	-------	------	70277	$255	70279	$455	70283	$820

MXT™-1301

(7% cyanopropyl/93% dimethyl polysiloxane)

Applications
volatile organics, pharmaceutical

		0.28mm ID columns						0.53mm ID columns					
		15-meter		30-meter		60-meter		15-meter		30-meter		60-meter	
df (µm)	max. temp.	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price
0.25	280°C	76021	$235	76024	$400	76027	$715	76022	$240	76025	$445	76028	$795
0.50	270°C	76036	$235	76039	$400	76042	$715	76037	$240	76040	$445	76043	$795
1.0	260°C	76051	$235	76054	$400	76057	$715	76052	$240	76055	$445	76058	$795
1.5	250°C	76066	$235	76069	$400	76072	$715	76067	$240	76070	$445	76073	$795
3.0	240°C	-----	-----	-----	-----	-----	-----	76082	$240	76085	$445	76088	$795

MXT™-20

(80% dimethyl/20% diphenyl polysiloxane)

Applications
flavor aromatics, alcoholic beverages

		0.28mm ID columns						0.53mm ID columns					
		15-meter		30-meter		60-meter		15-meter		30-meter		60-meter	
df (µm)	max. temp.	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price
0.25	310°C	70321	$235	70324	$400	70327	$715	70322	$240	70325	$445	70328	$795
0.50	300°C	70336	$235	70339	$400	70342	$715	70337	$240	70340	$445	70343	$795
1.0	295°C	70351	$235	70354	$400	70357	$715	70352	$240	70355	$445	70358	$795
1.5	280°C	70366	$235	70369	$400	70372	$715	70367	$240	70370	$445	70373	$795
3.0	260°C	70381	$235	70384	$400	70387	$715	70382	$240	70385	$445	70388	$795

MXT™-1701

(14% cyanopropyl/86% dimethyl polysiloxane)

Applications
pesticides, PCBs, pharmaceutical, alcohols, solvents

		0.28mm ID columns						0.53mm ID columns					
		15-meter		30-meter		60-meter		15-meter		30-meter		60-meter	
df (µm)	max. temp.	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price
0.25	280°C	72021	$235	72024	$400	72027	$715	72022	$240	72025	$445	72028	$795
0.50	270°C	72036	$235	72039	$400	72042	$715	72037	$240	72040	$445	72043	$795
1.0	260°C	72051	$235	72054	$400	72057	$715	72052	$240	72055	$445	72058	$795
1.5	250°C	72066	$235	72069	$400	72072	$715	72067	$240	72070	$445	72073	$795
3.0	240°C	-----	-----	-----	-----	-----	-----	72082	$240	72085	$445	72088	$795

MXT™-Wax

(Crossbond® Carbowax®)

Applications
FAMEs, flavors, solvents, BTEX, essential oils, EPA Method 603

		0.28mm ID columns						0.53mm ID columns					
		15-meter		30-meter		60-meter		15-meter		30-meter		60-meter	
df (µm)	max. temp.	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price
0.25	250°C	70621	$240	70624	$400	70627	$715	70622	$240	70625	$445	70628	$795
0.50	240°C	70636	$240	70639	$400	70642	$715	70637	$240	70640	$445	70643	$795
1.0	230°C	70651	$240	70654	$400	70657	$715	70652	$240	70655	$445	70658	$795
1.5	220°C	-----	-----	-----	-----	-----	-----	70666	$255	70669	$455	70672	$820

MXT™-502.2

Applications
volatile organics

		0.28mm ID columns						0.53mm ID columns					
		30-meter		60-meter		105-meter		30-meter		60-meter		105-meter	
df (µm)	max. temp.	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price
1.6	270°C	70919	$400	70920	$695	70921	$945	-----	-----	-----	-----	-----	-----
3.0	270°C	-----	-----	-----	-----	-----	-----	70908	$515	70909	$915	70910	$1200

MXT™-Volatiles

Applications
volatile organics, trihalomethanes

		0.28mm ID columns						0.53mm ID columns					
		30-meter		60-meter		105-meter		30-meter		60-meter		105-meter	
df (µm)	max. temp.	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price	cat.#	price
1.25	275°C	70924	$400	70926	$695	70928	$945	-----	-----	-----	-----	-----	-----
2.0	275°C	-----	-----	-----	-----	-----	-----	70925	$475	70927	$825	70929	$1400
3.0	275°C	-----	-----	-----	-----	-----	-----	70922	$475	70923	$825	-----	-----

Sim Dist MXT™-1

6m, 0.53mm ID, 0.15µm, cat.# 70101 $200*

*cat.# 70101's are tested at 400°C but may be run at 445°C at a lower sensitivity with additional conditioning.

Sim Dist MXT™-2887

ASTM Method 2887

10m, 0.53mm ID, 2.65µm, cat.# 70199 $175

**cat.# 70199's maximum temperature is 360°C.

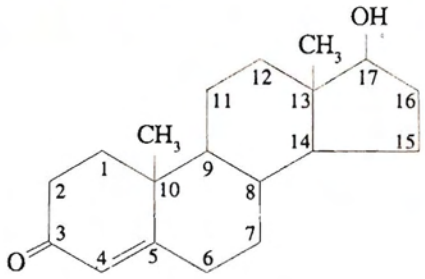
Note: All maximum operating temperatures are for 30m columns. Maximum temperatures may be slightly lower for longer lengths.

Clinical Corner

Analysis of Anabolic Steroids

Steroids are a group of polycyclic compounds that have a wide variety of functions in human biochemistry. They have been identified as precursors to vitamins and bile acids as well as forming the base structure for many different hormones. The anabolic steroids, or androgens, are responsible for many of the virilizing characteristics associated with male development. Increases in height and muscle development can be linked to increased androgen production during the onset of puberty in boys. The potential for abuse of anabolic steroids is linked to this developmental phenomenon. Athletes have used anabolic steroids since the 1950's (1) to try to add lean body mass and subsequently enhance athletic performance. The abuse of steroids today has spread from the competitive arena to those that are just seeking to improve their own physical appearance by "bulking up". Because of the increased use of anabolic steroids and the potential for harmful side effects when used in large doses, anabolic steroids were classified as a Controlled Substance and placed under Schedule III effective February 27, 1991 (2). The Controlled Substances Act regulates the manufacture, distribution or dispensing of anabolic steroids in bulk form, whereas the use and misuse of anabolic steroids is usually controlled by the different sports regulating bodies.

Figure 1 - Chemical structure of testosterone.



The anabolic steroids are all structurally related to testosterone (Figure 1). Modifications at the 3,5,9 and 17 positions give a variety of androgens that not only differ in molecular weight and boiling point, but also in pharmacological activity. The abundance of structurally related compounds multiplies the difficulty of detecting and confirming the identity of anabolic steroids in both clinical samples and bulk formulations. Analysis of anabolic steroids is most commonly performed by gas chromatography with detection by mass spec. Some of the higher molecular weight steroids, like 1-dehydrotestosterone benzoate and 1-dehydrotestosterone undecylenate require high temperature for extended periods of time in order to be eluted from standard film thickness columns. In order to reduce the effective elution temperature and reduce the overall analysis time, columns made with thin films of non-polar stationary phases should be used.

Retention time for the steroids . . . will be affected by the choice of column length, stationary film thickness and stationary phase polarity.

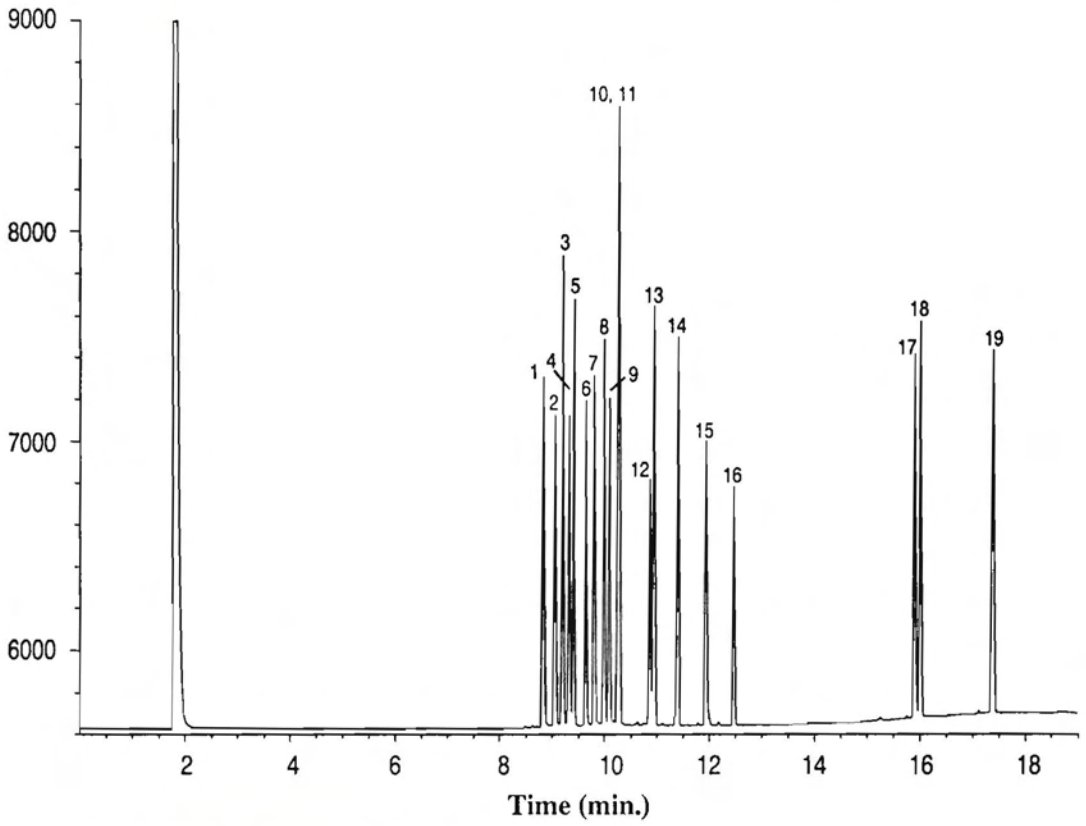
Figure 2 shows the analysis of a mixture of anabolic steroids on an Rtx™-5 column. An analysis time of less than 18 minutes was achieved by using a 0.10 micron film thickness. When film thicknesses greater than 0.10 microns were used, the last three steroids could not be eluted except by using final hold times in excess of twenty minutes with a corresponding deterioration in peak shape. Retention time is also strongly influenced by the choice of stationary phase. Although lower polarity stationary phases are needed to maintain reasonable analysis times and peak shapes, non-polar stationary phases do not provide enough resolution to maintain separation between all of the steroids. A 5% phenyl stationary phase provides enough polarity to improve the resolution of the early eluting steroids without substantially lengthening the retention time of the latest eluting steroids. A 30-meter length column was used to minimize retention times without sacrificing resolution.

Several factors must be taken into account when selecting a column for anabolic steroids. Retention time for the steroids, as well as resolution, will be affected by the choice of column length, stationary film thickness, and stationary phase polarity. By combining a low polarity stationary phase like the Rtx™-5 with a thin film, minimal length format (30m, 0.25mm ID, 0.10µm), reasonable analysis times can be achieved while maximizing the resolution between most of the anabolic steroids. ■

References

1. Wright, J. *Anabolic Steroids and Sports.* Natick, MA: Sports Science Consultants 3, 1978.
2. Federal Register, Vol. 56 No. 30, p. 98-99.

Figure 2 - The analysis of a mixture of anabolic steroids on an Rtx™-5 column.



COMPOUNDS	
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

30m, 0.25mm ID, 0.10µm Rtx™-5 (cat.# 10208)
0.5µl split injection of anabolic steroids, concentration = 1000ng/µl

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

*Maximum operating temperatures on the 30m, 0.10µm Rtx™-5 have been raised to 340°C to provide faster analysis times for high molecular weight compounds.

Ordering Information

Rtx™-5	30m, 0.25mm ID, 0.10µm	cat.# 10208, $380

To order any Restek product, call 800-356-1688 (ext. 3).

Standards Spotlight

Standards for Underground Storage Tank Monitoring

• Composite fuel standards for many state programs • High concentration for maximum utility • • Complete preparation / QA documentation for audits •

Monitoring for leaks from underground storage tanks (UST) has become a major endeavor for many environmental laboratories. This endeavor is complicated because the U.S. Environmental Protection Agency has not adopted a uniform procedure for performing this analysis. Many environmental laboratories accept samples from across state boundaries, and the analytical methods vary from state-to-state.

In the May 1992 issue of *The Restek Advantage*, UST standards were introduced following both the Wisconsin and the EPA UST Work Group, API, Midwest Research Institute, Enseco, Inc. procedures. In each of these methods, instrument calibration is performed using standards that contain representative hydrocarbons in known concentrations. While the standards are not as complex as actual fuels, they may provide an approximation of the fuel contamination in the sample. In the July 1992 issue of *The Restek Advantage*, column selection for UST Monitoring methods was discussed in detail. The methods covered in the May and July issues, along with what we provide in this issue, should give an extensive amount of information to laboratories involved in monitoring UST leakage.

Numerous states have adopted methods which require laboratories to perform instrument calibration using standards prepared from actual petrochemical products. In some cases, these methods mandate the use of standards prepared from materials actually stored in the tank being investigated. It would be impossible to provide calibration standards where these type of requirements exist. However, many other states require the use of composite standards prepared from petrochemical products obtained from multiple sources. These states include (but may not be limited to) Wisconsin, Oregon, California, and South Carolina.

Restek is committed to providing high quality products at a reasonable cost, in addition to timely delivery. If the product you require is not listed below, please contact us at 1-800-356-1688 to discuss the possibility of obtaining a custom chemical standard.

Retention Time Standard

This hydrocarbon mixture may be used during initial screening of the samples to determine retention time windows for each type of petroleum product. Again, individual state requirements vary in this regard. It is generally accepted that gasoline would elute in the window established from C5 (or C6) to C10 (or C12). Diesel fuel would elute in the window established from C10 (or C12) to C24 (or C28). Retention above C24 (or C28) would indicate the presence of oils or lubricants.

LUST Retention Time Standard

Contains the following compounds at a concentration of 2500µg/ml each in 1ml methylene chloride.

pentane (C5)	hexane (C6)
decane (C10)	dodecane (C12)
tetracosane (C24)	octacosane (C28)
triacontane (C30)	tetracontane (C40)

Cat.# 31099 $28 ea.
31099-500 $58 ea. w/data pack
31199 $252 10pk. w/data pack

Unleaded Gasoline Composite

Samples of regular and premium grade unleaded gasoline were each collected from three different sources (six samples total). Equal volumes of each were blended to form a composite unleaded gasoline sample. The composite blend was then used to produce the mixture listed below at a concentration of 2500µg/ml in 1ml purge & trap grade methanol.

Unleaded Gasoline Composite Standard

Cat.# 30081 $25 ea.
30081-500 $35 ea. w/data pack
30181 $225 10pk. w/data pack

Diesel Fuel Composite Standard

Samples of diesel fuel were collected from three different sources. Equal volumes of each were blended to form a composite diesel fuel sample. The composite blend was then used to prepare the mixture listed below at a concentration of 5000µg/ml in 1ml methylene chloride.

Diesel Fuel Composite Standard

Cat.#	**31093**	**$25 ea.**
	31093-500	**$35 ea. w/data pack**
	31193	**$225 10pk. w/data pack**

Kerosene Fuel Composite Standard

Samples of kerosene fuel were collected from three different sources. Equal volumes of each were blended to form a composite kerosene fuel sample. The composite blend was then used to prepare the mixture listed below at a concentration of 5000µg/ml in 1ml methylene chloride.

Kerosene Fuel Composite Standard

Cat.#	**31094**	**$25 ea.**
	31094-500	**$35 ea. w/data pack**
	31194	**$225 10pk. w/data pack**

Gasoline Surrogates

State requirements for the addition of surrogate standards during sample preparation/extraction of suspect gasoline contaminated water and soil vary. The listed surrogates have been identified in several state methods.

Gasoline Surrogate Standards

Individual mixtures at 10,000µg/ml in 1ml purge & trap grade methanol.

compound	each	each w/data pack	10pk. w/data pack
4-bromofluorobenzene	30082	30082-500	30182
	$25	$35	$225
α,α,α-trifluorotoluene	30083	30083-500	30183
	$25	$35	$225
1-chlorooctane	30084	30084-500	30184
	$25	$35	$225

Diesel Surrogates

State requirements for the addition of surrogate standards during sample preparation/extraction of suspect diesel contaminated water and soil vary. The listed surrogates have been identified in several state methods.

Diesel Surrogate Standards

Individual mixtures at 10,000µg/ml in 1ml methylene chloride.

compound	each	each w/data pack	10pk. w/data pack
p-terphenyl	31095	31095-500	31195
	$25	$35	$225
2-fluorobiphenyl	31096	31096-500	31196
	$25	$35	$225
o-terphenyl	31097	31097-500	31197
	$25	$35	$225
1-chlorooctadecane	31098	31098-500	31198
	$25	$35	$225

Restek's Commitment to Environmental Laboratories

Setting the Pace for Others to Follow - Quality, Service, and Price

Restek introduced chemical standard products in July 1990 with a firm commitment to provide our customers with only the highest quality materials commercially available. We also are committed to providing standards that are applicable to the ever changing methods and regulations introduced by the U.S. EPA.

Restek was the **first** company to offer the **complete data pack documentation** required by the EPA Superfund 3/90 statement of work for commercially purchased standards. Our data pack format and content was and has always been accepted by EPA auditors. It was not until a year later that a few competitors offered similar documents. Some still do not. Our data packs are shipped immediately with the product, not weeks after the standards are received.

Restek was the **first** and is still the **only** company to offer **high concentration surrogates and matrix spike mixtures 000µg/ml and higher)**. These products are offered in 1ml and 5ml quantities. Why? You save money and it's more convenient than breaking open five to twenty-five 1ml ampuls to make 50-250ml of working solution.

Restek's inventory control guarantees immediate delivery on all stock products! **Over 99% of chemical standard orders for stock products are shipped the same day when ordered by 4pm!** That's what we call SERVICE.

What about our prices? Restek chemical standard prices are very competitive. In fact, **we have not increased prices for any chemical standard product since they were first introduced** (even products introduced in 1990). We are still using our 1990 price guidelines for products developed today. How can we do that? We have improved our productivity, efficiency, and have negotiated with our suppliers to hold costs for raw materials. If we can do it, why can't everyone else?

Restek Chemical Standards Commitment for 1993

Increased quality by conforming to ISO 9000 guidelines!
Same level of service - Same day shipment!
No price increases for Chemical Standards!

Hints for the Capillary Chromatographer

Quantitative Analysis

(This is Part 2 of our series on Quantitative Analysis covering external standard and area percent techniques. Part 1, which appeared in the September issue, covered internal standard techniques.)

PART 2: External Standard Technique

This quantitative technique requires the use of analyte specific calibration standards. Samples analyzed using this technique cover a broad application spectrum. One of the most common applications of the external standard technique is in pesticide residue determinations.

When can the external standard technique be used?

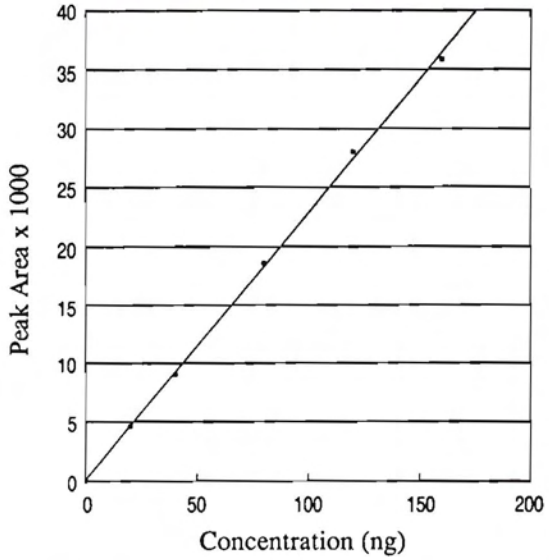
1) If each analyte has a unique detector response.
2) If detector response will not vary during some specific length of time.
3) If retention times remain constant.
4) If injection size can be precisely controlled.

How are external standards used for quantitation?

Prior to sample analysis, calibration standards are prepared for each target compound. These calibration standards are prepared at several different concentration levels which bracket the expected range that target compounds will appear in the sample. For semi-quantitative work, a single point calibration may be performed. For quantitative work, a five point calibration is typically used. The calibration standards are then analyzed in series from lowest to highest concentration. The resultant peak areas for each standard analyte are determined and a retention time window is established. This data is used later for comparison to unknown peaks obtained during sample analysis.

Data obtained from the analysis of calibration standards may be generated by the same two techniques used for the internal standard method. One way to use the external standard technique uses a calibration curve for each compound. The calibration curve is generated by plotting the peak area on the y-axis and the concentration injected on the x-axis. Figure 1 shows a linear calibration curve. After the initial calibration curve has been prepared, analyte linearity can be determined for each analyte over the expected concentration ranges. If a non-linear response is observed, corrective action should be taken. A non-linear response is commonly seen at the extremes in concentration. At low concentrations, non-linearity typically occurs from analyte adsorption or breakdown. At high concentrations, non-linearity typically occurs from detector or column overload. Quantitation should not be performed at concentrations where non-linear response is observed, or erroneous results will occur. Responses for each sample target compound are then compared to the standard calibration curve and the concentration of the analyte is determined by reading the intercept point on the x-axis. This amount of analyte is then used to back calculate the concentration of the analyte in the actual volume of sample.

Figure 1 - Typical Calibration Curve



The second way to use the external standard technique involves the use of response factors for each target compound. Response factors are calculated by dividing the peak area of the calibration standard by the concentration injected. The response factors can be compared for each analyte at various concentrations to determine if they are uniform over the calibration range. A linear plot of response factors (y-axis) versus concentration (x-axis) will be indicated by a flat line. Figure 2 shows a linear plot of response factors. If the response factors are uniform over the calibration range, an average response factor may be calculated and used for the concentration range. Unknown samples are then analyzed, and the resulting peak areas of the target compounds are multiplied by the average response factor to determine analyte concentration in the sample. Figure 3 shows the calculation to determine sample analyte concentrations. The retention time of the target compound must lie within the retention time window of the calibration standard.

Figure 2 - Response Factor Plot

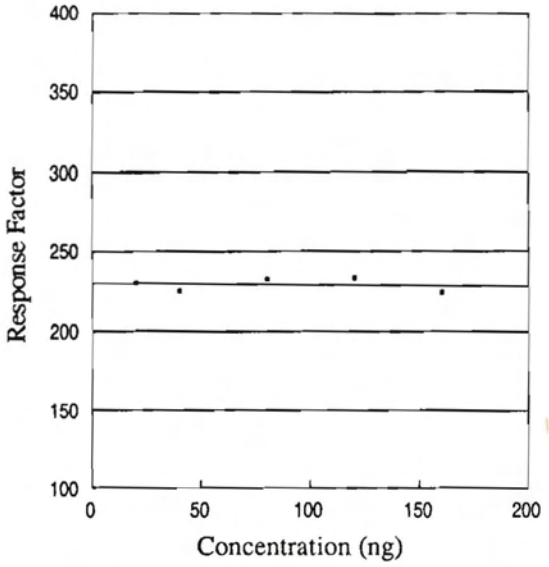


Figure 3

$$\text{Concentration} = \frac{\text{Area (unknown)}}{\text{RF}}$$

What problems are encountered using external standards?

Generating a calibration curve or response factor corrects for the varying response of each analyte. However, there is no mechanism to compensate for instrument condition variations (detector response, injection size, inlet performance) after the initial calibration runs have been made. One way to determine if responses have changed involves analyzing a "continuing check sample". A continuing check sample is a calibration standard prepared at a median concentration level. The check sample is analyzed at a regular time interval to monitor the performance of the system. The frequency of the check sample analysis is largely dependent upon the instrument and samples being analyzed. Extremely dirty samples can contaminate the inlet, detector, or column and may require frequent analysis of the continuing check sample. The peak areas or response factors from the check sample are compared to the values obtained in the initial calibration sequence to monitor instrument response changes. A large change in response necessitates generating a new calibration curve.

Another problem that decreases accuracy of the external standards method is inconsistent injection techniques. The problem is minimized by using automated techniques (i.e. autosamplers) instead of manual injection techniques, but it can still be a major source of error. Changes in room temperature can create variations in the solvent volume, directly effecting the absolute amount being injected into the instrument.

The external standard method also suffers from the problem of shifting retention times. When the initial calibration is established, absolute retention times and/or retention time windows are also established for each target compound. Over extended time periods, instrument variation in oven temperature, column flow rate, or sample matrix effects on the column can cause changes in the retention times of compounds. Comparison of the sample analyte retention times may fall outside the windows previously established. One solution is the establishment of large retention time windows which can lead to misidentification of closely eluting analytes.

To improve the accuracy of analyses done by external standard techniques, confirmation can be done on a second column of different polarity. Dual column confirmation increases both qualitative and quantitative data.

Area Percent Technique

This technique does not require the use of calibration standards. A sample is analyzed and the area of each component in the sample is measured. The total area of all analytes in the mixture is then used to calculate the relative percent composition of each component in the mixture.

This technique is useful only if the relative percent composition of each compound within the mixture is required. This form of quantitative analysis has been used for analysis of natural products such as fatty acid methyl esters and flavor components where impurity concentrations may be monitored in respect to major components. It is also useful for complex hydrocarbon mixtures in petrochemical products.

The area percent technique is also routinely employed is in the determination of compound purity. The assumption is made that the impurities will respond similarly to the main component. It is assumed that the impurities can be volatilized and analyzed under the same conditions as the compound of interest.

When should the area percent technique be used?

1) If ALL compounds in the sample respond with equal intensity.
2) If standards for all target compounds are not readily available.

Although no calibration standards are required for the area percent technique, it is advisable that a standard of each compound of interest be prepared from pure material at the same concentration. The absolute response obtained for each individual compound can be used to confirm that all responses are equal. These standards can also be used to establish retention times and windows.

The quantitation method employed should be chosen based on sample type and the requirements of the analysis. The internal standard technique offers the most accurate quantitation, but requires extensive calibration. The external standard also requires extensive calibration, but consistent retention times and injection volumes are necessary for accurate identification and quantitation. The area percent technique requires less calibration, but is only effective when all target compounds exhibit similar detector response. ■

Peak Performers

Restek's High Quality Thermolite™ Septa

Now Available in Two New Sizes, 7 & 17mm

Receive a free can cooler, coffee mug, or stein with the purchase of Thermolite™ Septa!

Instrument	Septum Size
Hewlett-Packard	
5890 series	10mm/11mm
5700 series	9.5mm/10mm
Varian	
Packed column inj.	9.5mm/10mm
Split/splitless inj.	10mm/11mm
Perkin Elmer	
Sigma series, 900,990	11mm
8000 series	11mm
Tracor	
550, 560	9.5mm
220,222	12.5mm
Gow-Mac (all models)	9.5mm
new! **FISONS/Carlo Erba**	
8000 Series	17mm
new! **Pye/Unicam**	7mm

Septum Diameter	25-Pack Free Can Cooler cat.#	price	50-Pack Free Coffee Mug cat.#	price	100-Pack Free Stein cat.#	price
5mm (3/16")	20351	$35	20352	$55	20353	$95
6mm (1/4")	20355	$35	20356	$55	20357	$95
new! 7mm (.275")	20381	$35	20382	$55	20383	$95
9.5mm (3/8")	20359	$35	20360	$55	20361	$95
10mm (0.4")	20378	$35	20379	$55	20380	$95
11mm (7/16")	20363	$35	20364	$55	20365	$95
12.5mm (1/2")	20367	$35	20368	$55	20369	$95
new! 17mm (.670")	20384	$38	20385	$75	20386	$150
Shimadzu Plug	20372	$35	20373	$55	20374	$95

Learn How to Properly Install a Capillary Column in Just 35 Minutes

The technical wizards at Restek introduce a new instructional video that takes the mystery and frustration out of capillary column installation.

Restek's video features chromatography instructors with extensive training in capillary GC. The video offers proper installation training and practical operating hints for the novice or expert chromatographer. Information covered in the video can be used immediately in the laboratory to improve the performance of your capillary column and GC.

Ideal Training Tool for New Chromatographers!

This essential resource covers critical points in the proper installation of a capillary column such as:

- Choice of carrier gas
- Instrument preparation
- Trouble-free installation
- Leak-checking
- Setting carrier and detector gas flows
- Column conditioning

The installation sequence allows you to install your capillary column correctly, minimize downtime, and get your GC up and running quickly. The video follows the proposed ASTM practice for installing and maintaining capillary columns to insure that your lab conforms with industry standards.

The video includes a printed installation guide and other important technical literature. We guarantee this video will be the most useful instructional tool you'll ever see!

Restek's Capillary Column Installation Video
Cat.# 20490, $149

Restek's Silcosteel® Tubing

nert, flexible, and strong.
- High thermal stability (400°C).
- Available in long lengths (call for availability of >200ft.).
- Priced below Glass Lined Tubing™ (GLT).

Silcosteel tubing is ideal for:
- transfer lines for purge & trap, mass spectrometers, FTIRs, and other instruments.
- sample loops & sampling lines for active compounds.
- replacing fragile glass packed columns.
- on-line process chromatography.

Restek's Silcosteel® is made by a unique process that bonds micron layers of pure, flexible fused silica onto the inner surface of stainless steel tubing. Silcosteel® is deactivated by the same process used to manufacture Restek's fused silica capillary columns to yield a highly inert surface. Unlike GLT, it can be bent into many configurations without heating. Treat Silcosteel® tubing like fused silica capillary columns. It can be cut with a standard tubing cutter or a high speed cut-off wheel. The inner surface can be rinsed with a variety of solvents, including water, without damaging the deactivation layer.

Silcosteel® can be bent in 90° angles, but it is extremely important to make gentle bends with rounded corners. The minimum bend diameter is 2 inches for 1/16" OD, 4 inches for 1/8" OD, and 6 inches for 1/4" tubing. Sharp bends kink the tubing and crack the fused silica layer, causing a loss of inertness.

Metal Swagelok®-type fittings can be tightened on Silcosteel® tubing without causing damage since the flexible fused silica coating compresses as the fitting is tightened and the ferrule bites into the metal wall. We recommend using Silcosteel® treated fittings to maintain the high degree of inertness.

Fittings and custom tubing can also be deactivated with Silcosteel®. Call 1-800-356-1688 for an estimate.

Silcosteel® Fused Silica Lined Stainless Steel Tubing Ordering Information

Length	ID 0.011" (0.28mm) OD 0.022" (0.53mm) cat.#	price	ID 0.021" (0.53mm) OD 0.031" (0.8mm) cat.#	price	ID 0.010" (0.25mm) OD 1/16" (1.6mm) cat.#	price	ID 0.020" (0.51mm) OD 1/16" (1.6mm) cat.#	price
6ft.	20570	$75	20563	$75	20550	$75	20524	$75
25ft.	20571	$275	20564	$275	20551	$275	20525	$275
50ft.	20572	$500	20565	$500	20552	$500	20526	$500
200ft.	20573	$1,800	20566	$1,800	20553	$1,800	20527	$1,800
>400ft.	20574	$7/ft.	20567	$7/ft.	20554	$7/ft.	20528	$7/ft.

Length	ID 0.030" (0.76mm) OD 1/16" (1.6mm) cat.#	price	ID 0.040" (1.02mm) OD 1/16" (1.6mm) cat.#	price	ID 0.085" (2.16mm) OD 1/8" (3.18mm) cat.#	price	ID 0.210" (5.33mm) OD 1/4" (6.35mm) cat.#	price
6ft.	20530	$75	20538	$110	20545	$95	20555	$110
25ft.	20531	$275	20539	$400	20546	$360	20556	$400
50ft.	20532	$500	20540	$725	20547	$685	20557	$725
200ft.	20533	$1,800	20541	$2,700	20548	$2,450	20558	$2,700
>400ft.	20534	$7/ft.	20542	$10.50/ft.	20549	$10/ft.	20559	$10.50/ft.

New! Straight Silcosteel® Tubing

- Ideal for adsorbent traps and thermal desorption tubes.
- Available in 1/8" and 1/4" ODs.
- Easily cut to specific lengths.

The inertness of Silcosteel® makes it ideal for the construction of adsorbent traps and thermal desorption tubes. Straight lengths of tubing are also useful for transfer lines and instrument interfaces. In response to customer requests, we are now offering 18" straight lengths of 1/8" and 1/4" Silcosteel® tubing. This tubing can be cut to your exact requirements using a standard tubing cutter. Straight Silcosteel® is available in individual pieces or in economical 5-packs.

ID 0.085" OD 1/8" cat.#	price	ID 0.210" OD 1/4" cat.#	price
20575	$40 each	20577	$40 each
20576	$150/5-pack	20578	$150/5-pack

THE RESTEK ADVANTAGE

new! Rtx™-65TG Allows Fast, Selective Analysis of Triglycerides

- Unique selectivity
- High thermal stability (370°C)
- Specially tested with triglycerides
- High temperature capillary tubing

Triglycerides are naturally occurring esters of fatty acids and glycerols. They are widely analyzed in the food industry for natural oil and fat characterization and fat adulteration. Health conscious consumers are additionally concerned with minimizing their dietary intake of saturated fatty acids which can promote heart disease. The fatty acid groups in the triglyceride molecule can be classified as saturated: myristic acid (C14:0), palmitic acid (C16:0), or stearic acid (C18:0); or unsaturated: oleic acid (C18:1), linoleic acid (C18:2), or linolenic acid (C18:3). Typically, triglycerides are characterized by degree of unsaturation. For example, a triglyceride molecule containing the groups stearic acid, oleic acid, and linoleic acid (denoted SOL), would have a greater degree of unsaturation than that of tripalmitin (denoted PPP). Table I shows the nomenclature of fatty acids and triglycerides (1).

Table I - Fatty Acid and Triglyceride Nomenclature

Fatty Acids

La	lauric acid, dodecanoic acid, C 12:0
M	myristic acid, tetradecanoic acid, C 14:0
P	palmitic acid, hexadecanoic acid, C 16:0
S	stearic acid, octadecanoic acid, C 18:0
A	arachidic acid, eicosanoic acid, C 20:0
Be	behenic acid, dodosanoic acid, C 22:0
Lg	lignoceric acid, tetracosanoic acid, C 24:0
O	oleic acid, *cis*-9-octadecenoic acid, C 18:1
L	linoleic acid, *cis*, *cis*-9,12-octadecadienoic acid, C 18:2
Ln	linolenic acid, *cis*, *cis*, *cis*-9,12,15-octadecatrienoic acid, C 18:3
Ga	gadoleic acid, *cis*-11-eicosenoic acid, C 20:1

Glycerides

PPP	tripalmitin, CN = 48, NUFA = 0
PLO	palmito-linoleo-olein, CN = 52, NUFA = 2

Non-polar phases only allow Carbon Number separations of triglycerides

Capillary columns are the preferred analytical tool for triglyceride analysis since they yield shorter analysis times, higher efficiency, and better quantitation than packed column GC, HPLC, or SFC. On non-polar stationary phases, such as OV-1, SE-54, or 100% dimethyl polysiloxane, triglycerides are separated according to carbon number or molecular weight. However, no resolution is achieved for differences in unsaturation within the unsaturated fatty acids, i.e. POP, SOS, and POS would all appear as one peak. Triglyceride separation by degree of unsaturation, as well as carbon number, requires a highly polar stationary phase.

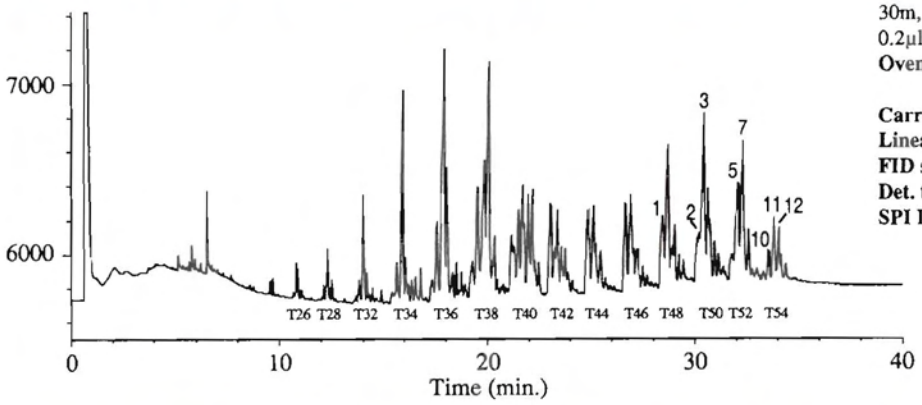
High thermal stability is also important for a triglyceride column. Triglyceride polarity increases with the degree of unsaturation in the fatty acid and with the total number of double bonds present. The triglyceride with the most double bonds has the highest polarity and the longest retention time. Therefore, high temperature is required to elute the higher polarity triglycerides and maintain short analysis times.

Lower response of high molecular weight triglycerides has been observed and was thought to be caused by thermal decomposition of triglycerides in the injection port. However, the decreased response can also be caused by both high molecular weight discrimination in the injection port and increased solute band broadening in the polar phase. Low bleed polar phases are also extremely important for triglyceride analysis because high column bleed can cause quantitation difficulties.

While polar stationary phases such as 50% phenyl/ 50% methyl offer the necessary selectivity, they have

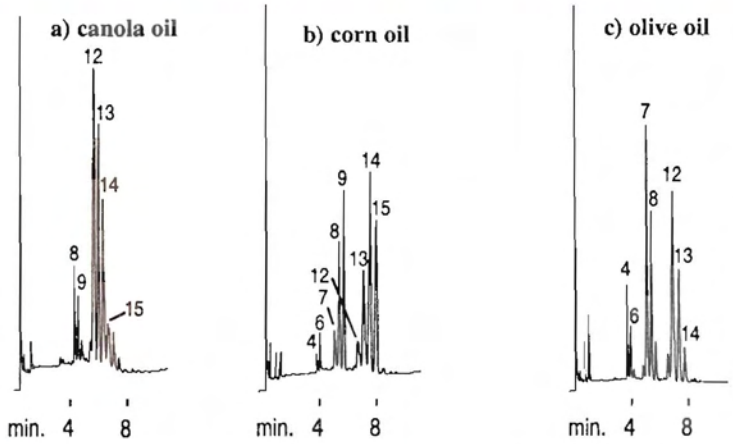
in this issue...

Figure 1 - Rtx™-65TG gives excellent resolution of butter fat triglycerides in under 32 minutes at 365°C.



30m, 0.25mm ID, 0.10µm Rtx-65TG (cat.# 17008)
0.2µl direct injection of 1% butter fat in isooctane
Oven temp.: 80°C (hold 1 min.) to 240°C @ 30°C/min. then to 360°C @ 4°C/min. (hold 5 min.)
Carrier Gas: hydrogen
Linear velocity: 70cm/sec.
FID sensitivity: 16 x 10^{-11} AFS
Det. temp.: 380°C
SPI Injector: high performance capillary insert 60°C, 300°C/min. to 400°C (hold 5 min.)

Figure 2 - Obtain fine structure resolution by degree of unsaturation with common oils on a 15-meter Rtx™-65TG.



15m, 0.25mm ID, 0.10µm Rtx-65TG (cat.# 17005)
0.8µl injection of sample
Oven temp.:
a) 355°C (hold 1 min.) to 365°C @ 1°C/min.
b) 350°C (hold .5min.) to 365°C @ 1°C/min.
c) 350°C (hold 1 min.) to 365°C @ .5°C/min.
Carrier Gas: hydrogen
Linear velocity: 50cm/sec.
Inj. & Det. temp.: 365°C
FID sensitivity: 8 x 10^{-11} AFS
Split ratio: 40:1

Peak List for Figures 2 & 3

1	PPP
2	PPS
3	PPO
4	POP
5	PSO
6	PLP
7	POO
8	PLO
9	PLL
10	SSO
11	OOS
12	OOO
13	OLO
14	OOL
15	LLL

traditionally suffered from low thermal stability. Phenyl/ methyl polysiloxanes typically exhibit lower maximum operating temperatures compared to methylsilicones due to stearic hindrance and lack of complete phase cross-linking.

Highly selective Rtx™-65TG yields separation by carbon number and unsaturated fatty acids of triglycerides

The chemists at Restek have coupled innovative polymer synthesis with advanced deactivation techniques to produce a highly polar stationary phase with high thermal stability ideal for triglyceride analysis. The Rtx™-65TG, 65% phenyl/ 35% methyl polysiloxane, is selective in resolving triglycerides according to degree of unsaturation, as well as carbon number, and has a maximum operating temperature of 370°C.

Figure 1 shows butter fat triglycerides run on a 30m, 0.25mm ID, 0.10µm Rtx™-65TG using a septum equipped programmable injector (SPI). The column shows excellent separation and peak symmetry of the triglycerides by both degree of unsaturation and carbon number. Fine structure resolution of T50 triglycerides, PPS/ PPO, T52 PSS/PSO, POO, and T54 SSS/SSO/SOO/OOO is obtained with minimal column bleed at 365°C (8 X 10^{-11} AFS) and a 32 minute analysis time. Figure 2 shows the analysis of canola, corn, and Italian olive oil on the Rtx™-65TG. The oils were analyzed on a 15m, 0.25mm ID, 0.10µm. Excellent separation of the unsaturates for T50, 52, and 54 is achieved in 8 minutes.

The Rtx™-65TG is ideal for triglyceride analysis. Separation by degree of unsaturation within unsaturated fatty acids, as well as carbon number, can be achieved in under 30 minutes with minimal column bleed at 365°C. Because Rtx™-65TGs are specially tested with a temperature programmed triglyceride test mixture, they are guaranteed for low column bleed and high efficiency. The Rtx™-65TG is available in both 15 and 30 meter lengths in 0.25, 0.32, and 0.53mm column IDs with a 0.10µm film thickness.

References

(1) Geeraert and Sandra, "Capillary GC of Triglycerides in Fats and Oils Using a High Temperature Phenyl Methyl Silicone Stationary Phase," *Journal of HRC & CC*, Vol, 8, Aug. 1985, pp. 415-419.

Rtx™-65TG Ordering Information

15m, 0.25mm ID, 0.10µm	cat.# 17005	$265
30m, 0.25mm ID, 0.10µm	cat.# 17008	$420
15m, 0.32mm ID, 0.10µm	cat.# 17006	$290
30m, 0.32mm ID, 0.10µm	cat.# 17009	$450
15m, 0.53mm ID, 0.10µm	cat.# 17007	$305
30m, 0.53mm ID, 0.10µm	cat.# 17010	$500

Hints for the Capillary Chromatographer

Quantitative Analysis

(This is Part 1 of a two part series on Quantitative Analysis. Part 2, in November's issue, will cover external standard and area percent techniques.)

PART 1: Internal Standard Technique

This quantitative technique requires the use of analyte specific calibration standards, plus the addition of internal and surrogate standards. It offers the highest quantitative accuracy compared to other techniques. It is commonly used for many environmental monitoring methods as well as forensic and clinical analyses.

When should the internal standard technique be used?

1) If each analyte has a unique detector response.
2) If detector response varies slightly over time.
3) If analyte retention times vary slightly from run-to-run.
4) If injection size varies slightly over time.

What are the differences between internal and surrogate standards?

Internal standards, as we will be defining them, are the standards that are added to the sample just prior to analysis. The same internal standards are also added to the calibration standards. Surrogate standards are added to the sample at the beginning of the sample preparation sequence. They are used to determine matrix effects and recoveries of target compounds during sample work-up.

What are the criteria in selecting internal and surrogate standards?

1) They should be chemically similar to the target compounds.
2) They must chromatograph similarly to the target compounds under the same run conditions.
3) They can be integrated consistently as a single entity either in the standard or unknown.

When performing analyses with routine GC detectors, the internal and surrogate standards should be completely resolved from all other analytes and interferences in the sample. When performing GC/MS analyses, these standards are typically deuterium or ^{13}C labeled analogs of the target compounds. Differentiating target compounds from labeled analogs can be accomplished by using quantitation ions based on their different molecular weights. Selection of the most appropriate internal and surrogate standards is critical to obtaining accurate quantitative results.

How many internal and surrogate standards should be used?

When target compounds encompass a wide range of boiling points, molecular weight discrimination can occur. Therefore, it is advisable to select multiple internal and surrogate standards which encompass a wide boiling point range and elute from the chromatographic column at various retention times (early, middle, late). If the sample contains multiple chemical classes (i.e. acids, bases, etc.) then the internal and surrogate standards chosen should also reflect the differences in functional groups.

If the sample is relatively simple in composition and the boiling point distribution is narrow, then single internal and surrogate standards will suffice. They should be chosen to elute near the mid-point of the analysis. The internal standard should be spiked into the unknown samples just prior to the GC analysis. The surrogate standards are spiked into the unknown samples prior to sample work-up. Both the internal and surrogate standards should be spiked into the calibration standards.

How are internal standards used for quantitation?

Calibration standards, which contain all the target compounds, are prepared at various concentration levels to bracket the working range of the detector. Each calibration standard is spiked with the identical amount of internal and surrogate standards. The calibration standards are analyzed in series (low concentration to high concentration) and the resultant retention times and peak areas are recorded for each analyte and the surrogate standards to calibrate the instrument.

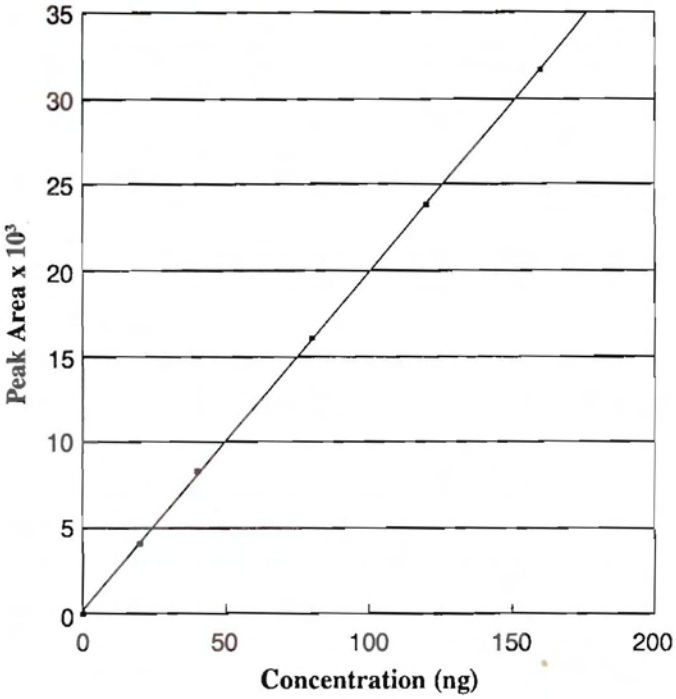
Relative retention times (RRTs) and relative responses factors can then be calculated for each target compound and the surrogate standards. Relative retention time windows are established for each compound and used to verify compound identity in the unknown sample. In the case of GC/MS, the mass spectra of each compound is compared to library spectra to confirm identity. Relative retention times are more accurate since they take into account slight shifts in absolute retention time that may occur from run-to-run. Figure 1 shows the calculation for RRT.

Figure 1

$$\text{Relative Retention Time (RRT)} = \frac{\text{Retention time (compound)}}{\text{Retention time (IS)}}$$

Responses or calibration curves are constructed for each compound using the peak area response plotted on the y-axis and the amount of the analyte injected on the x-axis (Figure 2). The linearity of the calibration curve will indicate whether the analyte responds proportionally to concentration over the range of operation or if the compound shows adsorptive effects at low concentration levels.

Figure 2 - Typical Calibration Curve



As an alternative to generating individual calibration curves for each compound, the analyst can calculate relative response factors (RRF). This is performed by taking into account the concentrations of the analytes and the internal standards. The calculations for RRFs are shown in Figure 3. If the amount of internal standard added to the calibration standards and samples are identical, the internal standard's concentration does not have to be included in any of the calculations. If the amount of internal standard added to the calibration standards varies with the analyte's concentration, then the variation must be accounted for in the calculations. If the compound exhibits linear response over the concentration range analyzed, a plot of concentration (x-axis) versus RRF (y-axis) will result in a relatively flat line (Figure 4). The RRFs can then be used to calculate the concentration of target compounds in samples (Figure 5).

Figure 3

$$\text{Relative Response Factors (RRF)} = \frac{\text{Area (compound) x Conc. (IS)}}{\text{Area (IS) x Conc. (compound)}}$$

Figure 4 - Relative Response Curve

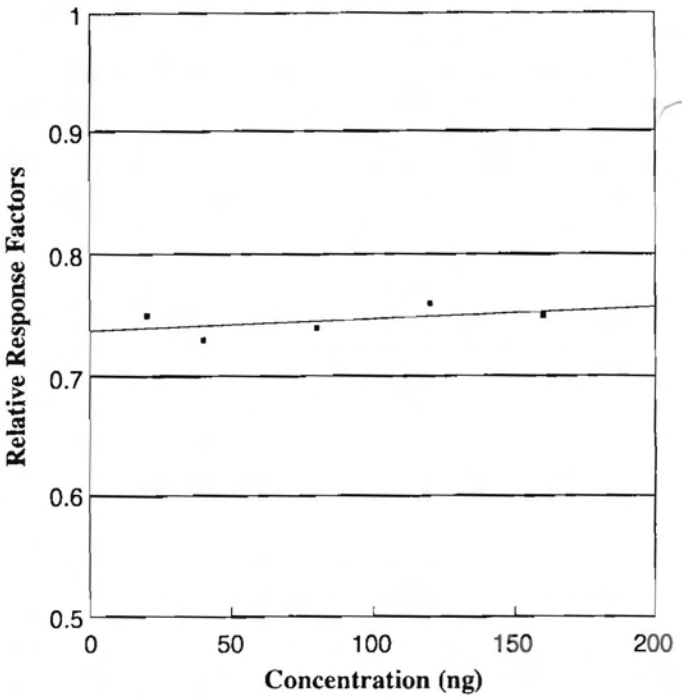


Figure 5

$$\text{Concentration} = \frac{\text{Area (unknown) x Conc. (IS)}}{\text{Area (IS) x RRF}}$$

How often is re-calibration necessary?

While the internal standard technique offers superior quantitative results, it is also advisable to run a continuing calibration check at regular intervals to determine if detector response or adsorption is affecting compound response factors. After a specified length of time (~once per 8 hours) or number of unknown samples (~10-25 samples), a mid-point calibration standard is analyzed. The RRFs are calculated and compared to the values obtained in the initial calibration of the instrument. If significant differences are noted, corrective action should be taken. Always recalibrate after performing routine maintenance procedures such as changing inlet sleeves, reinstalling or changing the column, or resetting the flow rates. While the internal standard technique offers a compensation mechanism for small changes in retention time, detector response or injection size, it does not eliminate the need for routine maintenance on the inlet system, detector, and column.

The method used for quantitation should be chosen based on sample type and the requirements of the analysis. The internal standard technique offers the most accurate quantitation, since it allows for minor variation in instrument response and injection size. The major drawback to this technique is the requirement of extensive calibration.

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- How column length affects resolution & analysis times
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- Column materials
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Analyzing Free Fatty Acids Without Derivatization

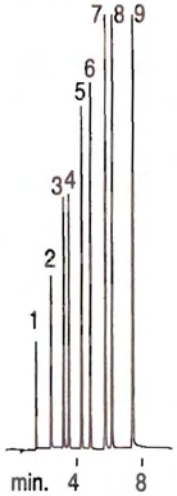
Dietary intake of fats has become an important concern for the health conscious public. Therefore, laboratories involved in analyzing food products are often faced with the difficult task of analyzing fatty acids. The lack of volatility coupled with their adsorptive nature has complicated gas chromatographic analysis of free fatty acids. As a result, many analysts derivatize fatty acids to their methyl ester state. Derivatization increases the volatility of free acids and reduces their activity making them more amenable to GC analysis. However, derivatization adds additional sample prep costs and some uncertainty to the analysis due to the possibility for sample loss or incomplete methylation. Therefore, many analysts would prefer to analyze fatty acids in their free state.

Volatile Fatty Acids

Several capillary column techniques have been developed for the analysis of volatile fatty acids. One approach is to use very thick film non-polar stationary phases for analyzing volatile free acids. Thick film columns are necessary to increase the sample capacity of non-polar phases since they have little affinity for highly polar acids. Figure 1 shows the analysis of C_2 to C_7 organic acids on a 30 meter, 0.53mm ID, 5.0µm Rtx™-1 column. Excellent separation of these volatile acids can be achieved in less than 8 minutes with minimal peak tailing.

Figure 1 - A thick film Rtx™-1 column resolves lower molecular weight free fatty acids.

COMPOUNDS	
1	acetic acid
2	propionic acid
3	isobutyric acid
4	n-butyric acid
5	isovaleric acid
6	n-valeric acid
7	isocaproic acid
8	caproic acid
9	heptanoic acid

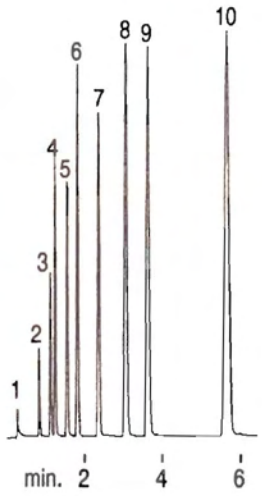


30m, 0.53mm ID, 5.0µm Rtx-1 (cat.# 10179)
0.2µl injection of a 10-20ng/µl free fatty acid standard in water
Direct injection using a Uniliner.
Oven temp.: 60°C to 180°C @ 15°C/min.
Inj. & det. temp.: 250°C
Carrier Gas: hydrogen
Linear velocity: 50cm/sec. (flow rate: 6cc/min.)
FID sensitivity: 4 x 10^{-11} AFS

Another approach for analyzing volatile fatty acids is to use highly polar bonded Carbowax® capillary columns that have been deactivated for acidic compounds. Figure 2 shows the analysis of C_1 to C_7 fatty acids on a 15 meter, 0.53mm ID, 1.0µm Stabilwax®-DA column. The strong affinity of the acids on this polar column results in excellent separation even with a 15 meter column. Analysis time is reduced to 6 minutes using this shorter length column. The acidic deactivation of the Carbowax® column produces excellent peak shape for these free fatty acids.

Figure 2 - A 15-meter Stabilwax®-DA column shows excellent resolution and reduced analysis time of volatile free fatty acids.

COMPOUNDS	
1	formic acid
2	acetic acid
3	propionic acid
4	isobutyric acid
5	n-butyric acid
6	isovaleric acid
7	n-valeric acid
8	isocaproic acid
9	caproic acid
10	heptanoic acid

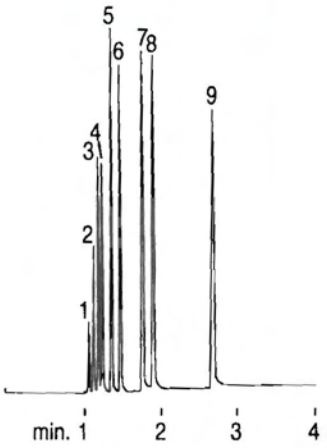


15m, 0.53mm ID, 1.0µm Stabilwax®-DA (cat.# 11052)
0.2µl injection of a 10-20ng/µl free fatty acid standard in water
Direct injection using a Uniliner.
Oven temp.: 145°C isothermal
Inj. & det. temp.: 250°C
Carrier Gas: hydrogen
Linear velocity: 80cm/sec. (flow rate: 10cc/min.)
FID sensitivity: 4 x 10^{-11} AFS

Moderately polar stationary phases can also be used for the analysis of volatile free fatty acids. Figure 3 shows the analysis of C_2 to C_7 organic acids on a 30 meter, 0.25mm ID, 0.25µm Rtx™-200 column. This trifluoropropylmethyl stationary phase shows good separation and peak shape of these volatile acids and the analysis time is less than 3 minutes, making the Rtx™-200 an excellent screening column.

Figure 3 - Fast analysis of low molecular weight free fatty acids can be achieved with an Rtx™-200 column.

(same peak list as Figure 1)



30m, 0.25mm ID, 0.25µm Rtx-200 (cat.# 15023)
0.8µl split injection of a free fatty acid standard
Concentration approximately 10 to 20ng/µl

Oven temp.:	90°C isothermal
Inj. & det. temp.:	250°C
Carrier Gas:	hydrogen
Linear velocity:	40cm/sec. (flow rate: 1.4cc/min.)
FID sensitivity:	4 x 10^{-11} AFS
Split vent:	40cc/min.

Saturated & Unsaturated Fatty Acids

Polar stationary phases are also capable of resolving saturated and unsaturated fatty acids. Figure 4 shows the analysis of saturated and unsaturated fatty acids on a 30 meter, 0.53mm ID, 0.25µm Stabilwax®-DA column. Palmitic acid (C16:0) can easily be resolved from Palmitoleic acid (C16:1), and Stearic acid (C18:0) can be resolved from Oleic (C18:1), Linoleic (C18:2), and Linolenic acid (C18:3) on this column.

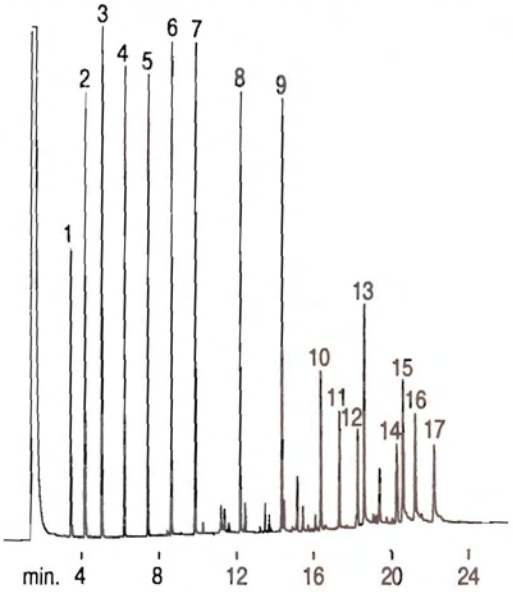
Methylation of fatty acids for GC analysis may not always be necessary. Several options are available for analyzing fatty acids in their free form using capillary columns. Column selection will depend on the molecular weight range of fatty acids and the resolution required to separate saturated from unsaturated forms.

Product Listing

Rtx™-1			
	30m, 0.53mmID, 5.0µm	cat.#10179	$455
Rtx™-200			
	30m, 0.25mmID, 0.25µm	cat.#15023	$400
Stabilwax®-DA			
	15m, 0.53mmID, 1.0µm	cat.#11052	$260
	30m, 0.53mmID, 0.25µm	cat.#11025	$455

Figure 4 - Saturated and unsaturated free fatty acids can be separated on a 30-meter Stabilwax®-DA column.

COMPOUNDS	
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 - enanthic 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



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

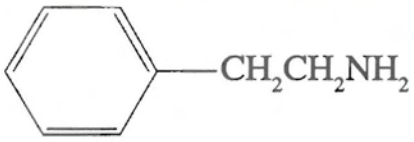
Oven temp.:	100°C (hold 2 min.) to 250°C @ 8°C/min.
Inj. & det. temp.:	280°C
Carrier Gas:	helium
Linear velocity:	40cm/sec. (flow rate: 5.2cc/min.)
FID sensitivity:	8 x 10^{-11} AFS

Clinical Corner

Analysis of Sympathomimetic Amines

Amphetamine, methamphetamine and other structurally related compounds belong to the class of compounds known as sympathomimetic amines. The base compound for this class is beta-Phenylethylamine (Figure 1). Substitutions can be made on either the aromatic ring, the alpha and beta carbons, or on the terminal amino group to produce a wide variety of closely related compounds, both structurally as well as pharmacologically. Physiological effects of sympathomimetic amines include central nervous system (CNS) stimulation, increased heart and respiratory rates and appetite suppression.

Figure 1 - Base structure for sympathomimetic amines.



Amphetamine and methamphetamine, as drugs of abuse, were very popular in the late 60's and have been experiencing a resurgence in popularity over the last decade. Diversion of legitimately manufactured material from pharmaceutical companies had been a major supply source for illicit drug dealers. Cutbacks in methamphetamine production in the late 1960's and the classification of amphetamine as a Schedule II controlled substance in 1970 severely restricted the amount of pharmaceutically produced material that was entering the illicit drug pipeline. In response to the decrease in supply, many drug dealers turned to the clandestine synthesis of amphetamine, methamphetamine and structurally related analogs. Most clandestine labs produce methamphetamine because of the simplicity of the reaction, the ready availability of raw materials and high product yields. However, stimulants sold on the street can vary considerably in their content and purity. Substitution of other sympathomimetic amines and even unrelated, non-stimulatory substances is a frequent occurrence.

Forensic analyses for amphetamine and methamphetamine are focused on two main areas: the analysis of street drug samples and the testing of biological specimens for the presence of controlled substances. The range of substances encountered in street preparations has made the identification of these controlled substances a very complex analytical problem.

Substituted phenylethylamines are fairly polar and quite basic due to the terminal amino group in the molecule. These physical characteristics yield a molecule that is difficult to chromatograph on stationary phases that have not been base modified. The preferred packing material for this analysis had been Apiezon L modified with KOH. Since most capillary columns do not contain base modifiers, analysis of amphetamine and methamphetamine by capillary GC can produce peak tailing and adsorption depending on the degree of inertness of the column. To overcome these problems, derivatization of the terminal amino group to form a less polar, more neutral molecule with better chromatographic properties is the preferred method of sample preparation. As with all compounds that have a high potential for sample adsorption, we recommend the use of deactivated glassware and sample vials during sample preparation. Acylation, using reagents like TFAA and HFBA, and silylation, using TMCS or BSTFA*, are the two most common derivatization techniques. In addition to improving the chromatographic characteristics of these amines, derivatization can also improve the lower detection limit and aid in the identification of the different sympathomimetic amines. The fluoroacyl derivative can be analyzed by ECD with detection limits in the low ng/ml range. Confirmational analysis by GC/MS is also enhanced by derivative formation. Comparison of ion ratios using unique, high mass fragments produced by derivatized analytes provides a higher degree of confidence in confirming the identity of sympathomimetic amines than using low mass ions from underivatized compounds.

Derivatized sympathomimetic amines can be analyzed on low to intermediate polarity columns like the Rtx™-5. Figures 2 and 3 show the analysis of derivatized samples of amphetamine, methamphetamine and a set of substances commonly encountered when analyzing for stimulants. Two different derivatizing reagents, TFAA and HFBA, were employed. Limits of detection in the low nanogram range on-column are achieved because of the excellent peak shape and overall response. All compounds analyzed in Figures 2 and 3 are present at a concentration of 2.5ng/µl in the final extract. Using a 5ml sample size and a final reconstitution volume of 100µl, this would correspond to a pre-extraction concentration of 50ng/ml in a biological specimen, well below the NIDA cutoff of 100ng/ml. A side benefit of comparing two different derivatives can be the difference in resolution between closely eluting compounds. Peaks 2 and 3, phentermine and phenylpropanolamine, are completely resolved by using HFBA versus TFAA as the derivatizing reagent.

Critical factors for successful sympathomimetic amine analysis include derivatization of the amine group, analysis of derivatized compounds using a low polarity highly inert capillary column and the use of deactivated glassware and sample vials.

Figure 2 - Deactivatization improves peak

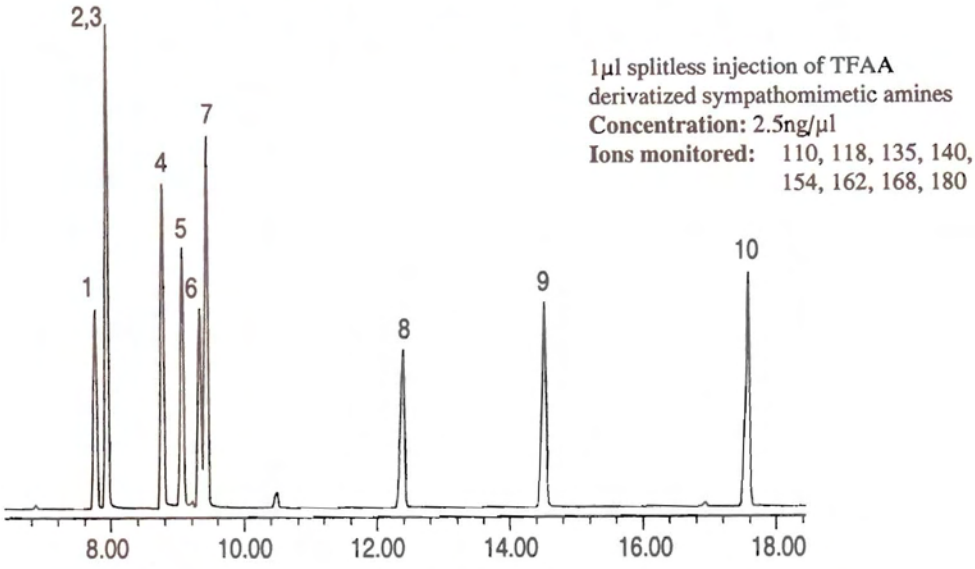
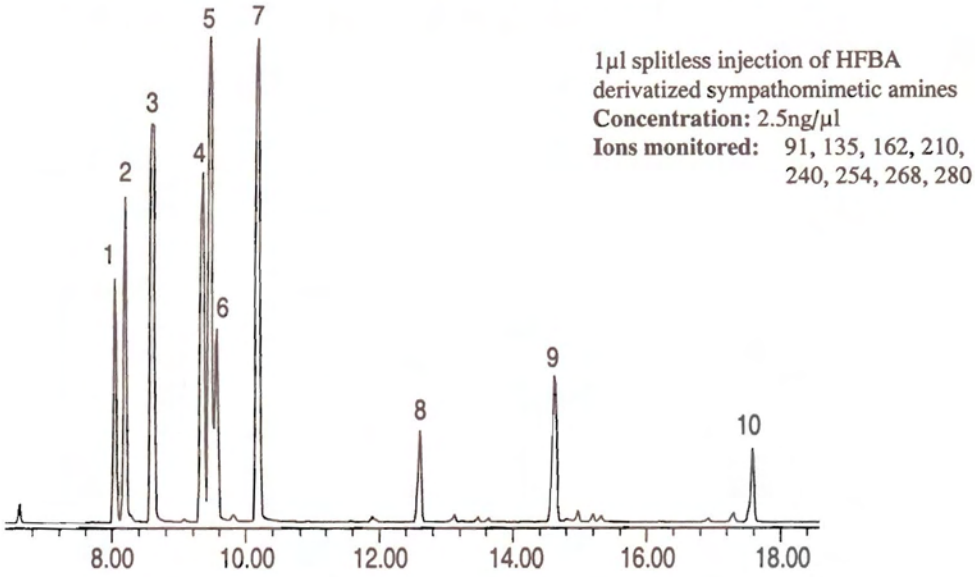


Figure 3 - Improved separation of sympathomimetic amines using HFBA.



Peak List & Conditions for Figures 2 & 3

30m, 0.25mm ID, 0.25µm Rtx-5 (cat.# 10223)

Oven temp.:	40°C (hold 1 min.) to 150°C @ 30°C/min. then to 300°C @ 5°C/min.		
Inj. Temp.:	225°C	**Detector:**	MSD
Carrier Gas:	helium	**Splitless hold time:**	1 min.
Ionization:	EI	**Electron energy:**	70ev
Scan rate:	1.08 cycles/sec.		
Interface temp.:	275°C		

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

TFAA = trifluoroacetic anhydride, **HFBA** = heptafluorobutyric anhydride, **TMCS** = trimethylchlorosilane, **BSTFA** = N,O-bis(trimethylsilyl)trifluoroacetamide

Standards Spotlight

Chemical Standards for EPA Method 8240

• Quantitative calibration, surrogate, and internal standard mixtures • Convenient packaging • • Data packs available for audit documentation •

EPA Method 8240 is used to quantitate volatile organics from a variety of solid waste samples. Typically, laboratories perform this analysis using purge & trap GC/MS procedures. In limited cases, direct injection GC/MS techniques may be used.

We have attempted to include as many analytes as possible from the target compound list in this method. Work is currently being performed to expand the products listed below. Many of the analytes are included in the Appendix IX ground water monitoring list.

8240 Volatiles Mix #1

2000µg/ml each in 1ml P&T grade methanol

allyl chloride	ethyl methacrylate
methacrylonitrile	methyl methacrylate
2-picoline	propionitrile
pyridine	styrene
acetonitrile	

Cat.# 30077 $25 ea.
30077-500 $55 ea. w/data pack
30177 $225 10pk. w/data pack

8240 Volatiles Mix #2

2000µg/ml each in 1ml P&T grade methanol

cis-1,4-dichloro-2-butene
trans-1,4-dichloro-2-butene
1,4-dioxane
iodomethane
pentachloroethane
1,2-dibromoethane
1,2-dibromo-3-chloropropane

Cat.# 30078 $25 ea.
30078-500 $55 ea. w/data pack
30178 $225 10pk. w/data pack

Method 8240 Volatiles Kit

Contains 1ml (1 ampul) of each of the following mixtures:

30042* - 502.2 Calibration Mix #1
30006* - VOA Calibration Mix #1
30007* - VOA Calibration Mix #2
30056* - 8010A Calibration mix #2
30077 - 8240 Volatiles Mix #1
30078 - 8240 Volatiles Mix #2
30011* - VOA Internal Standard Mix
30005* - VOA Matrix Spike Mix
30004* - VOA Surrogate Spike Mix
30075* - 8240/60 System Performance Check Mix

Cat.# 30079 $240 ea.
30079-500 $460 ea. w/data pack

*For a complete description of each mixture, please refer to Restek's Environmental Catalog.

Recommended column for this method:

60m, 0.53mm ID, 3.0µm Rtx™-502.2 (cat.# 10909)

Restek is Committed to Providing the Highest Quality Chemical Standards

We are constantly monitoring method modifications proposed by government regulatory agencies. Should additional method improvements be introduced, chemical standard mixtures will be modified to meet customer requirements.

Chemical Standards for EPA Method 8040

• *Quantitative calibration and surrogate standard mixtures* • *Convenient packaging* • *Data packs available*

EPA Method 8040 is used for the quantitative determination of phenolic compounds from various waste samples. The method is performed using either GC/FID or GC/ECD depending upon analyte concentration. If low detection levels are required, GC/ECD analysis is required after derivatization of the analytes using pentafluorobenzyl bromide (PFB).

Method 8040 does not recommend specific internal standards for use. The analyst must select an appropriate internal standard (if desired) based upon knowledge of the sample. Deuterated analogs of analytes may not be used due to chromatographic coelution problems.

8040 Phenols Mix #1
2000µg/ml each in 1ml isopropanol

4-chloro-3-methylphenol
2,4-dichlorophenol
2-methyl-4,6-dinitrophenol
3-methylphenol
2-nitrophenol
4-nitrophenol
pentachlorophenol
phenol
2,4,6-trichlorophenol

Cat.# 31088 $25 ea.
31088-500 $55 ea. w/data pack
31188 $225 10pk. w/data pack

8040 Phenols Mix #2
2000µg/ml each in 1ml isopropanol

2-chlorophenol
2,6-dichlorophenol
2,4-dimethylphenol
2,4-dinitrophenol
2-methylphenol
2,4,5-trichlorophenol
2,3,4,6-tetrachlorophenol
4-methylphenol
2-sec-Butyl-4,6-dinitrophenol (Dinoseb)

Cat.# 31089 $25 ea.
31089-500 $55 ea. w/data pack
31189 $225 10pk. w/data pack

8040 Surrogate Mix
2000µg/ml each in 1ml methanol

2-fluorophenol
2,4,6-tribromophenol

Cat.# 31090 $25 ea.
31090-500 $35 ea. w/data pack
31190 $225 10pk. w/data pack

Additional Surrogates for EPA Methods:

2-fluorobiphenyl
2000µg/ml in 1ml methylene chloride

Cat.# 31091 $25 ea.
31091-500 $35 ea. w/data pack
31191 $225 10pk. w/data pack

1-fluoronaphthalene
2000µg/ml in 1ml methylene chloride

Cat.# 31092 $25 ea.
31092-500 $35 ea. w/data pack
31192 $225 10pk. w/data pack

Recommended columns for this method:

30m, 0.25mm ID, 0.25µm XTI™-5 (cat.# 12223)
30m, 0.32mm ID, 1.0µm XTI™-5 (cat.# 12254)
30m, 0.53mm ID, 1.5µm XTI™-5 (cat.# 12270)

EPA 8000 Series Mixes Available from Restek

Method	Description	Calibration Std(s). Catalog Number	Recommended Surrogates
8060	Phthalate esters	31031	
8070	Nitrosamines	31032	
8080	Organochlorine Pesticides & PCBs	31012 (pesticides) 32005 (toxaphene) 32006 - 32012 (PCBs) 32021 (technical chlordane)	32027 (TCMX) 32029 (DCB)
8090	Nitroaromatics & Cyclic Ketones	31033	31091
8100	PAHs (PNAs)	31011	31091 31092
8110	Haloethers	31034	
8120	Chlorinated Hydrocarbons	31035	

To order any Restek product, call 800-356-1688 (ext.3).

Peak Performers

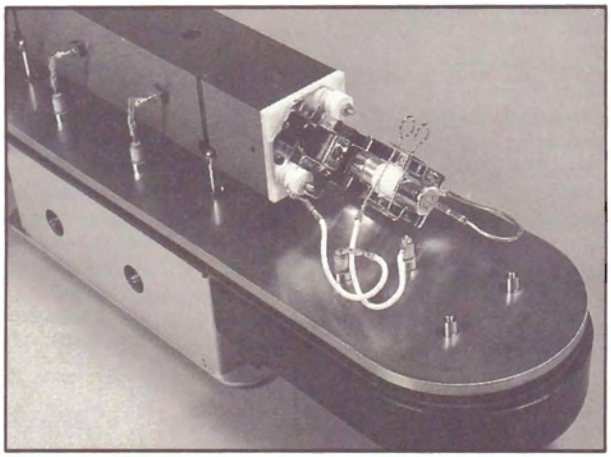
New HP 5971A MSD Electron Multiplier Provides Improved Sensitivity

- High output current capabilities
- Lifetime extended to 6,000-10,000 microamp hours
- Can be operated continuously at temperatures up to 200°C
- High thermal and physical shock capacity in the event of accidental venting.

This new multiplier design is exclusive for the HP 5971A MSD.* It features a cartridge body formed of pressed monolithic ceramic for exceptional thermal and electrical conductivity increasing sensitivity approximately two and a half times over other designs. The ceramic cartridge body eliminates dynode degrading impurities which can reduce the life of conventional glass multipliers.

This new snap-out/snap-in design simplifies replacement and reduces expensive downtime since the mounting hardware is permanently installed, assuring proper ion-optic alignment every time.

The initial purchase of the multiplier requires the mounting structure and the ceramic multiplier. Future replacements require only the ceramic multiplier.

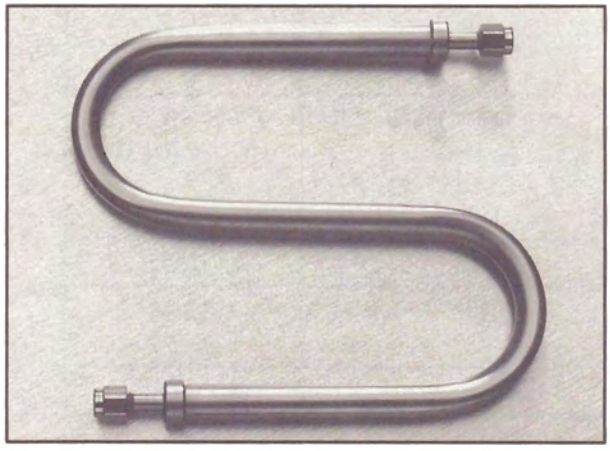


Electron Multiplier Mounting Structure and Ceramic Multiplier: cat.# 20642, $795 ea.

Replacement Multiplier Only: cat.# 20639, $695 ea.

* Similarly designed multipliers are available for other mass spectrometers upon request.

Restek's Molecular Sieve "S" Trap



Trace moisture in carrier gas can increase background noise and cause erratic baseline rise for sensitive detectors such as ECDs and mass spectrometers. FID baselines are more stable and column lifetime is increased when molecular sieve traps are used in front of oxygen traps. Because moisture reacts with most oxygen traps and decreases trap efficiency, molecular sieve traps also increase oxygen trap lifetime.
While glass indicating moisture traps offer convenience, they are fragile and add to environmental waste disposal problems since they are discarded when spent. Restek's new molecular sieve "S" moisture trap offers the best alternative. The "S" design allows the trap to be regenerated in the GC oven (HP5890, Varian 3000 series, etc.) so you never need to discard it. The metal body and single end fitting design eliminate the possibility of leakage or breakage. The built-in 40 micron frits prevent particulate contamination from damaging regulators or needle valves. Each molecular sieve trap is individually activated to insure maximum reactivity for removing moisture.

"S" Rechargeable Molecular Sieve Trap, 1/8" female Swagelok-type fittings: cat.# 20686, $120 ea.
"S" Rechargeable Molecular Sieve Trap, 1/4" female Swagelok-type fittings: cat.# 20685, $120 ea.

Deactivated Inlet Sleeves

Quartz Inlet Sleeves for HP GCs

Restek now offers 2 and 4mm ID Quartz split/splitless sleeves for HP GCs. The high melting temperature of quartz allows easy pyrolysis of sample residue in a muffle furnace or by heating in a flame. Because of the high purity of quartz, the sleeves stay inactive to many compounds even when the deactivation layer has been pyrolyzed. The 2mm ID sleeve replaces HP part# 5181-8818 or 18740-80220 and should be used for sample sizes under 2µl. The 4mm sleeve (not offered by HP) can be used with sample injection sizes up to 4µl. Remember, always pack straight splitless sleeves with wool when using rapid HP autosamplers.

2mm ID quartz sleeve: cat.# 20914, $20 ea.
cat.# 20915, $80/5-pk.

4mm ID quartz sleeve: cat.# 20912, $20 ea.
cat.# 20913, $80/5-pk.

4mm Splitless Sleeve for Varian GCs

Restek's new 4mm splitless sleeve design for Varian GCs offers several advantages over the standard 2mm splitless sleeve. One of these advantages is a larger buffer volume to contain the sample vaporization for sample sizes greater than 2µl. The 4mm ID also allows convenient installation of wool or beads to trap non-volatile residue or increase sample vaporization. As with all of Restek's sleeves, our proprietary deactivation guarantees excellent inertness to minimize active sample component decomposition.

cat.# 20904, $24 ea.
cat.# 20905, $92/5-pk.
cat.# 20906, $426/25-pk.

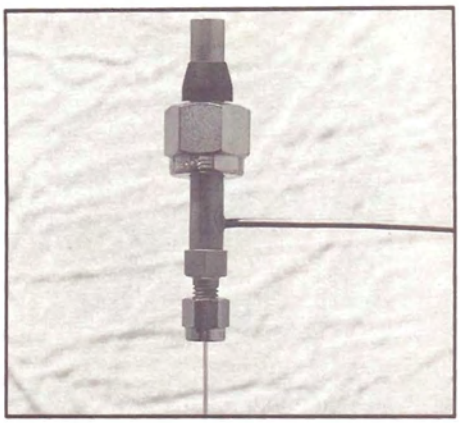
Auto SYS Cyclosplitter® for PE

This new Auto SYS Cyclosplitter® from Restek provides excellent sample vaporization and minimizes molecular weight discrimination by thoroughly mixing the sample through the cylindrical glass screw. The glass screw also traps non-volatiles, permitting up to five times as many injections before cleaning is required. The Cyclosplitter® sleeve increases column lifetime and decreases maintenance time. Designed to meet the manufacturer's exact specifications, the Cyclosplitter® is an excellent alternative to inlet packing materials and conventional splitter designs.

Auto SYS Cyclosplitter® for PE:
cat.# 20910, $40 ea.
cat.# 20911, $160/5-pk.

ECD/FID Dual Purpose Make-up Gas Fitting for HP

Now with a better make-up gas profile!



The Restek wizards improved the ECD make-up gas fitting for HP. Incorporating a better make-up gas profile, the new design uses a straight section of inert Silcosteel® tubing as the capillary column guide. The Silcosteel® replaces the fragile fused silica column guide found in the original equipment design. To use the ECD make-up gas fitting with an FID, simply remove the Silcosteel® guide and the FID jet fits neatly inside the fitting. A direct HP replacement fitting with the special end connector is also available for GCs that were factory equipped with make-up gas. The replacement fitting includes a 1/4" nut and graphite ferrule and a 1/16" nut with 0.4mm ID graphite capillary ferrule. A complete make-up gas kit is also available for plumbing GCs not factory equipped with make-up gas. The complete kit includes a make-up gas fitting, 1/4" nut and ferrule, 1/16" nut and 0.4mm ID graphite capillary ferrule, and a high quality fine metering valve.

HP ECD/FID make-up gas replacement fitting: cat.# 21301, $125 ea.
HP ECD/FID make-up gas kit: cat.# 21300, $225 ea.
HP ECD/FID Silcosteel® guide: cat.# 21302, $15/2-pk.

FREE Technical Literature

Restek has several free technical bulletins available that are designed to solve your chromatographic problems and answer your GC questions.

Helpful Hints for Analyzing Volatile Organics
Guide to Minimizing Septa Problems
Operating Hints for Split/Splitless Injectors
A Guide When Injecting Dirty Samples

If you would like to receive copies of any of our bulletins, call 1-800-356-1688.

To order any Restek product, call 800-356-1688 (ext. 3).

Restek Wins Awards for Manufacturing Excellence

We have recently received the 1991/92 National Business Incubator Graduate of the Year Award and Central/Eastern Pennsylvania's Manufacturing Entrepreneur of the Year Award.

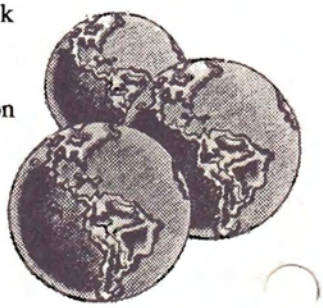
The National Business Incubator Graduate of the Year Award is presented by Coopers & Lybrand and the National Incubator Association to a company that started in a business incubator and has shown high employment growth and demonstrated commitment to the community.

Paul Silvis, Restek's president, was the recipient of the Central/Eastern Pennsylvania Manufacturing Entrepreneur of the Year Award. This award, founded by Ernst & Young and sponsored by *Inc.* magazine and Merrill Lynch, is given to entrepreneurs who have demonstrated excellence in such areas as innovation, financial performance and personal commitment to their business and communities. This regional award makes Mr. Silvis eligible to become the National Entrepreneur of the Year.

We are very proud of our achievements, but our success would not be possible without the support of our loyal customers. *Thank you for your support!*

Environmental Chemists . . .

Restek's Environmental Catalog is Here



If you do environmental work, Restek has the catalog for you. Our new Environmental Chromatography Products Catalog contains application chromatograms for numerous EPA methods, a listing of high quality calibration standards, and many new capillary columns and products for environmental analysis. Each catalog includes an EPA Method reference poster with operating hints and product recommendations for 500, 600, and 8000 Series Methods. To request a free copy of this informative catalog, call 800-356-1688.

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THE RESTEK ADVANTAGE

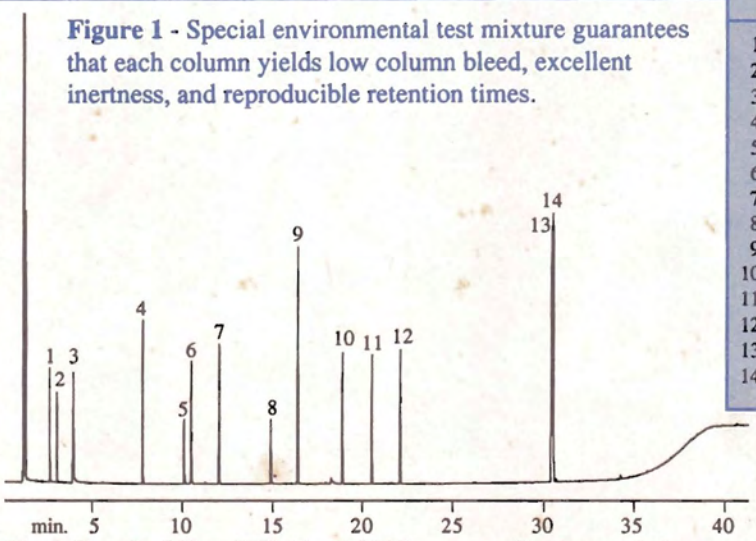
Improving the Analysis of Priority Pollutants

New polymer technology guarantees inertness and thermal stability

EPA Methods 625 and 8270 employ GC/MS detection to determine the concentration of semi-volatile organic compounds in extracts from waste water, soils, and solid waste matrices. The use of capillary columns in conjunction with these methods permits the simultaneous analysis of both acid and base/ neutral extracts in a single injection. This technique increases sample throughput since many compounds can be analyzed simultaneously. The inertness and resolution that can be achieved with capillary columns also improves quantitative accuracy.

The primary analytical column recommended by the EPA for the analysis of semi-volatile pollutants is a 5% diphenyl/ 95% dimethyl polysiloxane liquid phase (SE-54). Restek has developed the XTI®-5 stationary phase to meet the demanding requirements of semi-volatile analysis. Using new XTI® polymer technology, columns are produced that exhibit significantly reduced bleed (even at 360°C), are inert to active environmental compounds, and possess high efficiency to resolve closely eluting isomer pairs.

Figure 1 - Special environmental test mixture guarantees that each column yields low column bleed, excellent inertness, and reproducible retention times.



COMPOUNDS

1. 1,2-hexanediol
2. N-nitroso-di-n-propyl amine
3. benzoic acid
4. n-tetradecane
5. 2,4-dinitrophenol
6. 4-nitrophenol
7. 4-nitroaniline
8. pentachlorophenol
9. carbazole
10. eicosane
11. heneicosane
12. docosane
13. benzo(b)fluoranthene
14. benzo(k)fluoranthene

30m, 0.25mm ID, 0.25µm XTI®-5 (cat.# 12223)
1µl split injection, 7-17ng/compound on-column
Oven temp.: 100°C to 360°C @ 6°C/min. (hold 10 min.)
Inj./det. temp.: 360°C
Carrier gas: hydrogen
Linear velocity: 40cm/sec. set @ 100 (flow rate: 1.4cc/min.)
FID sensitivity: 8×10^{-11} AFS
Split vent: 40:1

in this issue...

Special Environmental Test Mixture Ensures XTI®-5 Meets EPA Method Criteria

Each XTI®-5 column is tested with a specially designed, temperature programmed test mix to meet strict performance requirements. The test mixture contains some of the most sensitive environmental compounds for testing inertness and reproducibility, and is temperature programmed to monitor the bleed at the column's maximum operating temperature. Figure 1 shows the XTI® environmental test mixture on a 30m, 0.25mm ID, 0.25µm XTI®-5. Excellent response of active pollutants such as 4-nitrophenol, 2,4-dinitrophenol, and pentachlorophenol is achieved on the XTI®-5 column. Additionally, high column efficiency is demonstrated by the resolution of the PNA isomers, benzo(b) and (k)fluoranthene.

XTI®-5 Provides the Inertness, Efficiency, and Thermal Stability Required for GC/MS Methods

Semi-volatile pollutants encompass a broad range of compound classes. The analysis of substituted phenols at low concentrations is difficult because they can be adsorbed on active surfaces or react with stationary phases that are too basic. Many capillary columns do not have the proper inertness or acidity for the trace analysis of phenols. Often the response of phenols is used as an indication of capillary column inertness for semi-volatile pollutants.

Column efficiency can be demonstrated with the analysis of polynuclear aromatic hydrocarbons (PNAs). The analysis of PNAs is complicated because there are several pairs of structural isomers with similar boiling points. Therefore, many of these compounds cannot be distinguished by their mass spectra. The high resolution capabilities of capillary columns allow these isomers to be accurately quantified. Another problem encountered during PNA analysis is that resolution of these compounds is often lost because they elute isothermally at the end of a temperature programmed run. Because XTI®-5 columns have extended thermal stability, even the high boiling isomers can now be eluted under temperature programmed run conditions.

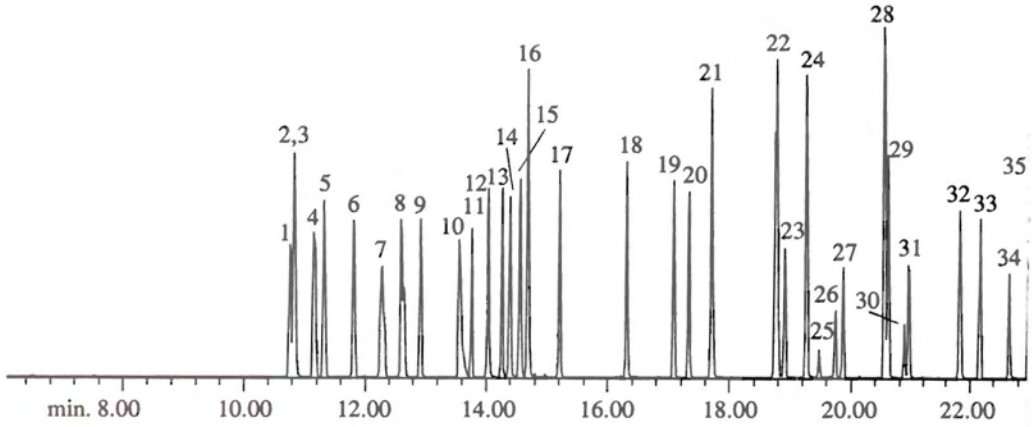
Figure 2 shows the analysis of the 52 acid and base/neutral semi-volatile pollutants listed in EPA Method 625 on a 30 meter, 0.25mm ID, 0.50µm XTI®-5. The 0.50µm film thickness increases sample capacity allowing calibration up to 200ng without overload. This 0.50µm XTI®-5 column has a 330°C maximum operating temperature, which reduces the analysis time to 38 minutes.

Low Bleed XTI®-5 Improves Quantitative Accuracy and Sensitivity

Thermal stability is extremely important when analyzing trace levels of high molecular weight semi-volatile compounds. Column bleed can present several problems when analyzing environmental samples. The rise in baseline associated with column bleed can lead to inaccurate quantitative results, confuse spectral interpretation, decrease sensitivity and, in extreme cases, cause mis-identification.

Semi-volatile organic pollutants encompass a broad range of compound classes. The monitoring requirements established by the EPA coupled with the complexity of the analysis demand the use of capillary columns that have high inertness, efficiency, and thermal stability. Recent polymer technology has been developed that substantially improves the capillary columns used in semi-volatile pollutant analysis. Columns produced with XTI® technology exhibit increased inertness and increased thermal stability, resulting in improved response of active compounds, lower column bleed, and faster analysis times. ■

Figure 2 - XTI®-5 provides excellent inertness, efficiency, and thermal stability for acid & base/neutral semi-volatile pollutants.



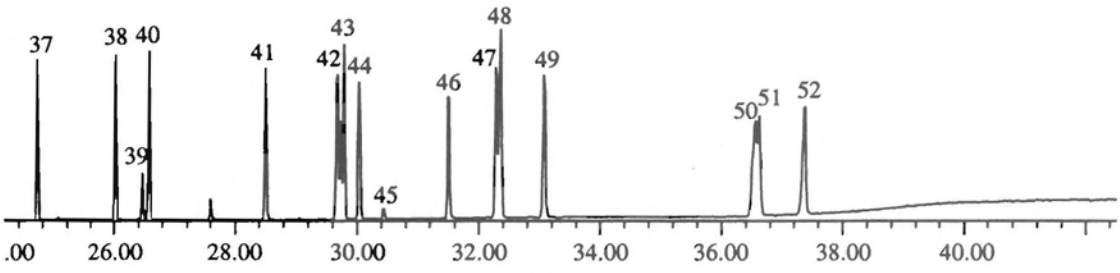
30m, 0.25mm ID, 0.50m XTI®-5 (cat.# 12238)
0.5µl splitless injection of EPA Method 625 Kit (cat.# 31036), on-column concentration: 100ppm

Oven temp.:	40°C (hold 6 min.) to 300°C @ 10°C/min. (hold 4 min.), then to 330°C @ 10°C/min.				
Inj. temp.:	280°C	**Carrier gas:**	helium	**Splitless hold time:**	0.5 min.
Detector:	MSD	**Linear velocity:**	80cm/sec. set @ 40°C	**Scan mode:**	TIC
Det. temp.:	300°C	**Purge flow:**	25mls/min.	**Scan range:**	35-500 AMU

Product Listing

XTI™-5 (bonded 5% phenyl - extended temperature and inertness) Polymer stable to 360°C

	df (µm)	temp. range	15-meter	30-meter
0.25mm ID	0.25	-60 360°C	cat.# 12220	cat.# 12223
	0.50	-60 330°C	cat.# 12235	cat.# 12238
	1.00	-60 325°C		cat.# 12253
0.32mm ID	0.25	-60 360°C		cat.# 12224
	0.50	-60 330°C		cat.# 12239
	1.00	-60 325°C		cat.# 12254
0.53mm ID	0.50	-60 330°C		cat.# 12240
	1.00	-60 325°C		cat.# 12255
	1.50	-60 320°C		cat.# 12270



BNAs for EPA 625	
1	phenol
2	bis(2-chloroethyl)ether
3	2-chlorophenol
4	1,3-dichlorobenzene
5	1,4-dichlorobenzene
6	1,2-dichlorobenzene
7	bis(2-chloroisopropyl)ether
8	hexachloroethane & N-nitroso-di-n-propylamine
9	nitrobenzene
10	3,5,5-trimethyl-2-cyclohexen-1-one(isophorone)
11	2-nitrophenol
12	2,4-dimethylphenol
13	bis(2-chloroethoxy)methane
14	2,4-dichlorophenol
15	1,2,4-trichlorobenzene
16	naphthalene
17	hexachloro-1,3-butadiene
18	4-chloro-3-methylphenol
19	hexachloro-1,3-cyclopentadiene
20	2,4,6-trichlorophenol
21	2-chloronaphthalene
22	acenaphthylene
23	2,6-dinitrotoluene
24	acenaphthene
25	2,4-dinitrophenol
26	4-nitrophenol
27	2,4-dinitrotoluene
28	fluorene
29	4-chlorophenylphenyl ether
30	diphenylamine
31	2-methyl-4,6-dinitrophenol
32	4-bromophenylphenyl ether
33	hexachlorobenzene
34	pentachlorophenol
35	phenanthrene
36	anthracene
37	di-n-butyl-phthalate
38	fluoranthene
39	benzidine
40	pyrene
41	butyl benzyl phthalate
42	benzo(a)anthracene
43	3,3'-dichlorobenzidine
44	chrysene
45	bis(2-ethylhexyl)phthalate
46	di-n-octyl phthalate
47	benzo(b)fluoranthene
48	benzo(k)fluoranthene
49	benzo(a)pyrene
50	indeno(1,2,3-cd)pyrene
51	dibenzo(a,h)anthracene
52	benzo(g,h,i)perylene

ezGC™ and *Pro ezGC™*

for Fast, Economical GC Method Development

The March 1993 issue of *The Restek Advantage* previewed *ezGC™*, a new software program that uses computer modeling to accurately simulate changes to a GC analysis. The software calculates the peak widths and retention times for a given set of chromatographic conditions and then displays the resulting chromatogram. In addition, the software predicts the optimum temperature program for a given analysis that provides baseline resolution in the shortest time. A chromatographer can see, within seconds, the effect of changes in column dimensions, carrier gas, and operating conditions instead of spending hours or days in the laboratory. The *ezGC™* software improves column selection, optimizes peak resolution, minimizes analysis times, and greatly increases laboratory productivity. Both method development labs and analytical labs performing routine analysis can benefit from *ezGC™*. The following examples demonstrate the capabilities of *ezGC™* using a typical set of columns and chromatographic conditions.

Let *ezGC™* optimize your GC method for resolution in the shortest analysis time

To begin optimizing a GC method, first obtain two sets of retention times for the components of interest at two different temperature programs. In the following example, retention times were collected from two injections of a volatile fragrance mixture using temperature program rates of 3 and 8°C/min. Next, the chromatographic conditions and retention times were entered using the menu driven screens of the *ezGC™* program.

Figure 1 - Easily enter information into *ezGC™* from menu driven screens.

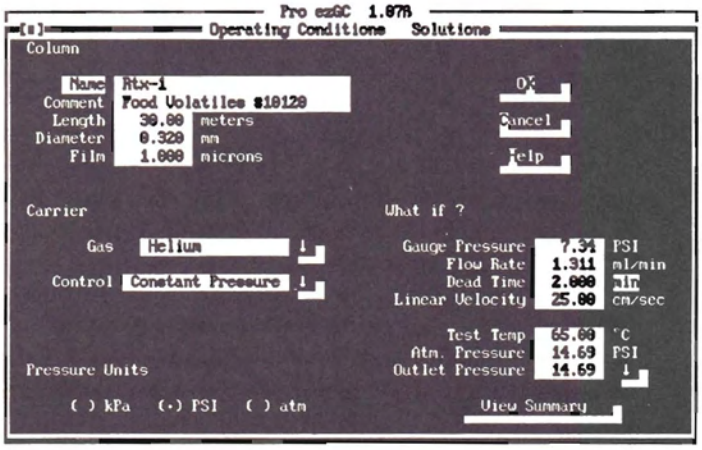
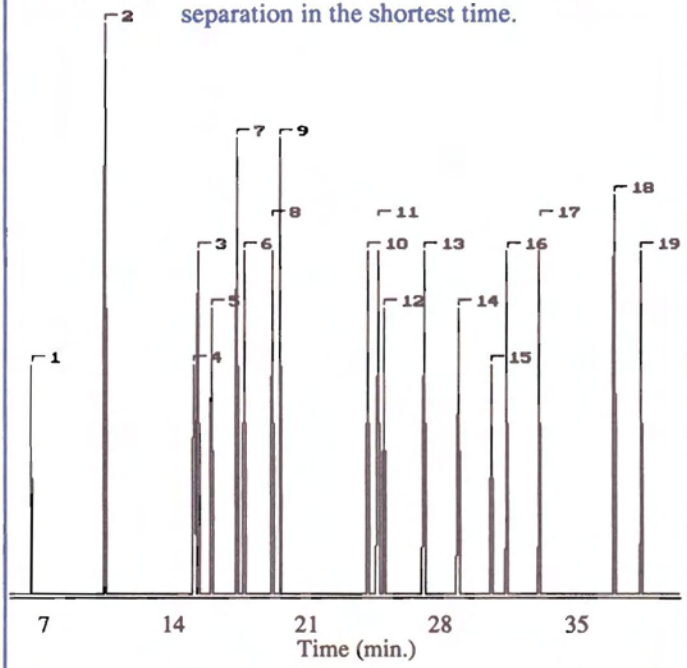


Figure 1 shows the screen used to input the retention time data. The first scenario we will investigate is where a particular column is already installed and the chromatographer wants to determine the conditions that will separate all components with the shortest analysis time. The *ezGC™* software can be set to automatically evaluate different temperature programs and print a solution list. The solution list is prioritized according to the temperature(s) that resolve the most components in the shortest time. A resolution factor can be specified, or defaulted to a value of 1.5, which approximates baseline separation. Figure 2 illustrates the predicted chromatogram obtained for 19 flavor and fragrance compounds using a 30 meter, Rtx®-1 column with a 1.0µm film thickness. The program predicted a 10 minute initial hold at 65°C, providing baseline resolution in under 40 minutes.

Figure 2 - Predict the optimum conditions for baseline separation in the shortest time.



30m, 0.32mm ID, 1.0µm Rtx®-1 (cat.# 10154)
Oven temp.: 65°C (hold 10 min.) to 280°C @ 5 °C/min.
Carrier gas: helium **Regulation:** Constant Pressure
Linear velocity: 25cm/sec. @ 65°C **Dead time:** 2 min. @ 65°C
Peak identifications are on page 5.

Determine how changes in column and carrier gas will improve separations

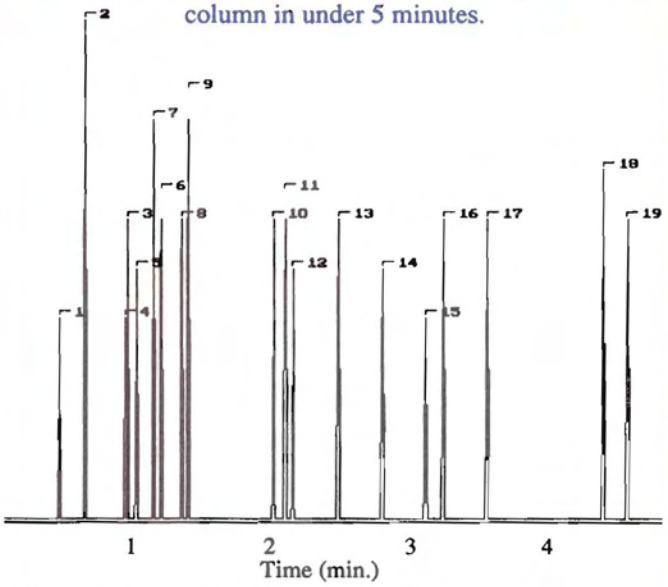
GC method development can be very costly when considering the expense of evaluating different stationary phases, column dimensions, and oven temperatures. Many chromatographers empirically optimize their chromatographic conditions for a particular column and then accept these as the "best separation" achievable. This is understandable considering the cost of buying and installing new columns. With *ezGC™* you can determine within minutes how changes in the column and operating conditions will effect separations (Table I). All that is needed are the retention times for your components from two temperature programs using any column which has the same stationary phase. Previously calculated indices can be retrieved either from disk or loaded from a commercial data base or library.

Table I - Parameters that can be optimized using *ezGC*

oven temperature and program rate (multi-ramp)
minimum analysis time
maximum resolution
column length
film thickness
internal diameter
carrier gas type (He, H_2, N_2, Ar)
carrier gas flow or velocity
constant pressure or pressure program

To illustrate how *ezGC™* can be used to quickly determine the optimum analysis time for a particular sample, Restek's applications group optimized each of the parameters listed in Table I for the flavor and fragrance compounds shown in Figure 1. *ezGC™* predicted that a 15-meter column, 0.25mm ID, a 0.25µm film thickness, in conjunction with a 25°C/min. temperature program would greatly decrease the analysis time without sacrificing resolution. The new optimum analysis was obtained using a lower-cost 15-meter column with an analysis time under 5 minutes with 1/10 the analysis time and 1/2 the column length originally used (Figure 3). *ezGC™* more than pays for itself.

Figure 3 - Complete separation obtained using a 15-meter column in under 5 minutes.



15m, 0.25mm ID, 0.25µm Rtx®-1 (cat.# 10120)

Oven temp.:	55°C to 230°C @ 25 °C/min.		
Carrier gas:	hydrogen	**Regulation:**	Constant Pressure
Linear velocity:	89.1cm/sec. @ 55°C	**Dead time:**	0.281 min. @ 55°C

Peak Identification for Figures 2 & 3.

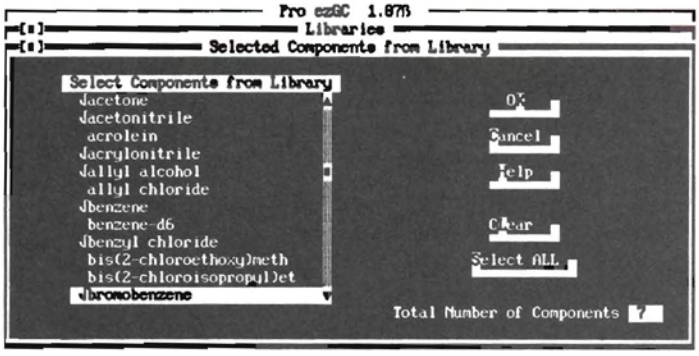
1	ethyl butyrate	8	*p*-cymene	15	eugenol
2	trans-2-hexenol	9	D-limonene	16	vanillin
4	benzaldehyde	10	methone	17	ethyl vanillin
3	α-pinene	11	menthofuran	18	ethyl laurate
5	camphene	12	menthol	19	amyl cinnamic aldehyde
7	β-pinene	13	carvone		
6	octanal	14	cinnamic alcohol		

Do method development without installing a column

Method optimization is much faster using *ezGC™*, but is it possible to make column selection easier? Restek now offers data bases of thermodynamic retention indices called libraries, making it possible to select a column and predict chromatographic separations without even installing a column. These libraries contain hundreds of commonly analyzed components. These libraries have been generated in Restek's Applications Laboratory using the most appropriate stationary phases. Entries are added to the library after certifying each identification using GC/MS. Once the libraries are complete, it is possible to select components by simply choosing the entry from the library section of either *ezGC™* or *Pro ezGC™*. Figure 4 is an example of the select menu, showing a portion of the Environmental Volatiles library. This library currently contains 138 organic components (along with surrogates and internal standards) commonly analyzed in water and solid wastes, analyzed on three different stationary phases (Rtx®-502.2, Rtx®-1 and Rtx®-624). Furthermore, using *Pro ezGC™* makes it possible for each laboratory to create their own "User" libraries, adding compounds which can be modeled along with library data supplied by Restek. Restek now offers five libraries and plans to introduce several more libraries in the upcoming months. We also have plans to continue expanding the number of compounds offered and the number of stationary phases in each library.

Almost every chromatographic method currently in use could be made more efficient by separating the components of interest in less time. The thought of spending days of additional method time and/or purchasing columns which may not give better results often keeps analysts from investigating these options. With *ezGC™* it is possible to quickly and easily determine: "What is the best column?" and "What is the optimum temperature program and carrier flow?". The advanced features of *Pro ezGC™* make GC computer modeling

Figure 4 - Select the specific components for a stationary phase to separate and then optimize your method for any column, oven temperature and carrier gas parameters.



(ezGC™ article is continued on page 6.)

even more powerful through accessing libraries generated in your own lab or purchased from Restek. With libraries, it is possible to evaluate a column and optimize a chromatographic method before installing the column in your GC. For the price of a 30-meter column, you can add this powerful capability to any IBM or compatible PC and begin answering the questions "How will my separation be effected if I change ..." and "What is the best column to separate the components in my sample?" ■

The *ezGC™* program series was created and developed by:
Analytical Innovations, Inc., Kettering, OH

AII Analytical Innovations, Inc.

In cooperation with: **Restek Corporation**

Product Listing

all software packages include both 5¼" and 3½" disks

ezGC™ Software
cat.# 21480

Pro ezGC™ Software
cat.# 21481

ezGC™ to _Pro ezGC™_ Upgrade
cat.# 21482

New! Retention Index Libraries

Food and Flavor Volatiles: A collection of 157 of the most common organic compounds in essential oils are included in this library. Retention indices are provided on the apolar Rtx®-1 and the polar Stabilwax® phases, which are commonly used for analysis of individual components in essential oils and essential oil purity.
Food and Flavor Volatiles: cat.# 21451

Drugs and Pharmaceuticals: This library contains over 100 drug and pharmaceutical compounds commonly analyzed in forensic, clinical and drug laboratories. Retention indices are provided for the parent compounds, analyzed using the Rtx®-5, Rtx®-50, and Rtx®-200 stationary phases. Classes of compounds include: barbiturates, benzodiazepines, opiates, anticonvulsants, anesthetics, and antihistamines.
Drugs and Pharmaceuticals: cat.# 21453

Environmental - Volatiles: A complete collection of the retention indices for the volatile organic compounds specified in EPA 500, 600, and 8000 series methods. A total of 137 components are included from the target analyte lists, recommended surrogates and internal standards for EPA methods 502, 524, 551, 601/602, 603, 624, 8010, 8015, 8020, 8240, and 8260. Stationary phases include Rtx®-1, Rtx®-502.2, and Rtx®-624.
Environmental - Volatiles: cat.# 21452

Solvents and Chemicals (Part 1): A collection of 147 Alkanes, Alcohols, Diols, Aldehydes, and Ketones in the carbon range from C_1 and C_{12} are included for the Rtx®-1, Rtx®-502.2, and Stabilwax®. Part 2 of the library (future introduction) will contain aromatics, esters, ethers, and miscellaneous chemical solvents.
Solvents and Chemicals (Part 1): cat.# 21450

Hints for the Capillary Chromatographer

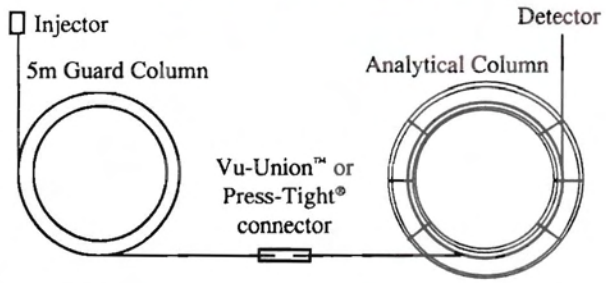
The Benefits of Guard Columns for Capillary Gas Chromatography

The use of guard columns has been commonplace in high performance liquid chromatography for many years. Their use as safeguards to protect the analytical column from highly retentive compounds and particles is well understood. It has only been in the past few years that the benefits of guard columns have been associated with capillary gas chromatography. Although guard columns prolong the life of capillary columns and protect them from sample contamination, they are not widely used. Understanding the basics of guard columns helps to dispel confusion and apprehension about their use.

What is a guard column?

A guard column for capillary chromatography is a short length of deactivated, uncoated fused silica tubing that is placed between the injection port and the analytical column (Figure 1).

Figure 1 - A Guard Column Connected to an Analytical Column

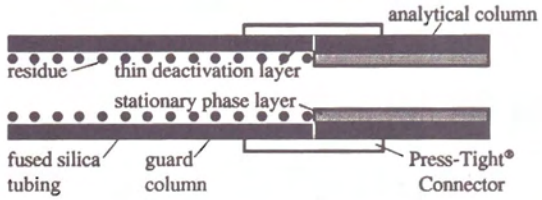


What advantages do guard columns offer?

Prolong column lifetime

A guard column protects and prolongs the lifetime of an analytical column in several ways. It traps non-volatile residue and prevents it from collecting at the head of the analytical column. This non-volatile residue may be very high molecular weight organic compounds, inorganic salts, or particulate materials. If these contaminants enter the analytical column, they can cause adsorption of active compounds and loss of resolution. When this contamination begins to affect sample analysis, a small section of the analytical column must be removed to restore proper performance. However, each time a section of the analytical column is removed, retention times change, some resolution is lost, and column length is decreased, eventually resulting in a useless analytical column. By trapping this contamination in the guard column, the analytical column remains the same length and stays cleaner longer.

Figure 2 - With a guard column installed, the interaction time is minimal between the sample & residue. Thus, decreasing maintenance requirements.



Sample residue deposits approximately 1-meter into the column inlet. Without a guard column, dirt is deposited onto a stationary phase, causing the sample to partition in and out of this dirty region. Adsorptive effects are more likely to occur. Guard columns stop dirt, but do not retain the sample since there is no stationary phase. The interaction between the residue and subsequent sample injections is minimal, and more injections can be performed before maintenance is required.

Decrease maintenance requirements

Since there is no stationary phase on the guard column, the amount of time the sample spends in it is minimized. This reduces the interaction between sample components and the contamination from non-volatile residue (Figure 2). Therefore, guard columns allow more injections to be made before residue interferes with analytical results.

Improve resolution

Many analysts are reluctant to use guard columns because they believe that they will lose resolution. In fact, guard columns actually increase separation efficiency. The guard column acts as a retention gap to help focus the sample at the head of the column. When a sample is injected, it first exists as vapor and aerosol. Without a guard column, the vapor begins to partition in and out of the column's stationary phase. The aerosol portion of the sample does not partition in the phase and moves out ahead of the vaporized sample. This results in broader, less efficient peaks and, in extreme cases, can cause split peaks. Since a guard column is not coated with stationary phase, there is no interaction with the vaporized sample or the aerosol. They move along together in a tighter band. The aerosol vaporizes in the guard column so that when the sample reaches the coated column it is completely vaporized. This produces sharper, more efficient peaks, as shown in Figure 3. Table 1 shows the results of analyzing 2,6-dimethyl-phenol on a 30m, 0.53mm ID, 1.0µm Stabilwax® column with and without a guard column. The efficiency of the 2,6-dimethylphenol peak was measured in each case and the results show a 3.1% increase in efficiency with the guard column.

Figure 3 - Guard columns increase separation efficiency because the aerosol completely vaporizes before it reaches the coated column.

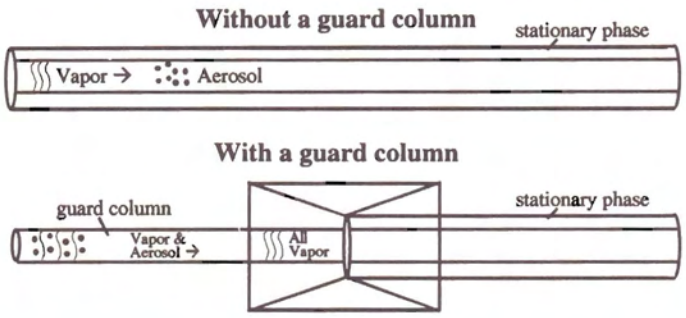


Table I - Column Efficiency Data
(1µl split injection of 2,6-dimethylphenol)

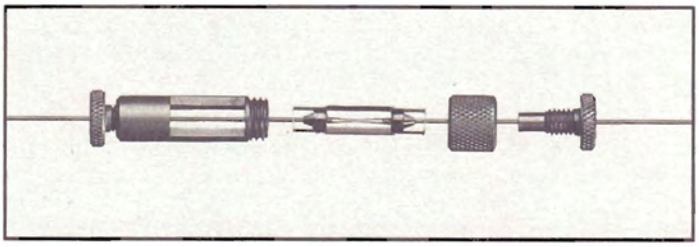
Without Guard Column	*With Guard Column*
Total plates = 51500	Total plates = 53100
Plates/meter = 1716	Plates/meter = 1770

3.1% increase in plates

How is a Guard Column Connected to an Analytical Column?

A Press-Tight® Connector is commonly used to connect a guard column to an analytical column. However, the higher temperature polyimides used by most capillary manufacturers today do not seal well with Press-Tight® connectors. They have been known to disconnect unexpectedly when used at temperatures exceeding 300°C. A new type of connector has become available that has both the benefits of Press-Tight® connectors and the dependability of ferrule seals. The connector, called a Vu-Union™, incorporates ferrules that seal inside a glass union to provide a dead volume free, inert sample pathway. Figure 4 shows the primary Press-Tight® sealing mechanism and the secondary ferrule fail safe sealing mechanism.

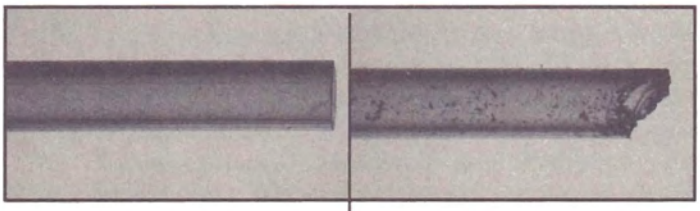
Figure 4 - A disassembled Vu-Union™ shows the primary and secondary sealing mechanisms.



The metal housing holds the tapered glass insert securely. Two thumb screws are positioned at the end of each union to tightly compress the ferrule in the insert. The primary, low dead volume seal occurs as the column ends are compressed in the press-tight taper region.

Whether you are using a Press-Tight® connector or a Vu-Union™, it is important to cut the column end squarely with a ceramic scoring wafer or sapphire blade. Pointed cutting devices are not recommended since they create a crevice on the side of the tubing and result in leakage. Once a square cut has been made, insert the fused silica tubing until you see a brown sealing ring (Figure 5). The presence of a uniform brown ring around the connector is a good indication that the connection will be dead volume free and not contribute to band broadening.

Figure 5 - A square cut is necessary to get a good column seal.



Square cuts made by sapphire scribes or ceramic wafers show the presence of complete sealing rings.

Cuts made by pointed devices allow carrier gas to leak around the column end.

When should a guard column be replaced?

As the guard column becomes contaminated with non-volatile residue, the performance of the chromatographic system will begin to deteriorate. This is normally exhibited as a drastic decrease in the response of active compounds or peak tailing.

How often must a guard column be replaced?

The life expectancy of a guard column depends on the length of the guard column, the amount of non-volatile residue in the samples, and the number of samples run. When analyzing dirty samples, the guard column becomes contaminated quickly. Normally, contamination deposits in the first .5 to .8 meters of the guard column. If a short guard column (1-meter or less) is used, it must be completely replaced when it becomes overly contaminated. If a longer guard column (5-meters) is used, the contaminated section can be removed without re-connecting the analytical column.

How long should a guard column be?

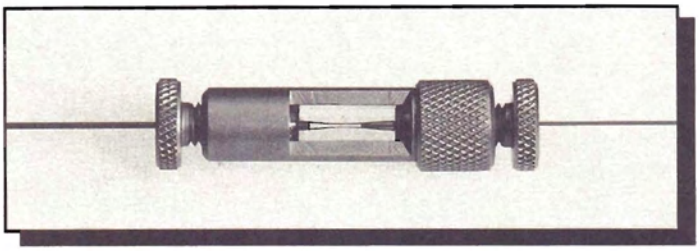
A guard column should be long enough to keep non-volatile residue from entering the column, but short enough so that the analysis time is not significantly increased. Five meter guard columns are more cost effective, reduce the frustrations of making the connection between the guard column and analytical column, and are preferred by most analysts. If a very long guard column (>10-meters) is used, the residence time of the sample components increases, resulting in longer analysis times and skewed peak shapes.

Guard columns help prolong the life expectancy of capillary columns and are an excellent, economical alternative to column replacement. Analysts working with dirty samples find that the use of guard columns significantly reduces column replacement costs and time lost in troubleshooting column contamination problems.

For more information, request your free copy of *A Guide When Injecting Dirty Samples.* ■

Restek's Vu-Union™*

A Leak-Free Press-Tight® Connector for Fused Silica Capillaries



The Vu-Union™ window allows observation of the column connection to ensure that it has been made properly without crushing the column ends.

- Combines the benefits of Press-Tight® connectors with the confidence of ferrule seals.
- Glass window allows visual confirmation of seal.
- Universal tapered glass insert for capillary chromatography seals column with ODs ranging from 0.35 to 0.74mm.
- Universal tapered glass insert for Supercritical Fluid Chromatography (SFC) seals columns with ODs ranging from 0.15 to 0.45mm.
- Uses either graphite or Vespel®/Graphite ferrules.
- Leak-free vacuum connections for MSDs.
- Will not unexpectedly disconnect.
- Maintains deactivation integrity to 400°C, useable at higher temperatures if deactivated surface is not required.
- Will not cause solvent or peak tailing.
- Deactivated tapered glass inserts will not adsorb active compounds.
- Compatible with high temperature polyimides and aluminum clad fused silica coatings.

Capillary Vu-Union™
- One deactivated glass tapered insert (fits column ODs ranging from 0.35 to 0.74mm).
- One metal housing body.

Order ferrules separately below
cat.# 20418

Replacement Vu-Union™ Deactivated Glass Inserts
(fits column ODs ranging from 0.35 to 0.74mm)
cat.# 20419

SFC Vu-Union™
- One deactivated glass tapered insert (fits column ODs ranging from 0.15 to 0.45mm).
- One metal housing body.

Order ferrules separately below
cat.# 20416

Replacement Vu-Union™ Deactivated Glass Inserts
(fits column ODs ranging from 0.15 to 0.45mm)
cat.# 20417

Graphite Ferrules for the Vu-Union™

- Easiest ferrules to use with the Vu-Union™
- Universal, fit Vu-Union™ and connect capillary columns to most GC inlets.
- 450°C maximum operating temperature.

Ferrule Size	Fits Column ID	10-pk. Cat.#	50-pk. Cat.#
0.4mm	0.25mm	20200	20227
0.5mm	0.32mm	20201	20228
0.8mm	0.53mm	20202	20224

Vu-Union™ Vespel/Graphite Ferrules

- Taper designed to fit Vu-Union™ perfectly.
- 60% Vespel®/40% graphite
- 400°C maximum operating temperature.

Ferrule Size	Fits Column ID	10-pk. Cat.#
0.4mm	0.25mm	20420
0.5mm	0.32mm	20421
0.8mm	0.53mm	20422

5-Meter Length Guard Columns

- Increase column lifetime.
- Allow more injections before sample residue degrades column performance.
- Prevent peak splitting during splitless analysis.
- Protect expensive analytical columns.
- Prevent damage from harmful materials.

Nominal ID	Nominal OD	Cat.#
0.05mm	0.35mm	10040
0.10mm	0.35mm	10041
0.15mm	0.35mm	10042
0.18mm	0.35mm	10046
0.25mm	0.35mm	10043
0.32mm	0.45mm	10044
0.53mm	0.70mm	10045

** Patent Pending*

Clinical Corner

Analysis of Barbiturates

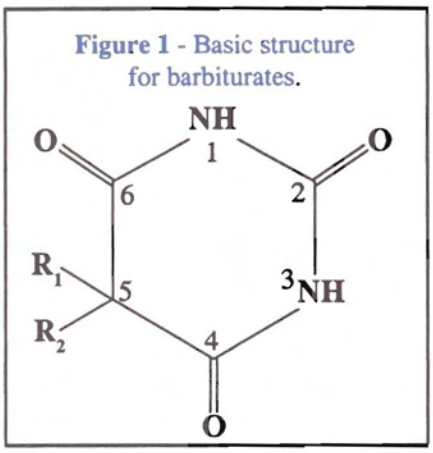


Figure 1 - Basic structure for barbiturates.

Barbiturates are a class of compounds that are central nervous system depressants. They are categorized as sedatives or hypnotics and are primarily used in the treatment of anxiety, insomnia and convulsive disorders. Physical effects of the barbiturates range from mild sedation to coma. Barbiturates are based on a pyrimidine ring structure. Substitution at the 2,4 and 6 positions gives the basic structure for the oxybarbiturates (Figure 1). Replacement of the oxygen at position 2 with sulfur results in the formation of thiobarbiturates. Barbiturates can be ranked according to their onset of activity, duration of action and degree of hypnotic potency. These pharmacological effects are influenced by the types of functional groups attached at position 5. The inclusion of alkyl or aryl groups, the number of carbons in the alkyl side chains, and the degree of branching will affect activity and toxicity.

Extended administration or abuse of barbiturates can lead to physical and psychological dependence. Tolerance to the effects of barbiturates on the central nervous system can be built up with continued exposure to the drug. While tolerance to the intoxicating effects of barbiturates may increase with use, there is very little increase in tolerance to the toxic side effects of high doses. As a result, the therapeutic index for barbiturates is lower than for other sedative/hypnotic drugs like the benzodiazepines. The barbiturates also have an additive effect when administered with other central nervous system depressants. The combination of the low therapeutic index and the additive effects of other CNS depressants makes monitoring for barbiturates an important aspect of drug overdose screening.

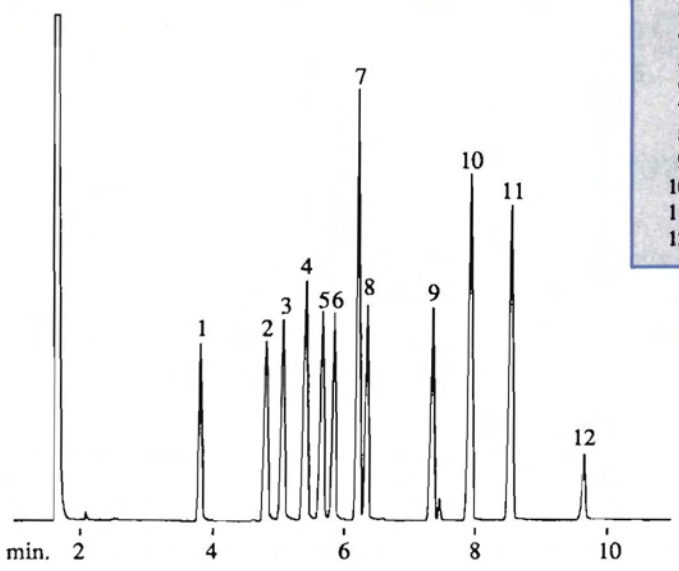
Barbiturates can be analyzed in either their underivatized or derivatized forms by gas chromatography. Derivatization of the barbiturates is most commonly performed by methylation of the amido nitrogens at positions 1 and 3. Methylating reagents like tetramethylammonium hydroxide (TMAH) and trimethylanilinium hydroxide (TMPAH) can be used for on column derivatization of the barbiturates. While derivatization can improve the peak shape and response, extraneous peak formation can interfere with some analyses. Proper injection port set-up is important in obtaining reproducible results with on column derivatization. Methylation of barbiturates is catalyzed by the addition of heat to the reaction mixture. After sample injection, the residence time of the barbiturates and the derivatizing reagent inside the injection port is very short. Since contact of the sample with the heated surface area inside the injection port liner needs to be maximized, liners that are packed with wool or that contain flow disrupting elements, like the Cyclosplitters®, are recommended. In addition, injection port temperatures should be maintained in excess of 250°C in order to efficiently complete the derivatization process.

Barbiturates can be analyzed in either their underivatized or derivatized forms by gas chromatography.

Analysis of barbiturates can also be performed on the underivatized compounds. However, underivatized barbiturates have a tendency to produce overloaded or tailing peaks. Maintenance injection port liners, guard columns and analytical columns regularly in order to achieve good peak shape and adequate resolution. Figure 2 on page 11 shows the separation of a set of underivatized barbiturates using an Rtx®-35 column. Lower polarity stationary phases like the Rtx®-5 can be used to separate the barbiturates, but intermediate polarity stationary phases tend to provide better peak shape and improved resolution

Barbiturates are an important part of drug screening. Extra care should be taken when analyzing barbiturates in either the derivatized or the underivatized form. Intermediate polarity columns combined with well maintained injection port liners and guard columns will contribute to better peak shape and resolution. ■

Figure 2 - Underivatized barbiturates on an Rtx®-35.



COMPOUNDS

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

30m, 0.32mm ID, 0.50µm Rtx®-35 (cat.# 10439)
1µl split injection of barbiturates
Oven temp.: 210°C (hold 2 min.) to 300°C @ 7°C/min. (hold 2 min.)
Inj. and 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

Optimized Using ezGC™!

Product Listing

Rtx®-35
30m, 0.32mm ID, 0.50µm,
cat.# 10439

Barbiturate Standards
1000µg/ml in methanol

amobarbital	cat.# 34028
aprobarbital	cat.# 34029
barbital	cat.# 34030
butabarbital	cat.# 34031
butalbital	cat.# 34032
hexobarbital	cat.# 34033
mephobarbital	cat.# 34034
methohexital	cat.# 34035
pentobarbital	cat.# 34036
phenobarbital	cat.# 34037
secobarbital	cat.# 34038
talbutal	cat.# 34039
thiamylal	cat.# 34040
thiopental	cat.# 34041

Restek offers a large variety of deactivated inlet sleeves for numerous manufacturer's GCs. Please refer to our General Catalog for details.

New from Restek ...

Quantitative Drug Standards

- Purity determination by multiple analytical techniques.
- Quantitative verification on each lot.
- Certificate of analysis supplied with each ampul.
- DEA exempt formulations.

To meet the varied needs of our customers, Restek now stocks quantitative drug standards. These standards are prepared to the same exacting criteria as our environmental calibration mixtures. The standard solutions are prepared by Restek from high quality raw materials.

Quantitative standards are prepared using precise gravimetric techniques after the raw materials have been approved for use. Concentration verification of each lot of product is performed by triplicate analysis of the final product.

The Certificate of Analysis supplied with each ampul, including compound structure, molecular weight, melting point, DSC purity, GC/FID purity, GC/NPD purity, and GC/MS and FT-IR identification. Also included is the calculated concentration of the analyte based upon gravimetric data. Concentrations reported are based upon the free acid/base weight of the compound, with corrections being made for the salt forms.

All mixtures are packaged under nitrogen in flame sealed, silanized amber ampuls. Recommended storage conditions are included on the label and outside package. Each ampul is then placed in a square plastic shell which includes an ampul breaker, extra silanized screw-top amber vial, and an extra label.

All standards are exempt from DEA regulations and can be purchased without a DEA license. Phone orders will be accepted and no additional paperwork is required. Due to DEA regulations regarding product exemption, custom drug standards are not available.

Drug Standards are not available in all countries. Please contact your local distributor for availability.

Standards Spotlight

ASTM Petrochemical Mixtures Available

Calibration and column test mixtures are now available for American Society for Testing and Materials (ASTM) Methods D2887 and D3710.* These standards are made with the same quality and care as our environmental standards. Stock products of single ampul and cost effective 10 packs are available for immediate shipment. Each standard is supplied with a data sheet indicating exact concentration and a sample chromatogram.

D2887 Column Test Mix

Contains 1% (w/v) each of n-hexadecane and n-octadecane dissolved in n-octane. Packaged 1ml per ampul.

Cat.# 31221 each
31321 10 pk.

D2887 Calibration Mix

Contains the following compounds at the concentration (% w/w) listed. Packaged 1ml per ampul.

Compound	Concentration (% w/w)
n-hexane	6%
n-heptane	6
n-octane	8
n-nonane	8
n-decane	12
n-undecane	12
n-dodecane	12
n-tetradecane	12
n-hexadecane	10
n-octadecane	5
n-eicosane	2
n-tetracosane	2
n-octacosane	1
n-dotriacontane	1
n-hexatriacontane	1
n-tetracontane	1
n-tetratetracontane	1

Cat.# 31222 each
31322 10 pk.

Recommended Columns:
Rtx®-2887, 10m, 0.53mm ID, 2.65µm (cat.# 10199)
MXT®-2887, 10m, 0.53mm ID, 2.65µm (cat.# 70199)

D3710 Calibration Mix

Contains the following compounds at the concentration (% v/v) listed. Packaged 1ml per ampul.

Compound	Concentration (% v/v)
2-methylbutane	10%
n-pentane	8
2-methylpentane	6
n-hexane	6
2,4-dimethylpentane	6
n-heptane	10
toluene	12
n-octane	5
p-xylene	14
n-propylbenzene	5
n-decane	4
n-butylbenzene	4
n-dodecane	4
n-tridecane	2
n-tetradecane	2
n-pentadecane	2

Cat.# 31223 each
31323 10 pk.

Recommended Columns:
Rtx®-1, 15m, 0.53mm ID, 5.0µm (cat.# 10177)
MXT®-1, 15m, 0.53mm ID, 5.0µm (cat.# 70177)

***About ASTM** - From the work of 132 technical standards-writing committees, ASTM publishes standard specs, tests, practices, guides, and definitions for materials, products, systems, and services. ASTM also publishes books containing reports on state-of-the-art testing techniques and their possible applications. These standards and related information are used throughout the world.

Expanded List of Underground Storage Tank Monitoring Standards

• Hard to find standards available • High concentration • Full data packs available •

Many customers have approached Restek with interest in obtaining standards that were previously unavailable from any supplier. We are pleased to announce that the materials listed here are now stock products.

We understand that these stock products may not be applicable to every laboratory requirement, so custom products can be made with the raw materials listed.

Aviation Gas Standard

This material is a 100 octane low lead fuel currently used in piston type aircraft. Prepared at 2500µg/ml in 1ml purge & trap grade methanol.

Cat.#	**30094**	**each**
	30094-500	**each w/data pack**
	30194	**10 pk. w/data pack**

Jet Fuel A Standard

Commercial Jet Fuel A prepared at 5000µg/ml in 1ml methylene chloride.

Cat.#	**31215**	**each**
	31215-500	**each w/data pack**
	31315	**10 pk. w/data pack**

Fuel Oil #4 Standard

Fuel Oil #4 is typically used in limited applications where preheating of the fuel prior to burning cannot be utilized. The fuel is a blend of distillate (Fuel Oil #2) and residual (Fuel Oil #6) to meet ASTM viscosity specifications. The Fuel Oil #4 standard offered here has a kinematic viscosity of 21.9 at 38°C (100°F). Kinematic viscosity measurement performed using ASTM D-445.

Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.#	**31216**	**each**
	31216-500	**each w/data pack**
	31316	**10 pk. w/data pack**

Fuel Oil #5 Standard

Fuel Oil #5 is also typically used in applications where little or no preheating of the fuel prior to burning is utilized. As with Fuel Oil #4, Fuel Oil #5 is a blend of distillate and residual to meet viscosity criteria. The Fuel Oil #5 standard offered here has a kinematic viscosity of 106.5 at 38°C (100°F). Kinematic viscosity measurement performed using ASTM D-445.

Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.#	**31217**	**each**
	31217-500	**each w/data pack**
	31317	**10 pk. w/data pack**

Fuel Oil #6 Standard

This oil, also called Bunker C or residual, is a black viscous fuel. Applications where it may be used all require the ability to preheat the fuel prior to pumping and burning.

Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.#	**31218**	**each**
	31218-500	**each w/data pack**
	31318	**10 pk. w/data pack**

Military Fuel Standards

With the mandated clean-up of military sites, the need for standards of these types has increased. The two most common aviation fuel types used today in the military are JP-4 and JP-5. The two standards listed below are available from materials received from Army stocks.

JP-4 Military Fuel Standard

Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.#	**31219**	**each**
	31219-500	**each w/data pack**
	31319	**10 pk. w/data pack**

JP-5 Military Fuel Standard

Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.#	**31220**	**each**
	31220-500	**each w/data pack**
	31320	**10 pk. w/data pack**

Diesel Range Organics (DRO) calibration standard requirements vary significantly from state-to-state. We now have a stock product to meet the requirements of the states of Tennessee and Mississippi.

DRO Mix (Tenn/Miss)

Contains the following compounds at 1000µg/ml each in 1ml methylene chloride.

Decane (C10)	Undecane (C11)
Dodecane (C12)	Tridecane (C13)
Tetradecane (C14)	Pentadecane (C15)
Hexadecane (C16)	Heptadecane (C17)
Octadecane (C18)	Nonadecane (C19)
Eicosane (C20)	Heneicosane (C21)
Docosane (C22)	Tricosane (C23)
Tetracosane (C24)	Pentacosane (C25)

Cat.#	**31214**	**each**
	31214-500	**each w/data pack**
	31314	**10 pk. w/data pack**

Recommended Columns for:

BTEX & Gasoline Range Organics:

Rtx®-502.2, 105m, 0.53mm ID, 3.0µm (cat.# 10910)
MXT®-502.2, 105m, 0.53mm ID, 3.0µm (cat.# 70910)
Rtx®-624, 105m, 0.53mm ID, 3.0µm (cat.# 10975)
MXT®-624, 105m, 0.53mm ID, 3.0µm (cat.# 70975)

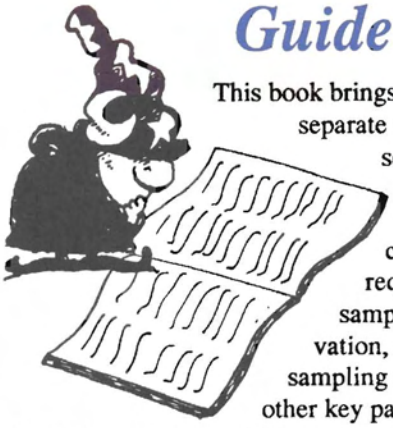
Diesel Range Organics:

Rtx®-1, 30m, 0.53mm ID, 1.0µm (cat.# 10155)
MXT®-1, 30m, 0.53mm ID, 1.0µm (cat.# 70155)
Rtx®-5, 30m, 0.53mm ID, 1.0µm (cat.# 10255)
MXT®-5, 30m, 0.53mm ID, 1.0µm (cat.# 70255)

Peak Performers

Don't be without this handy reference...

Guide to Environmental Analytical Methods



This book brings together in one work the separate methods required by several major environmental statutes. Included are method detection limits, calibration techniques, QC requirements, analytes, sample storage, sample preservation, instrumentation, field sampling requirements, and many other key parameters summarized from the environmental methods. A handy cross-reference of analytes arranged alphabetically tells users at a glance which methods are acceptable for each chemical listed. A glossary of terms and abbreviations and an EPA contact list are also included.

The *Guide to Environmental Analytical Methods* is based on *Variability in Protocols*, prepared by the United States Environmental Protection Agency's Risk Reduction Engineering Laboratory, Quality Assurance Office. The *Guide* was developed as a quick reference for personnel performing QA audits and for lab and environmental managers reviewing or developing QA Project Plans. Field personnel have found the *Guide* valuable for guidance in sampling protocol.

The *Guide to Environmental Analytical Methods* provides laboratories and field sampling personnel with information derived from stacks of references costing hundreds of dollars. Here's how using the *Guide* can benefit your operation:

- Saves hours spent searching through lengthy methods looking for information
- Instantly compares the capabilities and requirements of each method
- Simplifies the decision-making process regarding which method to use
- Invaluable reference for preparing QA audits and developing QA Project Plans
- Answers questions by field sampling personnel on sample amount, container, and preservative
- Serves as an invaluable starting point in training lab personnel about environmental methodology
- Useful as a bench reference to supplement more detailed information found in other volumes
- Supplies instantaneous answers to client's routine questions

Guide to Environmental Analytical Methods
Genium Publishing Corporation
cat.# 20465

More Reference Books Available from Restek

A Practical Guide to the Care, Maintenance and Troubleshooting of Capillary Gas Chromatographic Systems
Dean Rood
This guide places emphasis on avoiding GC problems. It is organized by the nature of chromatographic problems that are encountered. Each possible explanation for the problem refers back to the text of the book which outlines procedures to diagnose and repair the problem, and procedures to prevent or minimize the frequency and severity of the problem. A step-by-step flow chart is included to aid in pinpointing the problem area. The text can be used as a guide in the proper operation and maintenance of a capillary gas chromatograph to obtain maximum performance with minimal difficulties and effort. Explanations are on a practical level so that intimate knowledge of chromatography and chemistry is not required to fully benefit from the information presented.
Huethig Publishing, Ltd., 1991 • Xii + 192pp.
cat.# 20450

On-Column Injection in Capillary Gas Chromatography
Basic Technique; Retention Gaps; Solvent Effects
Konrad Grob
On-column injection techniques have become an attractive mode of sample introduction since it minimizes detrimental adsorption and non-linearity problems associated with split/splitless techniques. Grob's text is a must read treatise for the novice as well as the experienced chromatographer desiring to gain the on-column advantages. Basic technique is explained clearly with excellent schematics. Topics include: sample introduction, injector design, syringes, solvent and temperature effects. Entire chapters are spent discussing solvent effects and retention gaps. This is a real how and why guide to on-column injection.
Huethig Publishing, Ltd., 1987 • xx + 591pp.
cat.# 20453

Split/Splitless Sleeves for Fisons 8000/CE MEGA GCs

- Fits Fisons 8000/CE MEGA GCs
- Deactivated for exceptional inertness
- Cup Splitter and Laminar Cup Splitter designs for split injections
- Double Gooseneck design for splitless injections

To fulfill customer requests, Restek now offers additional sleeves for the Fisons 8000/Carlo-Erba MEGA GCs.* Two new split sleeves, the Cup Splitter and Laminar Cup Splitter, minimize the molecular weight discrimination encountered with other split sleeve designs. In addition, the laminar cup splitter performs exceptionally well for large volume injections.

The new Double Gooseneck sleeve increases splitless efficiency and decreases breakdown of active compounds. The gooseneck design also effectively contains the sample vaporization cloud, preventing backflash. Sold individually or in economical 5-packs, these sleeves are guaranteed to meet the original equipment specifications (OD 8mm, length 105mm).

Cup Splitter for Fisons 8000/CE MEGA GCs
cat.# 20950, each
cat.# 20951, 5-pk.

Laminar Cup Splitter for Fisons 8000/CE MEGA GCs
cat.# 20948, each
cat.# 20949, 5-pk.

Double Gooseneck for Fisons 8000/CE MEGA GCs
cat.# 20952, each
cat.# 20953, 5-pk.

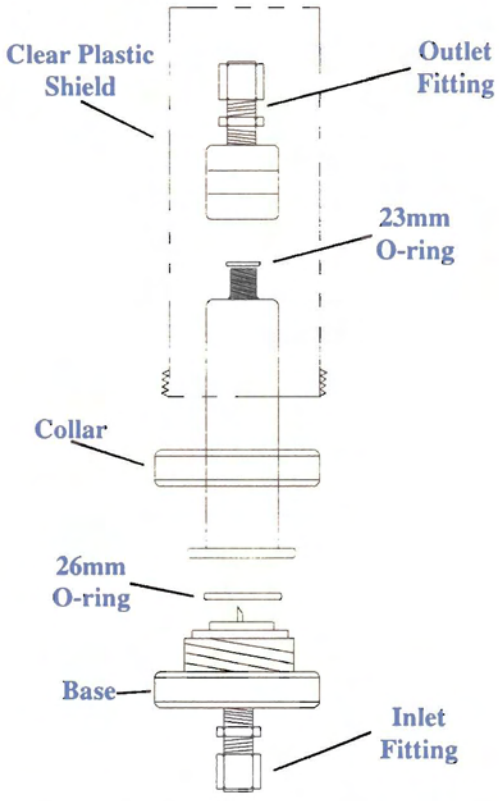
** Please see our new 1993/94 Chromatography Products Catalog for more Carlo-Erba GC sleeves.*

High Capacity Indicating Oxygen Trap

Please don't install a new Restek superior performance capillary column without making sure your carrier gas is O_2 and H_2O free! Columns last longer, show less bleed, and detector noise is minimized when the carrier gas is purified with a High Capacity Indicating Oxygen Trap.

- Indicator changes from dark blue to brown as O_2 and H_2O are trapped.
- Heavy duty, sturdy body design virtually eliminates breakage.
- High capacity (>300s.c.f.) design lasts longer than three smaller traps.
- Economic replacement cartridges easily changed.
- Useable with multiple GC systems.
- Removes impurities at flow rates up to 230s.c.f./hr.
- Removes O_2 and H_2O impurities without introducing other contaminants or by-products.
- Also removes trace contaminants from GC carrier gases.
- Useable with all carrier gases. (He, H2, N2, Meth/AR)
- 150psig operating pressure, up to 100°C operating temperature.
- Built-in microparticulate frit.

Cartridge housing & one cartridge:
1/4" tube compression fittings, cat.# 20623
1/8" tube compression fittings, cat.# 20624
Refill cartridge (fits either 1/4" or 1/8"), cat.# 20625



News from Restek

We will be There!

When the Minnesota Chromatography Forum's Spring Symposium begins on May 4, the Restek Wizards will be on hand to present papers, exhibit our products, and demonstrate our new GC method development software, *ezGC™*. *Hope to see you there!*

Restek's Technical Literature:

Environmental Chromatography Products
Fused Silica Capillary Column Installation Guide
A Guide When Injecting Dirty Samples
Operating Hints for Split/Splitless Injectors
Guide to Minimizing Septa Problems
Helpful Hints for Analyzing Volatile Organics

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Fresh Off the Press!

Chromatography Products Catalog

Restek's new, 272-page 1993/94 Chromatography Products catalog contains over 105 pages of applications chromatograms along with many pages of new and innovative products.

We're interested in your opinion of the new catalog. If you have any suggestions, please call our Technical Service Department at 800-356-1688, ext. 4.

Dial Direct into Technical Service

You can now dial direct to our technical support hotline when calling Restek's voice mail system. Call our toll-free number, 800-356-1688, ext. 4, to access our technical support group.

Technical service is available Monday - Thursday from 8am-8pm EST, and Fridays from 8am-5pm EST. We are always working harder to provide you with the best technical support possible.

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THE RESTEK ADVANTAGE

Purge and Trap of Volatile Organics Using Narrowbore Capillary Columns

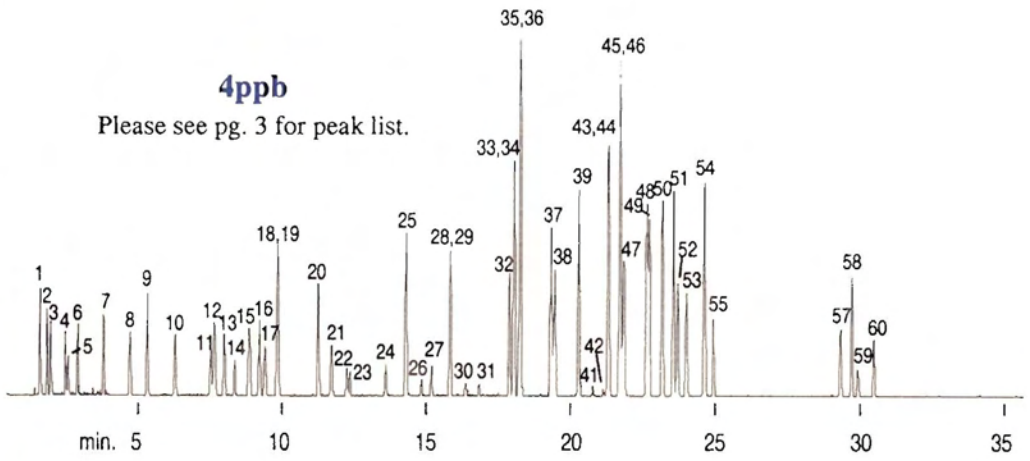
- Eliminates the need for a jet separator or secondary trapping.
- Narrowbore columns result in shorter analysis times.
- Meets MDLs for EPA methodology.
- Reduces cost of analysis.

Volatile organic analyses (VOA) are commonly run using GC/MS systems and 0.53 mm ID columns. There is now increasing interest in utilizing narrowbore (0.18 - 0.25 mm) columns for VOA analysis because a simpler GC/MS system configuration is used and shorter analysis times are obtained. Figure 1 shows an analysis of an EPA Method 524 calibration standard using a 0.18 mm ID column, split injection port and a direct connection to the GC/MS.

System Configuration

The narrowbore system utilizes a slightly different configuration than the more common systems used for 0.53mm ID columns. The injection port is a standard split/splitless inlet operated in the split mode. It is important to minimize dead volume between the transfer line from the purge and trap sampler and within the injection port, so Restek recommends connecting the purge and trap to the transfer line using a low dead volume union and a 1 mm ID injection port sleeve (See page 3). The exit end of the capillary column is directly interfaced into the mass spectrometer source, eliminating the need for a jet separator and an additional vacuum pump.

Figure 1 - Achieve MDLs on a narrowbore column.



20m, 0.18mm ID, 1.0µm Rtx®-502.2 (cat.# 40914)
4ppb of VOA standards
Oven temp.: 35°C (hold 5 min.) to 180°C @ 6°C/min., then to 210°C (hold 5 min.) @ 20°C/min.
Inj. temp.: 100°C
Detector: HP 5970 MSD
Det. temp.: 280°C
Linear velocity: 20cm/sec. set @ 35°C
Split ratio: 40:1
Scan range: 35-260
Purge & Trap: Tekmar 3000
Trap: Vocarb™ 3000
Trap Pressure Control: 6psi
Purge time: 11 min.
Desorb preheat temp.: 250°C
Desorb temp.: 260°C
Desorb time: 2 min.
Desorb flow rate: 20ml/min.

Chromatogram courtesy of Anne Williams, Tekmar Company

in this issue...

Optimum desorbtion flow rates for a purge and trap range between 20-30ml/min. When using a 0.53mm ID capillary column, the desorbtion flow rate is typically 10ml/min. This results in a wide sample band which usually requires refocusing to improve peak widths of early eluting components. When a short 0.53mm ID column is used, sub-ambient cooling is necessary. When using a long, thick film 0.53mm ID column, refocusing can be achieved at the head of the column. In the past, narrowbore columns were used but the purge and trap desorb flows were set at 1-2ml/min., which is the optimum flow for narrowbore columns. This low desorb flow results in unacceptable sample band widths and loss of resolution. One solution to this problem is to cold trap the sample band at the front of the narrowbore column. This technique requires an expensive cryofocusing unit and liquid nitrogen for operation.

A simpler, less expensive approach is to set a higher desorb flow of 20 to 30ml/min. and split the desorb flow using a split/splitless injection system. With a split ratio of 20:1 to 40:1 the column can be operated below 1ml/min., when it is close to the optimum flow rate. The faster desorb flow results in a very narrow sample band width. This low column flow rate is also compatible with a direct interface to all bench top GC/MS systems.

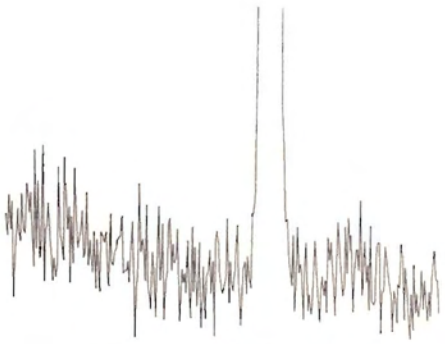
Meeting MDLs

Desorbing at high flow rates delivers a narrower band to the injector. However, splitting at the injection port not only reduces the amount of flow to the column, it also decreases the amount of the sample reaching the detector. To reach the required method detection limits it is recommended to purge a 25ml sample volume instead of 5ml. This will increase the amount of sample delivered to the system by 5 fold and compensate for some of the loss of sample caused by splitting. Even when purging 25mls, there is a 4 or 5 times reduction in the amount of sample reaching the detector. However, the improved efficiency of the narrowbore column produces narrower sample band widths which result in taller peaks and better signal to noise ratio. If the same amount of sample is injected onto both a narrowbore and wide bore column, the smaller diameter column gives a narrower peak width, resulting in a stronger signal at the apex. By using the larger sample size and taking advantage of an increased signal to noise, the required MDLs can be achieved. Figure 1 shows a chromatogram of a 4ppb VOA standard analyzed on a 20m, 0.18mm ID, 1.0µm Rtx®-502.2, directly interfaced into the mass spectrometer source. This chromatogram was generated from a system that had a 20ml/min. desorb flow rate and a split ratio of 40:1.

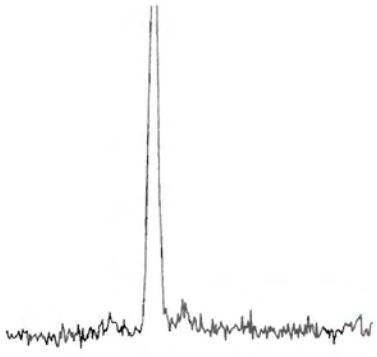
In a system using 0.53mm ID columns and a jet separator, the noise level is higher compared to a narrowbore system. It is possible that this increase is due to a higher flow rate of helium carrier gas entering the MS source. A comparison of the noise levels of the 0.53mm ID system to the narrowbore system configurations is shown in Figure 2. The noise level is 4 times greater on the 0.53mm ID system compared to the narrowbore configuration.

Figure 3 shows a chromatogram of a 200ppb standard analyzed on a 40m, 0.18mm ID, 1.0µm Rtx®-502.2. This chromatogram was generated from a system that had a 30ml/min. desorb flow rate, a split ratio of 30:1, and direct interface into the mass spectrometer source. Although the 20 meter column reduces analysis time by about 5 minutes compared to the 40 meter column, the resolution of several components is lost. The improvement in peak shape for the early eluting gases is a result of the Trap Pressure Control available on the LSC 3000 system.

Figure 2 - Noise comparison of a 0.53 mm ID column vs. a narrowbore column directly interfaced into the mass spec source. The noise level is 4 times less on the narrowbore setup.*



0.53mm ID
p/p noise = $4.5e^4$ units

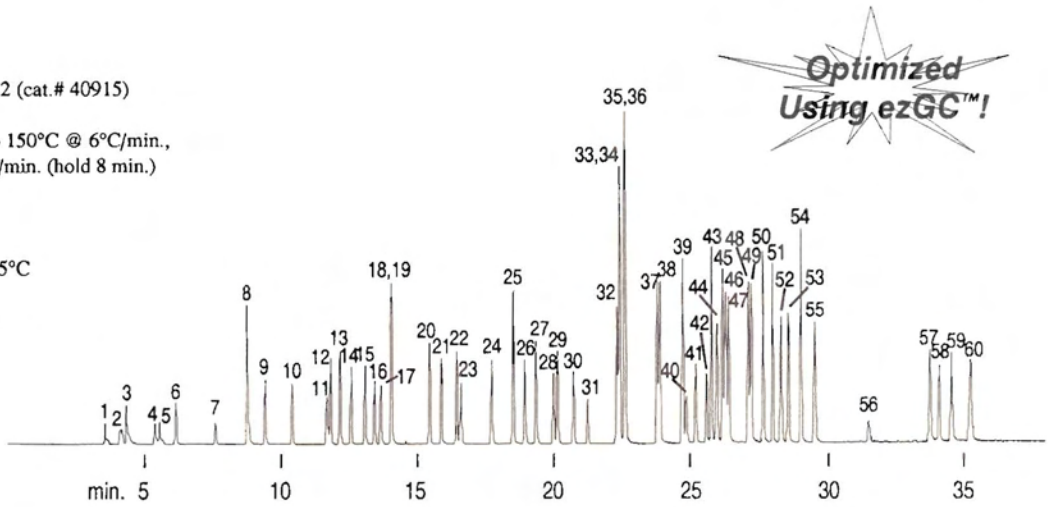


0.18mm ID
p/p noise = $1.0e^4$ units

**The x and y scale are identical.*

Figure 3 - Volatile organic analysis using a 40m, 0.18mm ID, 1.0µm Rtx®-502.2 with a split/splitless injection port and directly interfaced into a mass spec source.

40m, 0.18mm ID, 1.0µm Rtx®-502.2 (cat.# 40915)
200ppb of VOA standards
Oven temp.: 35°C (hold 4 min.) to 150°C @ 6°C/min., then to 220°C @ 8°C/min. (hold 8 min.)
Inj. temp.: 200°C
Detector: HP 5971 MSD
Det. temp.: 250°C
Linear velocity: 21cm/sec. set @ 35°C
Split ratio: 30:1
Purge & Trap: Tekmar LSC 2000
Trap: Vocarb™-4000
Purge time: 11 min.
Desorb preheat temp.: 250°C
Desorb time: 2 min.
Desorb flow rate: 30ml/min.



Peak List for Figures 1 and 3

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. 1,1-dichloroethane
11. 2,2-dichloropropane
12. cis-1,2-dichloroethene
13. bromochloromethane
14. chloroform
15. 1,1,1-trichloroethane
16. 1,1-dichloropropene
17. carbon tetrachloride
18. benzene
19. 1,2-dichloroethane
20. trichloroethene
21. 1,2-dichloropropane
22. bromodichloromethane
23. dibromomethane
24. cis-1,3-dichloropropene
25. toluene
26. trans-1,2-dichloropropene
27. 1,1,2-trichloroethane
28. 1,3-dichloropropane
29. tetrachloroethene
30. dibromochloromethane
31. ethylene dibromide
32. chlorobenzene
33. ethylbenzene
34. 1,1,1,2-tetrachloroethane
35. *m*-xylene
36. *p*-xylene
37. *o*-xylene
38. styrene
39. isopropylbenzene
40. bromobenzene
41. 1,1,2,2-tetrachloroethane
42. 1,2,3-trichloropropane
43. propylbenzene
44. bromobenzene
45. 1,3,5-trimethylbenzene
46. 2-chlorotoluene
47. 4-chlorotoluene
48. tert-butylbenzene
49. 1,2,4-trimethylbenzene
50. *sec*-butylbenzene
51. *p*-isopropyltoluene
52. 1,3-dichlorobenzene
53. 1,4-dichlorobenzene
54. n-butylbenzene
55. 1,2-dichlorobenzene
56. 1,2-dibromo-3-chloropropane
57. 1,2,4-trichlorobenzene
58. hexachlorobutadiene
59. naphthalene
60. 1,2,3-trichlorobenzene

Conclusion

The use of narrow bore columns, in the split mode, can significantly reduce the costs associated with the GC/MS analysis of volatile organics. Instrument costs are reduced by eliminating the use of a jet separator. Column costs are reduced by using shorter length, narrow diameter columns. Reduction in analysis time further adds to cost savings. Required MDLs are easily achieved by using a 25ml sample volume and the improved signal to noise ratio produced by narrow bore columns. ■

Product Listing

Rtx®-502.2

20m, 0.18mm ID, 1.0µm	cat.# 40914
40m, 0.18mm ID, 1.0µm	cat.# 40915
30m, 0.25mm ID, 1.4µm	cat.# 10915
60m, 0.25mm ID, 1.4µm	cat.# 10916

Rtx®-624

20m, 0.18mm ID, 1.0µm	cat.# 40924
40m, 0.18mm ID, 1.0µm	cat.# 40925
30m, 0.25mm ID, 1.4µm	cat.# 10968
60m, 0.25mm ID, 1.4µm	cat.# 10969

Injection Port Sleeves:

1mm ID Split Sleeve for HP 5890 GCs
cat.# 20972 each
cat.# 20973, 5-pk.

1mm ID Split Sleeve for Varian 1075/1077 GCs
cat.# 20970 each
cat.# 20971, 5-pk.

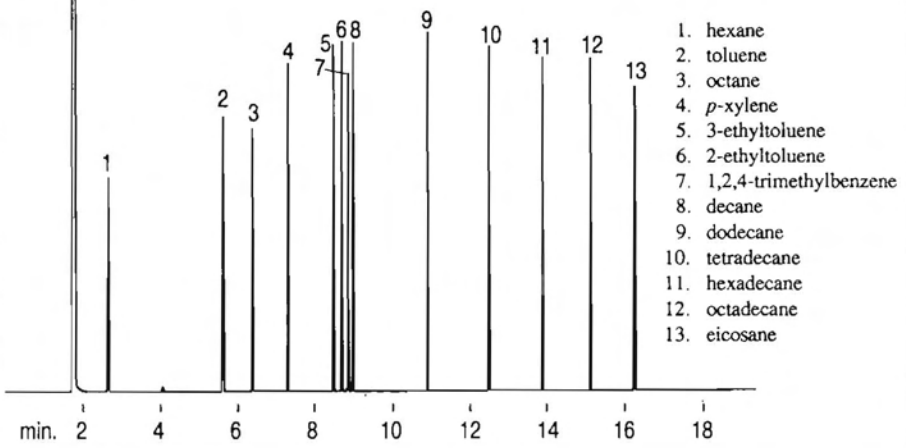
See Restek's *1993-1994 Chromatography Products Catalog* for a complete listing of VOA standards.

Fire Debris Analysis

Capillary gas chromatography is the method of choice for analyzing suspected arson samples from fire debris Because of the complex composition of most accelerants used in arson cases, it is crucial that positive identification be made of the material used to start the fire. The American Society for Testing Materials (ASTM) has developed standard test procedures for analyzing these samples. The information presented in this article references ASTM E1387-90, "Standard Test Method for Flammable or Combustible Liquid Residues in Extracts from Samples of Fire Debris by Gas Chromatography".

As with all analytical procedures, sample collection, preservation, chain of custody, and sample preparation play a crucial role in the process. Samples may be extracted using several different techniques (1) for introduction into the gas chromatograph and are beyond the scope of this article.

Figure 1 - Resolution Check Mix demonstrates proper column performance.



Run Conditions for Figures 1 - 3:
30m, 0.53mm ID, 1.5µm Rtx®-1 (cat.# 10170), 1.0µl split injection of E1387-90 Column Resolution Check Mix (cat.# 31224)
Oven temp.: 40°C (hold 3 min.) to 75°C @ 15°C/min., then to 275°C @ 20°C/min. (hold 5 min.)
Inj./det. temp.: 250°C/285°C **Carrier gas:** hydrogen **Linear velocity:** 50cm/sec. @ 40°C
FID sensitivity: 4.10×10^{-9}AFS **Split ratio:** 30:1

Appropriate capillary column selection is the first decision the analyst must make. The ASTM standard allows the use of any capillary column and conditions, provided that a Resolution Test Mix is completely resolved into the individual components. This resolution test mix consists of equal weights of the even numbered n-alkanes from C6 to C20, plus several aromatic compounds. The aromatics specified are: toluene, *p*-xylene, 2-ethyltoluene, 3-ethyltoluene, and 1,2,4-trimethylbenzene.

Several different stationary phases and column configurations can provide the resolution needed. Typically, laboratories can use 30-meter columns coated with either Rtx®-1 (100% dimethyl polysiloxane) or Rtx®-5 (5% diphenyl 95% dimethyl polysiloxane). Film thicknesses can vary from 1.0 to 1.5µm. Choice of column ID should depend upon sample capacity and detection system employed. The standard allows for the use of either FID, PID, or MS detectors. If MS detection is employed, use a 0.25mm ID column to minimize carrier gas flow. If FID or PID detection is employed, use a 0.53mm ID to maximize column capacity. By doing this, the analyst can minimize expensive duplicate analyses or dilutions if the concentration of accelerants are very high.

Figure 1 shows the complete resolution of all 13 components in the column resolution check mix on a 30m, 0.53mm ID, 1.5µm Rtx®-1 column with an FID detector. The linear velocity and temperature program chosen allow the entire analysis to be completed in about 16 minutes.

After establishing the correct conditions to obtain complete resolution of the test mix components, the analyst must then calibrate the instrument. In fire debris analysis, this involves purchase and preparation of common accelerants used to ignite fires and injection into the GC. Quantitation of unknown samples is not performed. The analyst must provide positive identification to the field investigators of any accelerants found in the samples collected. To do this, the analyst must be able to recognize typical chromatographic patterns of each accelerant. Figure 2 shows the chromatographic pattern obtained from an injection of an unleaded gasoline standard.

To further complicate this analysis, many factors will change the chromatographic pattern obtained from fire debris. The first is weathering of the material from the heat of the fire along with dilution of water used to extinguish the blaze. This

Figure 2 - Unleaded Gasoline Standard (unweathered)

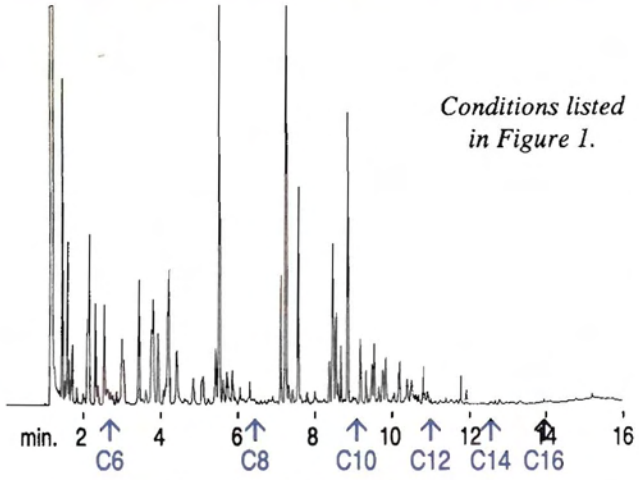
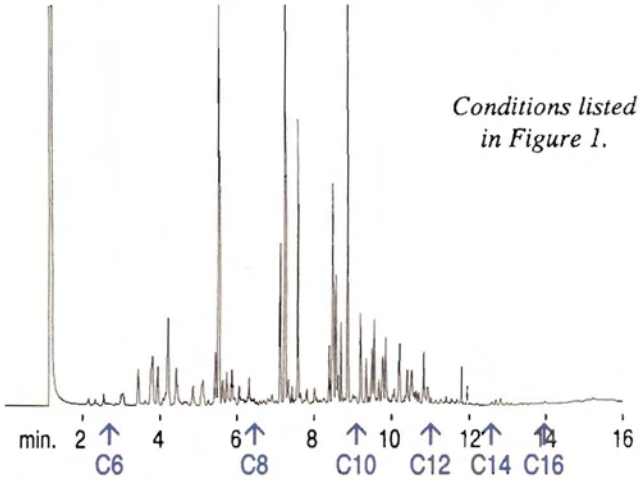


Figure 3 - Unleaded Gasoline (50% weathered)



weathering can dramatically change the chromatographic pattern of the material. Typically, lower boiling materials are lost by the heat, leaving the higher boiling compounds remaining. This type of weathering can be simulated in the laboratory by evaporating the material under controlled conditions. The advantage of performing the evaporation in the laboratory is that the exact amount of weight loss compared to the original starting material can be measured and controlled. Figure 3 shows the analysis of a 50% weathered unleaded gasoline. The gasoline has been weathered to a 50% weight loss and an exact concentration calibration standard was prepared with the remaining material. By analyzing known weathered products, the analyst can more readily recognize the type of original starting material.

Table I
Typical Hydrocarbons

Class #		Range	Examples
1	Light Petroleum Distillates (LPD)	C4-C8	Pocket lighter fuel, petroleum ethers, some rubber cement solvents
2	Gasoline	C4-C12	gasoline (ALL), some camping fuels
3	Medium Petroleum Distillates (MPD)	C8-C12	mineral spirits, paint thinners, some torch fuels, some charcoal fuels, some charcoal starters
4	Kerosene	C9-C16	Kerosene, No.1 Fuel Oil, Jet-A Fuel Oil, Jet-A Fuel, some charcoal starters, some torch fuels
5	Heavy Petroleum Distillates (HPD)	C10-C23	No. 2 Fuel Oil Diesel Fuel #2
0	Unclassified	Variable	alcohols, acetone, toluene, some lamp oils, camping fuels, lacquer thinners

There are five basic classes of complex petroleum distillates normally identifiable from arson samples. A sixth class of accelerants (Class 0) includes single compounds sometimes used and identified. Table I shows the complete list of classes, typical chromatographic range of each material (based upon hydrocarbon elution), and examples of each type.

There are additional variables which can make identification of the petroleum residue more difficult. Included would be co-extracted volatiles and pyrolyzates from the fire debris. The extent of these co-extracted interferences would, in part, be dependant upon the sample preparation method used. The ASTM method does provide minimum requirements for class identification for each type of accelerant, but in the end the experience of the analyst is crucial.

Adequate chromatographic resolution can be obtained on several different capillary columns. Typically, the best resolution can be obtained on a Rtx®-1 (100% dimethyl polysiloxane) stationary phase. Column configuration should be based upon the detection system being used and sample capacity. Calibration with weathered petrochemical standards also plays an important part in identification of accelerants extracted from fire debris samples. ■

References

1) ASTM Standard Practices for Fire Debris Extraction:
ASTM E1412-91 Dynamic Headspace Concentration
ASTM E1413-91 Passive Headspace Concentration
ASTM E1385-90 Steam Distillation Concentration
ASTM E1386-90 Solvent Extraction Concentration
ASTM E1388-90 Sampling of Headspace Vapors
ASTM E1389-90 Cleanup by Acid Stripping

Product Listing

Rtx®-1:

30m, 0.53mm ID, 1.5µm
cat.# 10170

30m, 0.32mm ID, 1.0µm
cat.# 10154

30m, 0.25mm ID, 1.0µm
cat.# 10153

See page 6 for a list of our new Weathered Petrochemical Standards!

Standards Spotlight

Weathered Petrochemical Standards Now Available!

May be used for:
- Underground Storage Tank Monitoring
- ASTM fire debris analysis.

Laboratories monitoring underground storage tanks often find it difficult to determine the type of petrochemical detected during an analysis. The main reason for this is the petroleum product has weathered from exposure to air, water, sunlight, and microbial action. All of these factors can lead to misidentification of the original product.

A similar situation occurs for forensic analysts investigating potential arson cases. When arson is suspected, samples are taken from the site and analyzed using ASTM E1387-90 methods. In these methods, quantitation is not performed but identification of the accelerant (if any) is crucial. Again, weathering of the petrochemical can drastically change the chromatographic profile when compared to non-weathered material.

Restek is now offering as stock products, weathered petrochemical products to meet many of these difficult situations. All of these standards are prepared from commercially acquired materials. The material is then weathered in the laboratory based upon a specific weight loss from the original weight of starting material. While we cannot duplicate all environmental or arson factors, these standards may be useful in identification of the type of petrochemical detected.

Unleaded Gasoline (ASTM Class 2 Accelerant)

These standards are prepared from a **single source (one refinery)** product. Samples of regular and premium grade unleaded gasoline were collected, then blended in equal volumes. The weathered materials indicate the percent weight loss from original starting material. These standards are prepared at 5000µg/ml in purge & trap grade methanol, 1ml per ampule.

Unleaded Gasoline Standard (unweathered)

Cat.#	30096	each
	30096-500	ea. w/data pack
	30096-510	5pk.
	30096-520	5pk. w/data pack
	30196	10 pk. w/data pack

Unleaded Gas Standard - 25% Weathered

Cat.#	30097	each
	30097-500	ea. w/data pack
	30097-510	5pk.
	30097-520	5pk. w/data pack
	30197	10 pk. w/data pack

Unleaded Gas Standard - 50% Weathered

Cat.#	30098	each
	30098-500	ea. w/data pack
	30098-510	5pk.
	30098-520	5pk. w/data pack
	30198	10 pk. w/data pack

Unleaded Gas Standard - 75% Weathered

Cat.#	30099	each
	30099-500	ea. w/data pack
	30099-510	5pk.
	30099-520	5pk. w/data pack
	30199	10 pk. w/data pack

Weathered Gasoline Kit

Contains one ampule (1ml) each of the following products:
- Unleaded Gasoline Standard (cat.# 30096)
- Unleaded Gas Standard - 25% Weathered (cat.# 30097)
- Unleaded Gas Standard - 50% Weathered (cat.# 30098)
- Unleaded Gas Standard - 75% Weathered (cat.# 30099)

Cat.#	30100	per kit
	30100-500	w/data pack

Weathered Petrochemical Standards (cont.)

Mineral Spirits (ASTM Class 3 Accelerant)

There are four general types of material classed as mineral spirits, dependent upon boiling point range (BPR). Type I mineral spirits include stoddard solvent (BPR of 149-182°C), Type II mineral spirits have a high flash point (BPR of 177-196°C), Type III are odorless mineral spirits (BPR of 149-196°C) and Type IV are low dry point spirits (BPR of 149-174°C).

The standards listed below were prepared from an equal volume blend of Type I, II, and III mineral spirits. The weathered material indicated the percent weight loss from the original starting material. These standards are prepared at 5000µg/ml in methylene chloride, 1ml per ampule.

Mineral Spirits Standard (unweathered)

Cat.#	31225	each
	31225-500	ea. w/data pack
	31225-510	5pk.
	31225-520	5pk. w/data pack
	31325	10 pk. w/data pack

Mineral Spirits Standard - 25% Weathered

Cat.#	31226	each
	31226-500	ea. w/data pack
	31226-510	5pk.
	31226-520	5pk. w/data pack
	31326	10 pk. w/data pack

Mineral Spirits Standard - 50% Weathered

Cat.#	31227	each
	31227-500	ea. w/data pack
	31227-510	5pk.
	31227-520	5pk. w/data pack
	31327	10 pk. w/data pack

Mineral Spirits Standard - 75% Weathered

Cat.#	31228	each
	31228-500	ea. w/data pack
	31228-510	5pk.
	31228-520	5pk. w/data pack
	31328	10 pk. w/data pack

Weathered Mineral Spirits Kit

Contains one ampule (1ml) each of the following products:

- Mineral Spirits Standard (cat.# 31226)
- Mineral Spirits Standard - 25% Weathered (cat.# 31227)
- Mineral Spirits Standard - 50% Weathered (cat.# 31228)
- Mineral Spirits Standard - 75% Weathered (cat.# 31229)

Cat.#	31237	per kit
	31237-500	w/data pack

Kerosene (ASTM Class 4 Accelerant)

These standards are prepared from a single source (one refinery) product. The weathered materials indicate the percent weight loss from original starting material. These standards are prepared at 5000µg/ml in methylene chloride, 1ml per ampule.

Kerosene Standard (unweathered)

Cat.#	31229	each
	31229-500	ea. w/data pack
	31229-510	5pk.
	31229-520	5pk. w/data pack
	31329	10 pk. w/data pack

Kerosene Standard - 25% Weathered

Cat.#	31230	each
	31230-500	ea. w/data pack
	31230-510	5pk.
	31230-520	5pk. w/data pack
	31330	10 pk. w/data pack

Kerosene Standard - 50% Weathered

Cat.#	31231	ea.
	31231-500	ea. w/data pack
	31231-510	5pk.
	31231-520	5pk. w/data pack
	31331	10 pk. w/data pack

Kerosene Standard - 75% Weathered

Cat.#	31232	each
	31232-500	ea. w/data pack
	31232-510	5pk.
	31232-520	5pk. w/data pack
	31332	10 pk. w/data pack

Weathered Kerosene Kit

Contains one ampule (1ml) each of the following products:

- Kerosene Standard (cat.# 31229)
- Kerosene Standard - 25% Weathered (cat.# 31230)
- Kerosene Standard - 50% Weathered (cat.# 31231)
- Kerosene Standard - 75% Weathered (cat.# 31232)

Cat.#	31238	per kit
	31238-500	w/data pack

Weathered Petrochemical Standards (cont.)

Diesel Fuel #2 (ASTM Class 5 Accelerant)

These standards are prepared from a **single source (one refinery)** product. The weathered materials indicate the percent weight loss from original starting material. These standards are prepared at 5000µg/ml in methylene chloride, 1ml per ampule.

Diesel Fuel #2 Standard (unweathered)

Cat.#	31233	each
	31233-500	ea. w/data pack
	31233-510	5pk.
	31233-520	5pk. w/data pack
	31333	10 pk. w/data pack

Diesel Fuel #2 Standard - 25% Weathered

Cat.#	31234	each
	31234-500	ea. w/data pack
	31234-510	5pk.
	31234-520	5pk. w/data pack
	31334	10 pk. w/data pack

Diesel Fuel #2 Standard - 50% Weathered

Cat.#	31235	each
	31235-500	ea. w/data pack
	31235-510	5pk.
	31235-520	5pk. w/data pack
	31335	10 pk. w/data pack

Diesel Fuel #2 Standard - 75% Weathered

Cat.#	31236	each
	31236-500	ea. w/data pack
	31236-510	5pk.
	31236-520	5pk. w/data pack
	31336	10 pk. w/data pack

Weathered Diesel Fuel #2 Kit

Contains one ampule (1ml) each of the following products:

Diesel Fuel #2 Standard (cat.# 31233)
Diesel Fuel #2 Standard - 25% Weathered (cat.# 31234)
Diesel Fuel #2 Standard - 50% Weathered (cat.# 31235)
Diesel Fuel #2 Standard - 75% Weathered (cat.# 31236)

Cat.#	31239	per kit
	31239-500	w/data pack

ASTM E1387-90 Fire Debris Analysis

Adequate column resolution is addressed in this protocol. Any capillary column can be used provided resolution of all analytes can be achieved. To demonstrate performance, a column resolution check mix must be analyzed prior to any sample analysis. Listed below is the required column performance mixture.

E1387-90 Column Resolution Check Mix

Contains the compounds listed at 2000µg/ml each in methylene chloride. Packaged 1ml per ampule.

hexane	octane	decane
dodecane	tetradecane	hexadecane
octadecane	eicosane	toluene
p-xylene	2-ethyltoluene	3-ethyltoluene
1,2,4-trimethylbenzene		

Cat.#	31224	each
	31224-500	ea. w/data pack
	31224-510	5pk.
	31224-520	5pk. w/data pack
	31324	10 pk. w/data pack

Introducing ...

Customer Choice Packaging

Since introducing chemical standards in 1990, we have offered customers a choice of purchasing either single ampules, single ampules with data packs, or economical 10 packs with data packs. Many laboratories may not be able to use 10 ampules of the same mixture within a reasonable period of time, but often purchase multiple single units.

To meet the needs of these customers, we are now offering environmental standards in 5-packs. The customer can choose a 5-pack at either a discount (compared to single ampule purchase) or a FREE data pack (for audit compliance).

For Restek environmental standards not listed here, use the five digit catalog number in our literature and add a three digit suffix for the product which best meets your needs. For example:

cat.# 31006 SV Internal Standard Mix
ADD "-510" for a 5-pack at a discount (31006-510)
ADD "-520" for a 5-pack with FREE data pack (31006-520)

The Choice is Yours!

Analysis of Cholesterol and Other Dietary Sterols

The Importance of Cholesterol

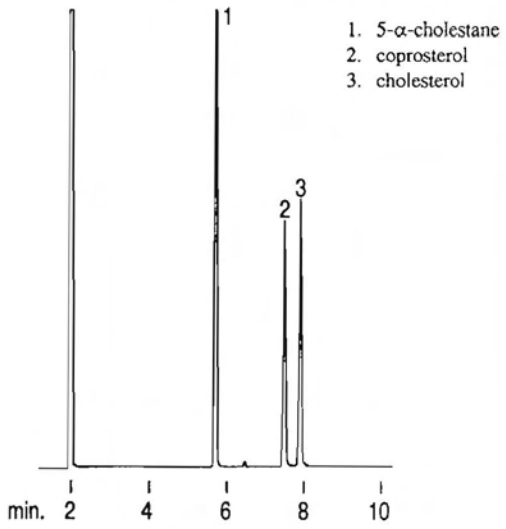
The association of elevated levels of blood serum cholesterol with increased risk of heart disease has been widely publicized over the last decade. Since more people are monitoring their cholesterol intake, the demand for the qualitative and quantitative determination of cholesterol content in many foodstuffs such as butter, eggs, baked goods, etc., is rather significant. Capillary gas chromatography provides an efficient means of cholesterol analysis (AOAC methods 970.51, 976.26). (1) Figure 1 shows cholesterol and its metabolite coprosterol, along with the internal standard 5-α-cholestane, elute in less than 10 minutes on a 30m, 0.25mm ID, 0.50μm XTI®-5. This column is an excellent choice for cholesterol because it is thermally stable to 330°C. It yields minimal stationary phase bleed and offers short analysis times.

The analysis of other sterols, such as plant sterols, are important as well. Plant sterols, or phytosterols, are constituents of food products such as vegetable oils. Brassicasterol, campesterol, stigmasterol, and β-sitosterol are commonly found in soybean, canola, olive and other vegetable oils that now replace cholesterol-rich sources of fat in the typical diet of the health-conscience consumer. Although similar in structure to cholesterol, plant sterols are not as readily absorbed as cholesterol, and decrease the artherogenic effects that cause heart disease in humans.

Some plant sterols, such as stigmasterol and ergosterol for example, are important due to their involvement in hormone production. Stigmasterol is used in the preparation of progesterone, and ergosterol is used to produce estradiol. (2)

All of the plant sterols mentioned above, combined with cholesterol, coprosterol, and lanosterol (a sterol found in wool wax) are well resolved on the XTI®-5 in under 12 minutes (Figure 2). Thus, qualitative and quantitative determination of many significant sterols can be performed using the XTI®-5 column at 330°C with 5-α-cholestane as an internal standard. ■

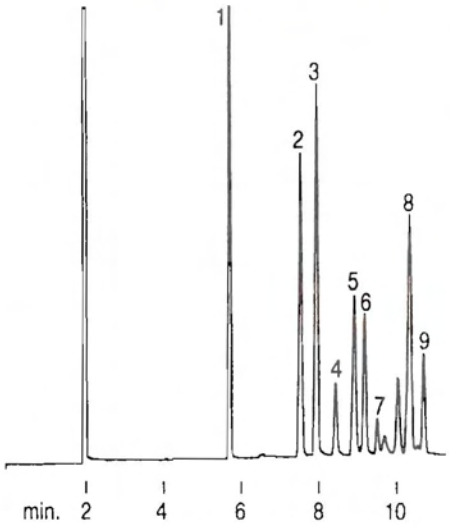
Figure 1 - Analysis of Cholesterol (AOAC 976.26) and Metabolite



30m, 0.25mm ID, 0.50μm XTI®-5 (cat.# 12238)
1.0μl split injection of cholestane, coprosterol, cholesterol
on-column conc.: 250ng

Oven temp.:	330°C isothermal
Inj. & det. temp.:	300°C
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 40°C
FID sensitivity:	8×10^{-11}AFS
Split ratio:	100:1

Figure 2 - Many sterols are separated using a single XTI®-5 column.



30m, 0.25mm ID, 0.50μm XTI®-5 (cat.# 12238)
1.0μl split injection
on-column conc.: 250ng

Oven temp.:	330°C isothermal
Inj. & det. temp.:	300°C
Carrier gas:	helium
Linear velocity:	30cm/sec. set @ 40°C
FID sensitivity:	8×10^{-11}AFS
Split ratio:	100:1

References

1) *AOAC Official Methods of Analysis*, 15th ed., 1990. pp. 976-7, 1103-1105.

2) Cook, R.P., *Cholesterol*, 1958. pp. 4-5.

Extending Septa Life

Factors Affecting Puncturability and Fragmentation

A good septum must possess more than low bleed to meet today's laboratory requirements. Long life and low fragmentation are important to minimize instrument downtime. If a laboratory can make 500 injections instead of 50 injections before changing the septum and inlet sleeve, then more analyses can be performed and the cost per analysis can be reduced. The time savings is greater than initially apparent when you consider the time it takes the system to re-equilibriate each time inlet maintenance is performed.

Thermolite® septa have been shown to exhibit the least amount of septum bleed and produce the least amount of artifacts or ghost peaks during a blank run. (1) But what about longevity and fragmentation? How many punctures can be made until the septum starts leaking and why? What causes septa fragments to be deposited in the injection port? These are questions our research group sought to answer in-order to provide the best, lowest maintenance septa possible.

Our experimental work focused on defining the variables that affect septa lifetime. The factors include:

- septum torque or tightness
- manual versus autosampler injections
- septum nut design

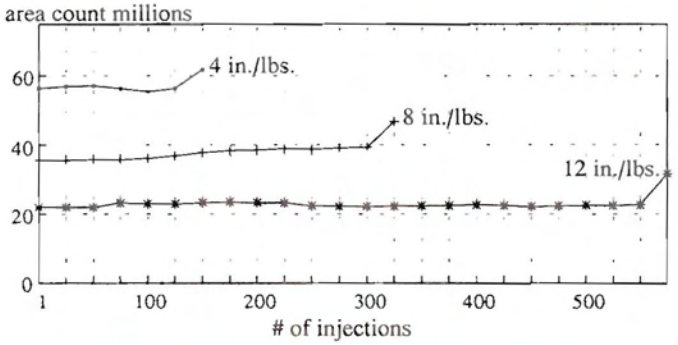
Most work in this study was performed using an HP 5890 GC with manual and autosampler injections. A PE Autosystem was used to double check the data. We expect Varian, Shimadzu, and other GCs to perform similarly but time did not permit verification prior to publication of this article. Testing was done by repeatedly injecting methanol and monitoring retention times and area counts. Septum leakage was monitored by sampling the air above the septum nut by using a thermal conductivity (TC) leak detector. Fragmentation was quantified by packing straight inlet sleeves with glass wool and measuring the amount of septa fragments deposited in the sleeves after a series of injections.

Interestingly, changes in peak area counts proved to be the best indicator of leaks. Since the HP 5890 GC is a back pressure controlled inlet, the component area count increased drastically as a septum leak developed. The increase in area count was directly related to the split ratio lowering as the back pressure regulator reduced flow to the split vent to compensate for the septum leak (2). Changes in peak areas did not occur with the head pressure controlled PE Autosystem. Septum leaks went undetected unless a TC leak detector was used to test septum integrity.

Septum Nut Torque

Torque was the most significant factor that influenced septum lifetime. In general, the tighter a septum nut, the more injections could be obtained until leakage occurred. Figure 1 shows a graph that compares 4, 8, and 12 inch pounds of torque with 11mm septum in an HP autosampler. A septum leak is signified when the area counts deviate sharply from a straight line. The same study was repeated with a 10mm septum and showed similar results in the HP inlet. Surprisingly, even when the septum nut was tightened at 20 inch pounds of torque, the autosampler syringe easily pierced the septum without bending.

Figure 1 - Septa Life Increases as Torque Increases

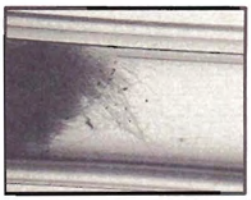


20m, 0.18mm ID, 0.40µm Rtx®-1 (cat.# 40111)
1µl split injection of methanol
GC: HP 5890 Series II w/HP 7673 autosampler
Oven temp.: 100°C isothermal
Inj./Det. temp.: 260°C
Detector: FID
Carrier gas: hydrogen

Manual vs. Autosampler Injections

Manual injections clearly caused more fragmentation and coring of the septum than injections made with an autosampler. Figure 2 shows a significant amount of septum particles deposited in an inlet sleeve after only 100 manual injections. Figure 2 also shows that fragmentation is almost non-existent even after 800 autosampler injections. This data indicates that using an autosampler will significantly minimize the need to perform inlet maintenance from septa fragmentation.

Figure 2 - Manual injections significantly increase fragmentation.



Septa fragments in Sleeve #1
Using Autosampler
After 100 injections

Septa fragments in Sleeve #2
Using Manual Injection
After 100 injections

Septa fragments in Sleeve #3
Using Autosampler
After 800 injections

Septum Nut Design

Autosamplers produce little fragmentation because the syringe needle penetrates the septum in the same place each time creating only one small hole. In addition, the high speed injection made by the HP and PE autosampler further reduces damage to the septum. Manual injections allow a wider area of needle penetration and cause the septa to fragment and fall apart. The needle traps fragments upon insertion and deposits them into the inlet during sample injection. In order to reduce the area of penetration, we tested an HP septum nut with a small needle guide that closely matched the OD of a 26 gauge needle to direct the syringe through the same hole during each injection. Figure 3 shows that fragmentation and coring was drastically reduced with a 26 gauge needle guide was compared to the standard size needle guide. Figure 3 also shows that the autosampler still produces less fragmentation and coring than the 26 gauge needle guide indicating that the speed of penetration also has a profound effect on coring.

Figure 3 - Septum coring is significantly reduced when the needle guide directs the syringe through the same hole.

Septum 1 ***Septum 2*** ***Septum 3***

***Septum 1** - coring from 100 manual injections with a standard HP septum nut.*

***Septum 2** - coring is reduced when a needle guide that closely matches the syringe OD is used.*

***Septum 3** - 800 injections with an autosampler still produces less coring than with a needle guide.*

The experimental results show that many factors affect Thermolite® septa lifetime with torque being the primary variable. In general, septa lifetime increased with the tightness of the septum nut. At a torque of 12 inch pounds, over 500 injections could be made without leakage. No difference was discerned between a 10 or 11mm septum for an HP split/splitless inlet. Injections made by an autosampler always resulted in longer septa lifetime and less fragmentation. The use of a special septum nut with a needle guide that closely fits the syringe OD significantly reduced coring and fragmentation for manual injections and approached the performance of an autosampler. ■

References

1) Request Restek's *Guide to Minimizing Septa Problems* for data showing bleed comparisons with other manufacturer's septa.
2) Request Restek's *Operating Hints for Split/Splitless Injectors* for more details on back pressure and head pressure designed inlets.

Product Listing

Needle Guide Septum Nut for HP 5890 GCs:
cat.# 21309 each
(Please see pg. 14 for complete product description.)

Thermolite® Septa

Septum Diameter	25-pack cat.#	50-pack cat.#	100-pack cat.#
5mm (3/16")	20351	20352	20353
6mm (1/4")	20355	20356	20357
7mm	20381	20382	20383
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

Instrument	Septum Size
Hewlett-Packard	
5890 series	10mm/11mm
5700, 5880 series	9.5mm/10mm
Varian	
packed column injector	9.5mm/10mm
split/splitless injector	10mm/11mm
Perkin-Elmer	
Sigma series, 900, 990	11mm
8000 series	11mm
Tracor	
550, 560	9.5mm
220, 222	12.5mm
Gow-Mac (all models)	9.5mm
Fisons/Carlo-Erba	17mm
8000 series	
Pye/Unicam	7mm

Effective January 1, 1994, septa prices will increase - so stock up now!

Hints for the Capillary Chromatographer

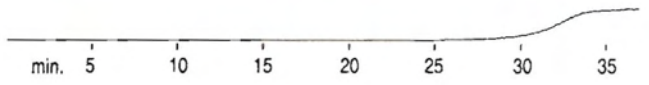
When is column bleed normal and when is it a problem?

What is column bleed?

Column bleed is the normal elution of stationary phase as the GC column is temperature programed. All columns will show a certain amount of bleed as the oven temperature increases. The question is whether the bleed is normal or excessive for your column and conditions. Column bleed only becomes a problem when it either interferes with quantitation or when it contaminates the detector.

A typical bleed profile for a temperature programmed run is shown in Figure 1. Column bleed is characterized by a gradual baseline rise which reaches a plateau at the final temperature of the program. This rise typically begins approximately 20 to 30°C below the maximum operating temperature of the column. Notice that there are no discrete peaks present in column bleed. The type of stationary phase as well as the dimensions of a capillary column will affect the amount of column bleed. For example, a polar phase usually exhibits more bleed than a non-polar phase. In general, the more stationary phase a column contains, the higher the column bleed. A long, wide bore, thick film column has more bleed associated with it than a short, narrow bore, thin film column. Operating at higher temperatures also increases bleed.

Figure 1 - Blank run showing normal column bleed.



30m, 0.25mm ID, 0.25µm XTI®-5 (cat.# 12223)

Oven temp.:	35°C (hold 1 min.) to 350°C @ 10°C/min. (hold 5 min.)		
Inj./det. temp.:	300°C	**Detector:**	MSD
Carrier gas:	helium	**Linear velocity:**	30cm/sec. set @ 35°C
Split ratio:	10:1	**Ionization:**	EI
Scan range:	50-400		

What are the most common causes of high column bleed?

It is important to recognize that there are different causes of excessive column bleed. Studies have shown (1) that there are several common GC problems which can cause high bleed. Let's consider the most common causes for excessive column bleed and what steps can be taken to minimize it.

The stationary phases used in capillary columns are susceptible to oxidation. If the column exhibits a high baseline rise, the column may have been subjected to oxygen at a relatively high temperature. This can be from a leak either in the injection port area or in the gas lines immediately preceding the injector. Oxygen can also be present in the carrier gas as a contaminant from the gas cylinder. It is important to prevent oxidation by using oxygen traps on the carrier gas lines and by carefully leak checking the flow system and inlet after column installation.

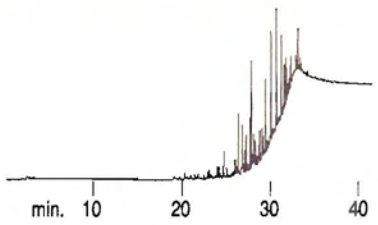
Exposing the column to high temperature without flow or operating the column at temperatures above the manufacturer's recommended maximum will also result in stationary phase damage. This most commonly occurs when a column is conditioned without confirming carrier gas flow, or when a cylinder of carrier gas empties during temperature programming. Restek recommends that flow through the column be verified, before conditioning, by either detecting a non-retained peak or by submerging the detector end of the column in a small vial containing methanol and observing the bubbles (Figure 2).

Figure 2 - Confirm column flow by submerging the column outlet in a vial of methanol.

If the column has been exposed to high molecular weight sample residue, the baseline may exhibit a rise similar to column bleed. When a column has been contaminated, discreet peaks are usually observed in the chromatogram at elevated temperatures. Frequently, solvent rinsing can rejuvenate the column by extracting the contamination. Phase degradation can also result from injecting samples containing strong acids or bases or excess derivatizing reagents.

Sometimes septum bleed is confused with column bleed because the electron impact spectra obtained with mass spectrometry are similar for both. Septa bleed is easily recognized as a distinct pattern of discrete peaks in a chromatogram, whereas column bleed normally does not result in individual peaks. In Figure 3, notice the pattern of multiple peaks just before the baseline begins to rise from the normal column bleed. The best techniques for minimizing septum bleed are using low bleed septa, frequently replacing used septa, using a septum purge, and completing a blank run when the column has been at temperatures below 100°C for several hours.

Figure 3 - Example of septum bleed.



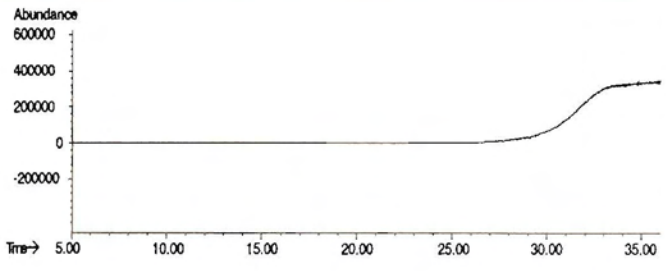
15m, 0.53mm ID, 1.0µm Rtx®-5 (cat.# 10252)
Oven temp.: 40°C (hold 15 min.) to 300°C @ 15°C/min.
Inj./det. temp.: 250°C/300°C
Carrier gas: hydrogen **Linear velocity:** 40cm/sec.

How can column bleed be accurately measured?

To determine how much column bleed is acceptable for an analysis, you must have an understanding of the necessary detection limits, the type of column being used, and the detector and signal sensitivity during operation. When analyzing trace components with very sensitive detectors, even a small amount of column bleed can interfere with the analysis. If using long length, thick film columns, more bleed will be experienced than with short length, thin film columns. The combination of stationary phase type and detection system used can have a profound effect on how much column bleed is exhibited. For example, nitrogen sensitive detectors (TSDs or NPDs) would exhibit a higher baseline signal from a cyanopropylphenyl stationary phase than Flame Ionization Detectors (FIDs).

It is important to be careful when interpreting the chromatogram obtained with a blank run. Some data systems use an autoscale feature which normalizes the intensity axis to the largest peak in the chromatogram. If there are no peaks, then the chromatogram is drawn with the baseline at full scale, giving the illusion that the column has high bleed (Figure 4).

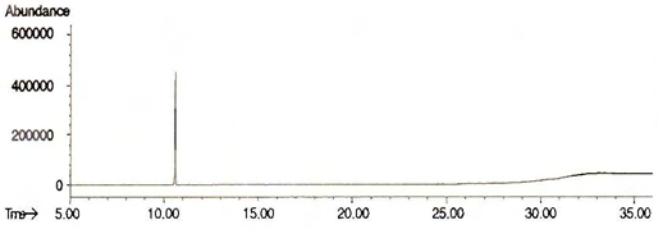
Figure 4 - Autoscaling can give the appearance of high bleed.



30m, 0.25mm ID, 0.25µm Rtx®-1 (cat.# 10123)
Oven temp.: 35°C (hold 1 min.) to 340°C @ 10°C/min. (hold 5 min.)
Inj./det. temp.: 300°C **Detector:** MSD
Carrier gas: helium **Linear velocity:** 30cm/sec. set @ 35°C
Ionization: EI **Scan range:** 50-400

A simple way of accurately measuring column bleed is to inject a known concentration (i.e. 25ng on-column) of a component that shows good response on the detector being used and temperature program the column to its maximum temperature. Measure the peak height of the component and compare it to the baseline offset from the bleed. Although the relative intensities of these two values depend upon several factors, these values can serve as a reference point to compare with other columns and systems. Figure 5 shows the bleed level on the same column shown in Figure 4, however, a 25ng injection of naphthalene was included as part of the blank run. Notice that Figures 4 and 5 have the same absolute amount of bleed (500,000 counts), but the bleed level in Figure 5 appears much lower because the plot is scaled relative to the 25ng naphthalene peak. Without the naphthalene injection, an analyst can be fooled into believing that the bleed level is much higher than it actually is.

Figure 5 - Baseline bleed compared to 25ng naphthalene.



Sample: 25ng on-column injection of naphthalene
See Figure 4 for other conditions.

How can column bleed be minimized?

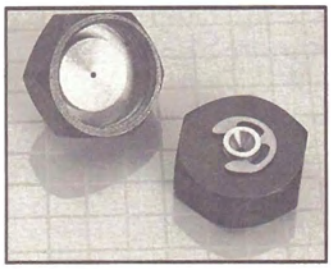
To minimize column bleed, there are several precautionary measures. All systems should be installed with oxygen and moisture traps on the carrier lines. When installing a column it is important to check the entire system for leaks. This includes any column connections, injection port fittings, and carrier lines. All columns should be conditioned following the manufacturer's recommendation. Additional routine conditioning may be required to remove high molecular weight residue, depending on the type of samples you are running. If the column becomes extremely contaminated from dirty samples, rinsing the column may be necessary in order to rejuvenate it. Routine replacement of the septum will eliminate leaks resulting from coring and/or cracking. On GC/MS systems, it is very easy to monitor for air and water leaks. Acceptable levels of air and water vary from system to system, so check with the manufacturer for the recommended limits.

Once there is a leak free system and the column is conditioned, make an injection of a standard sample and program the column to its maximum temperature. The relative height of the peak to the height of the maximum baseline will give a fair assessment of the column bleed. ■

1) M.A. Hayes, J.J. Harland, H.D. Rood and K.T. Klatt, "Proceedings of the Tenth Int. Symp. on Cap. Chrom.", May (1989).

Peak Performers

Needle Guide Septum Nut for HP 5890 GCs



Increase septa lifetime and decrease maintenance requirements with Restek's new septum nut for 26 gauge needles. This new septum nut directs the needle through the same hole, minimizing coring and leakage. For additional details on how our Needle Guide Septum Nut can extend septa lifetime, see the article, "Extending Septa Life", on pages 10-11. (Similar to HP part number 18740-60835 except with 26 gauge hole).

Needle Guide Septum Nut for HP 5890 GC:
cat.# 21309 each

0.3mm ID Ferrules for the Capillary Vu-Union™

Due to numerous customer requests, we have introduced 0.3mm ID graphite and Vespel®/graphite ferrules for the Vu-Union™. The 0.3mm ID ferrule fits 0.18 or 0.22mm ID tubing and is also recommended for some manufacturers 0.25mm ID tubing with ODs close to 0.3mm.

0.3mm ID graphite ferrules: cat.# 20233, 10pk.
0.3mm ID Vespel®/graphite ferrules: cat.# 20423, 10pk.

ezGC™ and *Pro ezGC™* Method Development Software

ezGC™ and *Pro ezGC™* method development software calculates the peak widths and retention times for a given set of chromatographic conditions and then displays the resulting chromatogram. In addition, the software predicts the optimum temperature program for a given analysis that provides baseline resolution in the shortest time. The advanced features of *Pro ezGC™* make GC computer modeling even more powerful through accessing libraries generated in your lab or purchased through Restek.

***ezGC™* Software:** cat.# 21480
***Pro ezGC™* Software:** cat.# 21481
***ezGC™* to *Pro ezGC™* Upgrade:** cat.# 21482
All software includes both 5¼" and 3½" disks.

Retention Index Libraries:

Food & Flavor Volatiles: cat# 21451
Drugs & Pharmaceuticals: cat.# 21453
Environmental - Volatiles: cat.# 21452
Solvents & Chemicals (Part 1): cat.# 21450

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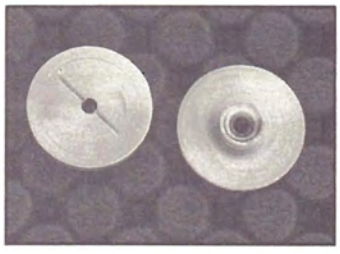
Order *Pro ezGC™* now and receive a free Retention Index Library. This offer ends December 31, 1993. Use the part numbers below when order your free library.

***Pro ezGC™* w/Food & Flavor Volatiles,** cat.# 21481-515
***Pro ezGC™* w/Drugs & Pharmaceuticals,** cat.# 21481-516
***Pro ezGC™* w/Environmental - Volatiles,** cat.# 21481-517
***Pro ezGC™* w/Solvents & Chemicals,** cat.# 21481-518

Improved Inlet Seals for HP 5890 GCs



The metal inlet seal at the base of an HP 5890 GC capillary injection port comes into contact with the sample when it vaporizes. This can cause decomposition of active components such as alcohols, pesticides, and drugs. To reduce breakdown and adsorption of active compounds, Restek offers gold plated and Silcosteel® treated inlet seals. The gold surface offers better inertness than standard stainless steel. Restek's unique Silcosteel® process places micron thin layers of fused silica and a deactivation layer over the stainless steel to provide inertness similar to a fused silica capillary column.

Gold Plated Inlet Seals (similar to HP part# 18740-20885):
cat.# 21305, 2-pk.
cat.# 21306, 10-pk.
Silcosteel® Treated Inlet Seals:
cat.# 21307, 2-pk.
cat.# 21308, 10-pk.

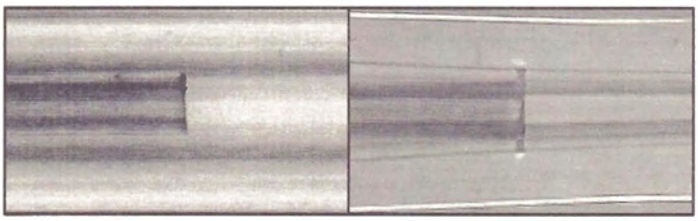
Direct Injection into HP 5890 Packed Column GCs

Restek has optimized the design of the direct injection inlet system for HP 5890 GC packed injection ports. The first area addressed was the tolerances on the disposable glass inserts. Chromatographers have expressed a desire to change the glass inserts by pulling them through the septum nut weldment at the top of the inlet. The current tolerances used by HP cause a large proportion of liners to get stuck at the top of the inlet, requiring complete removal of the direct injection metal sleeve adapter. Removal of the sleeve adapter increases maintenance time and forces the analyst to re-condition the column to stabilize the system. Restek closely monitors every glass insert to make sure it can be changed by simply removing the septum nut weldment and pulling it out with a needle file. In addition, each glass insert is deactivated with our high temperature silanization procedure to maintain the integrity of an inert fused silica capillary system.

Restek also developed a special glass insert (Uniliner®) that incorporates a press-tight taper at the base. The press-tight taper seals tightly onto the end of the fused silica capillary column reducing any chance of dead volume. Figure 1 shows the excessive dead volume between the column and standard HP type glass insert. The Uniliner® design seals tightly against the outside of a fused silica capillary, significantly reducing dead volume and peak tailing. Uniliner® type glass inserts are deactivated and designed to seal with 0.25, 0.32, and 0.53mm ID capillary columns.

The final area we addressed was to re-engineer the metal direct injection sleeve adapter. HP's sleeve adapter for packed purged ports requires a special male capillary nut that uses only short, non-standard ferrules. Restek's design uses a standard 1/16" Swagelok®-type male fitting that uses standard nuts and ferrules. We also strengthened the wrench pad at the base of the metal inlet sleeve to make it easier to tighten the column nut. Both the standard glass inserts and the Uniliner® type inserts work with either Restek's metal direct injection sleeve or HP's sleeve adapter for packed purged injection ports.

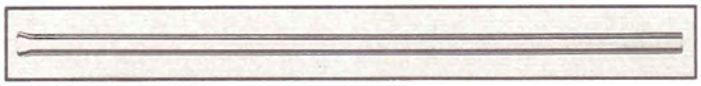
Figure 1 - The standard HP type glass insert allows dead volume, whereas the Uniliner® type insert seals tightly onto the column end.



Standard HP type glass insert *Uniliner® type insert*

DI Glass Inserts for an HP 5890 Packed Column GC

- Tolerances closely controlled.
- Can be removed from the septum nut weldment.
- Deactivated and fully inert to active compounds.
- Similar to HP part# 5181-3382 or #5080-8732.



cat.# 20967, 5-pk.
cat.# 20968, 25-pk.
cat.# 20969, 50-pk.

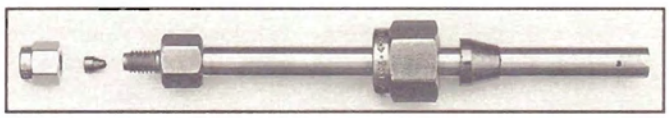
DI Uniliners® for an HP 5890 Packed Column GC

- Press-tight taper forms dead volume free connection to column.
- Minimizes solvent and peak tailing.
- Fits 0.25, 0.32, and 0.53mm ID MXT® or fused silica capillary columns.
- Can be removed from the septum nut weldment.
- Deactivated and fully inert to active compounds.
- Fits same sleeve adapter as HP part# 5181-3382 or #5080-8732.

cat.# 20964 each
cat.# 20965, 5-pk.
cat.# 20966, 25-pk.

DI Sleeve Adapter for an HP 5890 Packed Column GC

- Uses a standard 1/16" capillary nut and ferrules.
- Convenient wrench pad at base.
- Includes 1/4" graphite ferrule and SS nut.
- Works with HP or Restek's DI glass inserts or Restek's DI Uniliners® for an HP 5890 packed column GC.
- Similar to HP part # 19244-80540.



cat.# 21303, each

" some" **promos / Products / Offers** in the **ADVNews**
have been since been progressively superceded
/ UPDATED OR Since Discontinued
CHECK THE latest Restek ADVantage Newletter, Restek ESSENTIALS
. . . *Or* The Restek Catalog . . . *Or* other Resteb publications for updates
www.chromtech.net.au or NEW site 2015 > **www.chromalytic,net.au**

THE RESTEK ADVANTAGE

new! Micropacked GC Columns

Improve the Separation of Low Molecular Weight Compounds

- Higher efficiency compared to packed columns.
- Larger sample capacity than PLOT columns.
- All columns tested for packing and flow consistency.
- Silcosteel® tubing and endplugs improve inertness.
- Columns to fit packed and capillary injection ports
- Available with HayeSep® N, Q, R, S and Molecular sieve 5A and 13X packings.

Good efficiency and sample capacity

Column options for analyzing low molecular weight compounds have been limited. Packed columns filled with porous polymer packings offer one alternative, but yield limited resolution. Porous Layer Open Tubular (PLOT) capillary columns offer increased resolution, but have limited capacity. Also, small particles from PLOT columns can fragment from the column and cause contamination of valves and detectors. Another alternative for the analysis of low molecular weight compounds is micropacked columns.

Micropacked columns offer features from both packed and capillary columns. Due to their narrow diameter, micropacked columns exhibit a greater efficiency than standard packed columns. Micropacked columns offer much greater sample capacity than PLOT columns (1). Figure 1 shows a high concentration solvent mixture run by direct injection on a HayeSep® Q micropacked column. The symmetrical peak shape indicates no sample overloading even at these high concentrations.

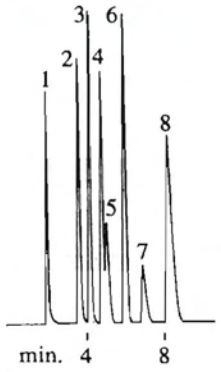
Guaranteed packing

All columns are individually tested for packing weight and flow consistency. Variation in packing weight and density is limited to less than 5% between columns. This results in micropacked columns that give reproducible retention times run- after-run and column-to-column.

Silcosteel® process improves inertness

All micropacked columns are made with Silcosteel® tubing and deactivated to provide greater inertness. The Silcosteel® process deposits a thin layer of fused silica over the active metal surface. The endplugs are made from braided stainless steel that has also been treated using the Silcosteel® process to insure that the sample only contacts inert surfaces. To demonstrate this improved inertness, a Silcosteel® treated micropacked column was compared to a non-Silcosteel® column. A mixture containing 1000ppm of 2,3-butanediol and n-octane was run on both columns at 220°C isothermal. Active

Figure 1 - HayeSep® Micropacked columns show excellent resolution and capacity.

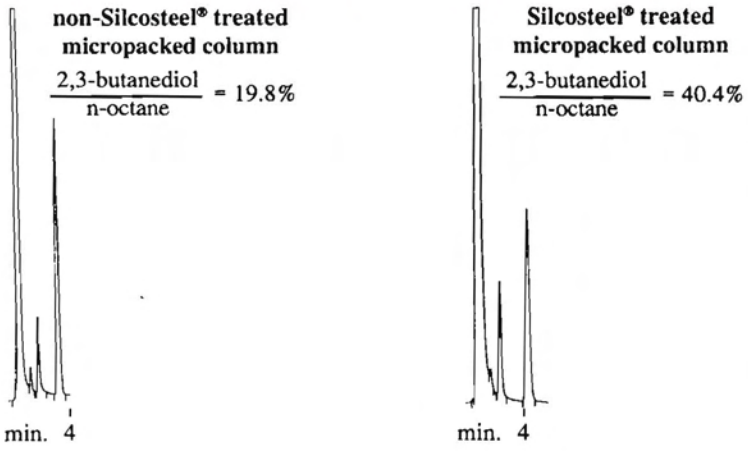


COMPOUNDS	
1	methanol
2	ethanol
3	acetonitrile
4	acetone
5	methylene chloride
6	n-pentane
7	chloroform
8	n-hexane

2m, 1mm ID HayeSep® Q Micropacked Column (cat.# 19107)
1ml direct injection of a neat solvent mixture
Oven temp.: 80° to 180°C @ 16°C/min. (hold 5 min.)
Inj. & det. temp.: 200°C **Carrier gas:** helium
Flow: 20ml/min. set @ 40°C
FID sensitivity: 512 x 10^{-11} AFS

in this issue...

Figure 2 - A Silcosteel treated micropacked column shows improved response of active compounds such as 2,3-butanediol.



2m, 1mm ID HayeSep® Q Micropacked Column (cat.# 19017)
0.5ml direct injection of 1000ppm mix of 2,3-butanediol and n-octane in methylene chloride
Oven temp.: 220°C isothermal
Inj. & det. temp.: 250°C **Carrier gas:** helium
Flow: 20ml/min. set @ 40°C
FID sensitivity: 32 x 10^{-11} AFS

compounds such as 2,3-butanediol will be adsorbed on untreated metal surfaces. Inert compounds such as n-octane do not exhibit adsorption. Therefore, the ratio of these components are a good indicator of column inertness. Figure 2 shows that the ratio of 2,3-butanediol to n-octane on the non-Silcosteel® treated column was only 19.8% and that the Silcosteel® treated micropacked column exhibited a ratio of 40.4%.

Micropacked columns fit packed and capillary injection ports

We have designed micropacked columns to fit both packed and capillary injection systems. The 1mm ID standard wall (1/16" OD) micropacked columns offer improved efficiency for packed column instruments without the expense of converting to capillary injection systems with no modifications. The 0.75mm ID thin wall (0.81mm OD) micropacked columns were designed to install directly into a capillary injector. Figure 3 shows a hydrocarbon mix run on a micropacked column using a split/splitless capillary injection system.

A wide range of porous polymer packings offered

Micropacked columns are available in a wide range of HayeSep® porous polymer packing materials. HayeSep® materials were chosen since they go through extensive preparation to yield the greatest column to column reproducibility currently available for porous GC packings. These materials cover a wide range of polarities for most common applications. Table I shows the composition, maximum temperatures, and common applications of the HayeSep® packings offered.

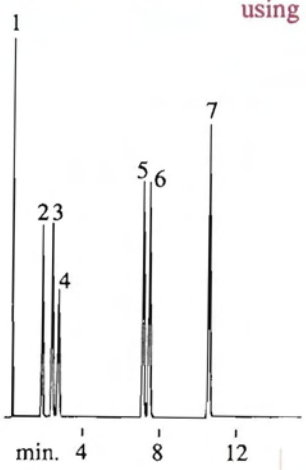
TABLE I

HayeSep® material properties

HayeSep® Material	Polymer Composition	Maximum Operating Temperature	Applications
N	DVB/EGDM	165°C	CO_2, Water, & Acetylene
Q	DVB	275°C	Hydrocarbons & Sulfur gases
R	DVB/NV2P	250°C	Methyl esters & Formaldehyde
S	DVB/4VP	250°C	Alcohols & Nitriles

DVB= divinylbenzene
4VP= 4-vinyl-pyridine
NV2P= N-vinyl-2-pyrollidinone
EGDM= ethyleneglycoldimethacrylate

Figure 3 - A hydrocarbon mix can be analyzed with a split/splitless capillary injection system using a 2m X 0.75mm ID micropacked column.



COMPOUNDS	
1	methane
2	ethylene
3	acetylene
4	ethane
5	propylene
6	propane
7	1-butene

2m, 0.75mm ID HayeSep® S Micropacked Column (cat.# 19011)
500ml split injection of a light hydrocarbon gas mixture
Oven temp.: 40°C (hold 3 min.) to 150°C @ 15°C/min. (hold 5 min.)
Inj. & det. temp.: 220°C **Carrier gas:** helium
Flow: 20ml/min. set @ 40°C
FID sensitivity: 32 x 10^{-11} AFS
Split ratio: 10:1

Two molecular sieve packings also available

Molecular sieve materials have shown a unique ability to separate permanent gases. Micropacked columns are available with either 5A or 13X molecular sieve packings. These two molecular sieve materials cover a wide range of permanent gas applications. Figure 4 show the analysis of permanent gases on a 13X micropacked column. At 40°C, all of the gases are separated in under 3 minutes.

TABLE II

Molecular Sieve Properties

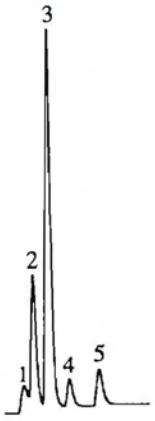
Material	Pore Size	Cation
5A	5A	Calcium
13X	10A	Sodium

Restek's new micropacked columns offer a unique mix of efficiency, sample capacity, and inertness. These columns provide the packed column user an improvement in separating efficiency. They offer the capillary chromatographer a less expensive alternative to PLOT columns for the separation of low molecular weight compounds. Micropacked columns also deliver improved sample capacity compared to PLOT columns. Available in a wide range of porous polymers and molecular sieves, these micropacked columns are ideal for the separation of low molecular weight compounds. ■

References

1) Tomas Herraiz, et. al., *Journal of Chromatography*, 388, pp. 325-333 (1987).

Figure 4 - A 13X molecular sieve micropacked column can separate permanent gases in less than 3 minutes.



COMPOUNDS	
1	hydrogen
2	oxygen
3	nitrogen
4	methane
5	carbon monoxide

2m, 1mm ID Molecular Sieve 13X Micropacked Column (cat.# 19005)
10ml direct injection of a permanent gas mixture (hydrogen spiked)
Oven temp.: 40°C isothermal
Inj./det. temp.: 150°C/100°C
Carrier gas: helium
Flow: 17ml/min. set @ 40°C
TCD filament temp.: 300°C

Particle Sizes for Micropacked Columns	
1mm ID	**0.75mm ID**
100/120 Mesh	120/140 Mesh

Micropacked Column Product Listing

Hayesep® R Micropacked				
length	1mm ID		0.75mm ID	
1 meter	19012	$75	19014	$75
2 meter	19013	$99	19015	$99
Hayesep® Q Micropacked				
length	1mm ID		0.75mm ID	
1 meter	19016	$75	19018	$75
2 meter	19017	$99	19019	$99
Hayesep® N Micropacked				
length	1mm ID		0.75mm ID	
1 meter	19020	$75	19022	$75
2 meter	19021	$99	19023	$99
Hayesep® S Micropacked				
length	1mm ID		0.75mm ID	
1 meter	19008	$75	19010	$75
2 meter	19009	$99	19011	$99

5A Molecular Sieve Micropacked				
length	1mm ID		0.75mm ID	
1 meter	19000	$75	19002	$75
2 meter	19001	$99	19003	$99
13X Molecular Sieve Micropacked				
length	1mm ID		0.75mm ID	
1 meter	19004	$75	19006	$75
2 meter	19005	$99	19007	$99

To order any Restek product, call 800-356-1688 (ext.3).

Analyze Sulfur Compounds in Petroleum Gases and Light Liquids

Using Selective Sulfur Detection with our New, 4.0µm Rtx™-1.

The presence of trace organic sulfur compounds in petroleum products can have detrimental effects on the lifetime and performance of catalysts used in hydrocarbon processing. Furthermore, government agencies have scheduled changes to the specifications for total sulfur concentrations in Phase II gasoline to a maximum of 40 ppm. High resolution gas chromatography with sulfur specific detection provides a technique for quantitation of individual sulfur components in a wide range of petroleum products. By combining the amounts of the individual components, it is also possible to determine the total sulfur concentration in the sample.

The American Society of Testing and Materials (ASTM) Committee D-2 on Petroleum products is evaluating a proposed test method[1] which utilizes a 30 meter x 0.32mm ID methyl silicone capillary column (Rtx™-1) and a sulfur specific detector (Sievers SCD). This method is applicable for measurement of individual sulfur compounds in hydrocarbon products that range from petroleum gases, to liquids with a final boiling point of 440°C. The advantage of this method compared to methods which determine a total sulfur concentration is that the system can be calibrated to report the composition and concentration of the organic sulfur components such as H_2S, COS, mercaptans, thiophenes, benzothiophenes, sulfides and disulfides. This additional information may be useful in optimizing sulfur removal processes employed in hydrocarbon processing and gasoline refining.

Figure 1 shows a typical analysis of the sulfur components in gasoline using the Rtx™-1 column and the Sievers SCD. A new, thick film (4µm) column was selected for this method so that a single column can be used for a wide range of compounds from the more volatile sulfur gases to the higher molecular weight dimethylbenzothiophenes. This film thickness also increases the column capacity for the higher concentration hydrocarbons so that column overloading does not adversely effect the peak shape of the sulfur compounds.

It is also important that the column have low bleed to avoid a loss of sensitivity in the SCD from the buildup of silicone dioxide deposits on the reaction tube. Restek's 4µm Rtx™-1 column provides low bleed and does not degrade the sensitivity of sulfur specific detectors. Each column is preconditioned and individually tested using a temperature programmed QA test to ensure low bleed.

The Sievers detector can be operated for simultaneous detection of hydrocarbons and sulfur using a modified flame ionization detector. The chromatogram in Figure 2 shows the corresponding hydrocarbon response from the same gasoline analysis shown in Figure 1. The SCD is reported to be very sensitive and specific for organic sulfur compounds and does not have the quenching problems associated with flame photometric detectors[2]. Another detector suitable for the proposed ASTM test method is the HP 5921A Atomic Emis-

Figure 1 - 300ppm Total Sulfur in Gasoline by SCD

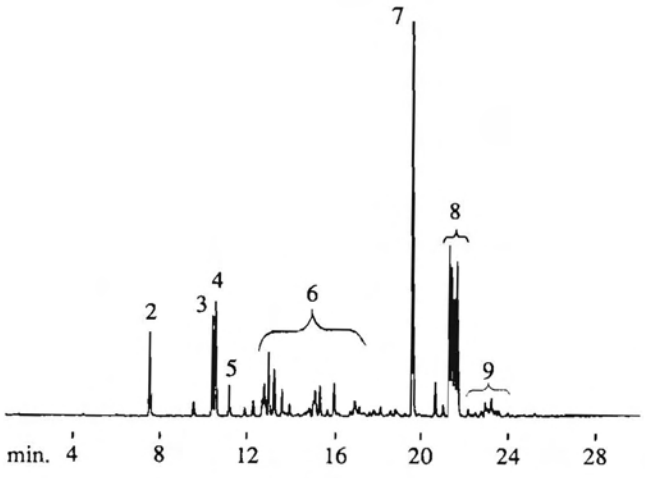
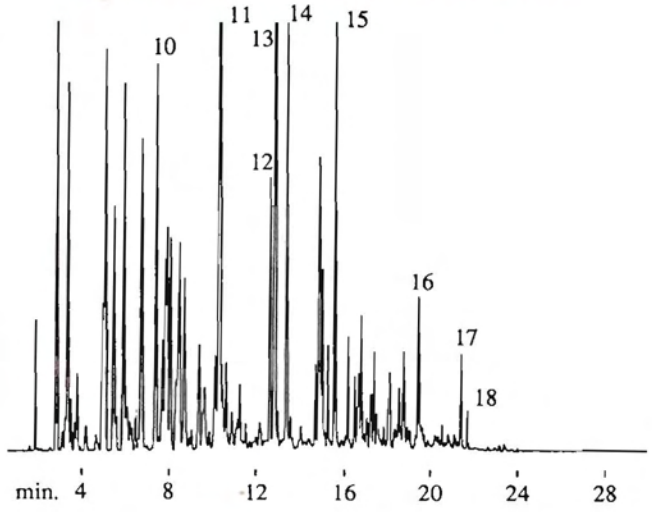
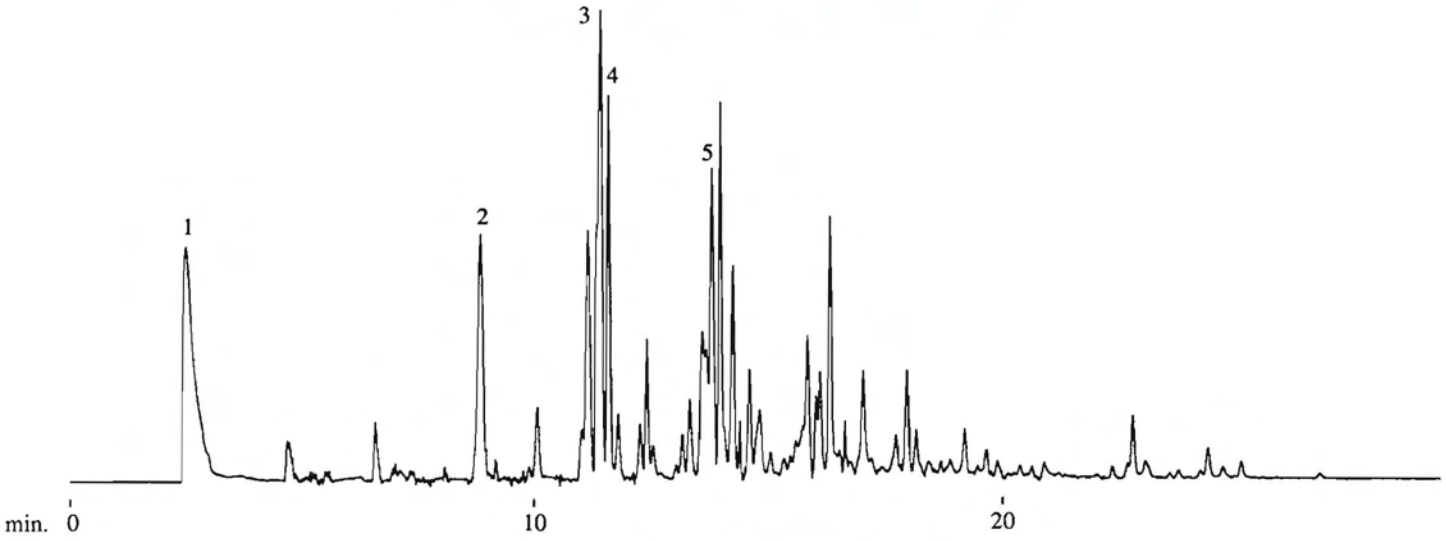


Figure 2 - Hydrocarbons in Gasoline by FID



30m, 0.32mm ID, 4.0µm Rtx™-1 (cat.# 10198)
1.0µl split injection of gasoline containing 300ppm total sulfur
Oven temp.: 40°C (hold 3 min.) to 275°C @ 10°C/min. (hold 5 min.)
Inj. & det. temp.: 275°C **Carrier gas:** helium
Linear velocity: 70cm/sec. (2.5ml/min.)

Figure 3 - 500ppm Total Sulfur in Naphtha by AED



30m, 0.32mm ID, 4.0µm Rtx™-1 (cat.# 10198)
1.0µl split injection of Naphtha containing 500ppm total sulfur
Oven temp.: 35°C to 275°C @ 10°C/min. (hold 5 min.)
Inj. & det. temp.: 275°C **Carrier gas:** helium
Linear velocity: 24cm/sec. (0.8ml/min.)

sion Detector (AED). By monitoring the 181 nm emission line of the AED[3], a sulfur specific chromatogram is obtained. Figure 3 shows the analysis of a petroleum naphtha containing approximately 500ppm total sulfur using the Rtx™-1 column and an AED.

High resolution gas chromatography along with the detection capabilities of the SCD and AED detectors provides a powerful analytical technique for determining total sulfur and individual sulfur components in a variety of hydrocarbon products. The 4µm Rtx™-1 column offers the resolution, inertness, capacity, and low bleed needed to meet the requirements of the proposed ASTM test method. ■

References

1. ASTM Committee D-2 Proposed Standard Test Method for the Determination of Sulfur Compounds in Petroleum Gases and Light Liquids by Gas Chromatography and Chemiluminescence Detection.

2. S.E. Eckert-Tilotta, S.B. Hawthorne and D.J. Miller *Journal of Chromatography*, 591 (1992) 313-323

3. B. D. Quimby and P.C. Dryden, "Multielement Simulated Distillation with the HP 5921A Atomic Emission Detector", Hewlett Packard Application Note 228-205.

Peak List for Figures 1-3

	COMPOUNDS
1	hydrogen sulfide
2	thiophene
3	2-methylthiophene
4	3-methylthiophene
5	2-ethylthiophene
6	alkylthiophenes
7	benzothiophene
8	methylbenzothiophenes
9	dimethylbenzothiophenes
10	benzene
11	toluene
12	ethylbenzene
13	m & p-xylene
14	o-xylene
15	1,2,4-trimethylbenzene
16	naphthalene
17	1-methylnaphthalene
18	2-methylnaphthalene

Product Listing

30m, 0.32mm ID, 4.0µm Rtx™-1, cat.# 10198, $425

Rtx™-200, Methyl Trifluoropropyl

Unique Selectivity Makes It Ideal for Many Analyses

• *Excellent thermal stability* • *Low bleed with FIDs, ECDs, and MSDs* • *Ideal confirmational column* •

Trifluoropropyl stationary phases have unique selectivity due to the electrophilic nature of the fluorine atom in the polymer backbone. This selectivity intensifies interactions with compounds that are electron rich such as ketones, nitro compounds, and Freon®s. This selectivity often resolves compounds that phenyl, cyano, and Carbowax® containing phases cannot.

While trifluoropropyls have been recognized for their unique selectivity, they have also suffered from low thermal stability, high bleed, poor inertness, and incomplete bonding of the stationary phase to the fused silica surface. Our trifluoropropyl polymer, the Rtx™-200, eliminates the common problems associated with typical trifluoropropyl phases. Because of the complete surface deactivation and high phase purity, inertness is exceptional, allowing highly active compounds to elute without tailing or adsorbing onto the column surface. Since the polymer is bonded to the surface and completely crosslinked, it can be solvent rinsed with a wide variety of solvents to clean sample residue from the polymer.

Extended thermal stability & low bleed

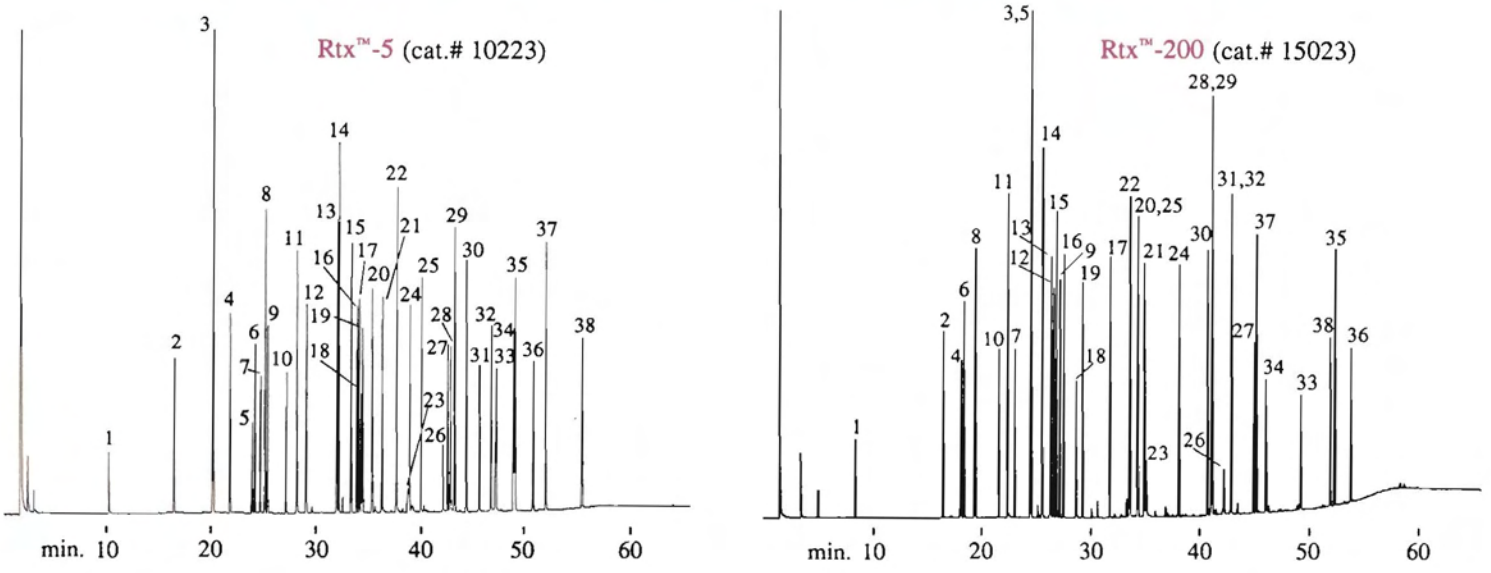
Rtx™-200's have the highest thermal stbility of any commercially available trifluoropropyl phase. The fluorine groups on an Rtx™-200 would normally produce high bleed with an ECD. However, the crosslinking technology developed for this phase results in extremely low ECD bleed, even at high temperatures. The Rtx™-200 polymer is stable to 360°C, however, the useable upper temperature limit for thicker films has been decreased to temperatures that provide tolerable bleed for most detectors.

Rtx™-200 can be used for a wide variety of applications

Toxicological analyses

Testing for the presence of drugs in biological fluids is a technique that is commonly used in drug overdose and drug abuse screening. The Rtx™-200 is an excellent column choice for toxicological analyses because of its selectivity for nucleophilic drugs. Compounds with electron donating groups, such as carbonyl, azo, and nitro, are preferentially retained on the Rtx™-200 when compared to compounds with similar base structures that do not contain these groups.

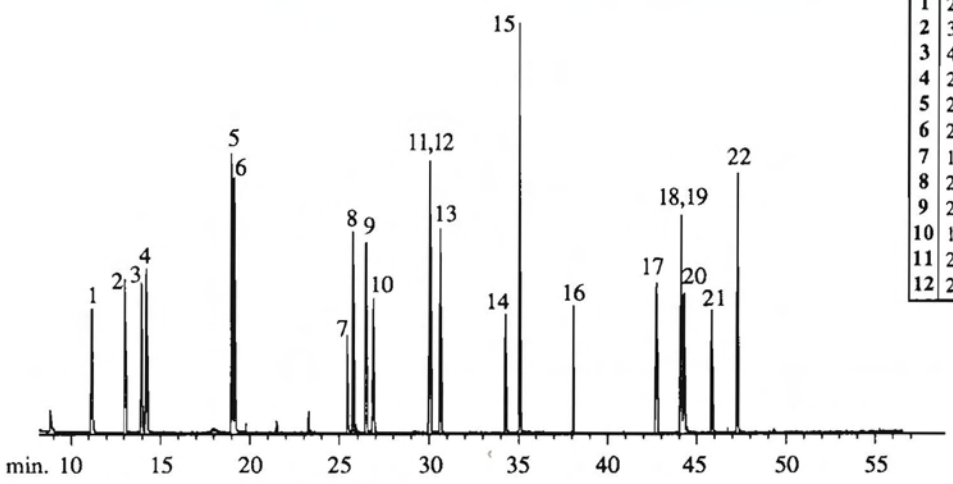
Figure 1 - Rtx™-200's unique selectivity improves specific separations of hard to resolve compounds.



30m, 0.25mm ID, 0.25µm
1.0µl split injection of 100µg/ml (2ng on-column)
Oven temp.: 100°C to 320°C @ 4°C/min. (hold 10 min.)
Inj./ det. temp.: 225°C/300°C
Carrier gas: helium
Linear velocity: 30cm/sec. @ 100°C
TSD sensitivity: 4 x 10^{-12} AFS
Split ratio: 50:1

1	benzocaine	9	chlorpheniramine	17	promazine	25	prazepam
2	cotinine	10	dextromethorphan	18	codeine	26	flurazepam
3	meperidine	11	methadone	19	morphine	27	haloperidol
4	caffeine	12	amitriptyline	20	diazepam	28	alprazolam
5	lidocaine	13	trimipramine	21	chlorpromazine	29	triazolam
6	phencyclidine	14	imipramine	22	chlorprothixene	30	thioridazine
7	doxylamine	15	medazepam	23	clobazam	31	verapamil
8	phenyltoloxamine	16	pentazocine	24	flunitrazepam	32	strychnine

Figure 2 - The Rtx™-200 yields excellent separation of nitro-explosive compounds.



1	2-nitrotoluene	13	2,3-dinitrotoluene
2	3-nitrotoluene	14	3,4-dinitrotoluene
3	4-nitrotoluene	15	3-nitrobiphenyl
4	2,3-diaminotoluene	16	2,4,6-trinitrotoluene
5	2,6-diaminotoluene	17	2,4,5-trinitrotoluene
6	2,4-diaminotoluene	18	4-amino-2,6-dinitrotoluene
7	1,4-dinitrobenzene	19	2,3,4-trinitrotoluene
8	2,6-dinitrotoluene	20	1,3-dinitronaphthalene
9	2-amino-6-nitrotoluene	21	2,6-diamino-4-nitrotoluene
10	1,3-dinitrobenzene	22	2-amino-4,6-dinitrotoluene
11	2,4-dinitrotoluene	23	2,2'-dinitrobiphenyl
12	2-amino-4-nitrotoluene		

30m, 0.25mm ID, 0.25µm, Rtx™-200 (cat.# 15023)
1.0µl splitless injection of 20ng/µl explosives
Oven temp.: 80°C (hold 2 min.) to 260°C @ 3°C/min. (hold 2 min.)
Inj./ det. temp.: 280°C/300°C
Carrier gas: helium
Linear velocity: 20cm/sec. @ 80°C
Splitless hold time: .6 min.
MSD Scan range: 45-300

Figure 1 shows the analysis of acidic, basic, and neutral drugs on an Rtx™-5 and Rtx™-200. Large changes in elution order between the two can be attributed to the presence of specific functional groups in the analytes. Benzodiazepines exhibit strong affinity on the Rtx™-200 because of an azo and carbonyl group in the molecule. Cotinine and caffeine are retained significantly longer on the Rtx™-200 than the other early eluting compounds due to the presence of a carbonyl group on the ring of both compounds.

Explosive analysis

Due to the nature of explosive compounds, they are typically performed using HPLC with either a C-18 or a CN reverse phase column. A common problem that arises when using HPLC columns is that the isomer pair; 2,4-diaminotoluene and 2,6-diaminotoluene are only partially resolved. The resolution can be reduced when one isomer exists at a much higher concentration than the other, causing a complete coelution between the two isomers. The Rtx™-200 column is ideal for analyzing explosives because of nitro groups in the analyte structure (Figure 2). The Rtx™-200 yields improved resolution between 2,6-diaminotoluene/2,4-diaminotoluene and excellent separation between 21 other explosives in under 60 minutes.

The Rtx™-200 is a highly selective stationary phase that is ideal for many types of analyses. Because of its unique selectivity, it offers analysts an alternative to other intermediate polarity columns. The maximum operating temperature, low column bleed, and excellent inertness make the Rtx™-200 a superb confirmation column. Rtx™-200's are available in a wide variety of lengths, film thicknesses, and diameters to solve your most difficult analytical separations. ■

Rtx™-200 Product Listing

length	df (µm)	0.25mm ID	0.32mm ID	0.53mm ID
15 meter	0.10	15005 $260	15006 $285	15007 $300
	0.25	15020 $250	15021 $275	15022 $290
	0.50	15035 $250	15036 $275	15037 $290
	1.00	15050 $250	15051 $275	15052 $290
	1.50		15066 $275	15067 $290
	3.00			15082 $290
30 meter	0.10	15008 $410	15009 $435	15010 $485
	0.25	15023 $400	15024 $425	15025 $475
	0.50	15038 $400	15039 $425	15040 $475
	1.00	15053 $400	15054 $425	15055 $475
	1.50		15069 $425	15070 $475
	3.00			15085 $475

length	df (µm)	0.25mm ID	0.32mm ID	0.53mm ID
60 meter	0.10	15011 $695	15012 $750	15013 $875
	0.25	15026 $685	15027 $740	15028 $850
	0.50	15041 $685	15042 $740	15043 $850
	1.00	15056 $685	15057 $740	15058 $850
	1.50		15072 $740	15073 $850
	3.00			15088 $850
105 meter	0.10	15014 $920	15015 $995	
	0.25	15029 $900	15030 $975	
	0.50	15044 $900	15045 $975	
	1.00	15059 $900	15060 $975	
	1.50		15075 $975	
	3.00			15091 $1400

length	df (µm)	0.18mm ID	length	df (µm)	0.18mm ID	length	df (µm)	0.18mm ID
10 meter	0.20	45001 $225	20 meter	0.20	45002 $350	40 meter	0.20	45003 $625
	0.40	45010 $225		0.40	45011 $350		0.40	45012 $625

Clinical Corner

Testing for Organic Volatile Impurities - Updates on USP 467

In the Third Supplement to the USP XXII-NF XVII, a new test for the analysis of Organic Volatile Impurities (OVI) in pharmaceutical products was published that became effective November 15, 1990. Since it's original appearance in the USP, this testing protocol has undergone many revisions and additions. Table I shows the current list of methods and their corresponding chromatographic systems. Many issues related to USP 467 have been addressed in the Pharmacopeial Forum including sample introduction, standard preparation, detection limits, sample amounts, and column selection.

Table I

	Column	Detector	Sample Introduction
Method I	5% phenyl/95% methyl polysiloxane (G27) 30m, 0.53mm ID, 5.0µm (cat.# 10279)	FID	Direct Aqueous Injection
Method II	1% polyethylene glycol/TPA (G25) on graphitized carbon (S12)	FID	Dynamic Headspace
Method III	1% polyethylene glycol/TPA (G25) on graphitized carbon (S12)	MSD	Dynamic Headspace
Method IV	6% cyanopropylphenyl/94% dimethylpolysiloxane (G43) 30m, 0.53mm ID, 3.0µm (cat.# 16085)	FID	Static Headspace
Method V	6% cyanopropylphenyl/94% dimethylpolysiloxane (G43) 30m, 0.53mm ID, 3.0µm (cat.# 16085)	FID	Direct Aqueous Injection
Method for Coated Tablets	0.2% polyethylene glycol/MW 1500 (G39) on graphitized carbon (S7)	FID	Static Headspace

Methods I and V are the most commonly used methods for OVI analysis. One of the drawbacks associated with these methods is the use of aqueous injections for sample introduction. High injection port temperatures can produce large expansion volumes for injections of as little as 1µl of water. When the expansion volume of the sample exceeds the buffer volume of the injection port liner, backflash can occur and some sample can be lost through the septum purge line. The injection port temperatures for Methods I and V were originally specified to be 180°C and 140°C respectively. At high temperatures, reproducibility of injections is poor. Data supplied by Bergren and Foust[1] demonstrated that a decrease in injection port temperature from 180°C to 70°C yielded lower relative standard deviations for peak area response for replicate injections. Revisions have been made to Method I to lower the injection port temperature to 70°C, but the injection port temperature for Method V has not been revised as of the date of this article.

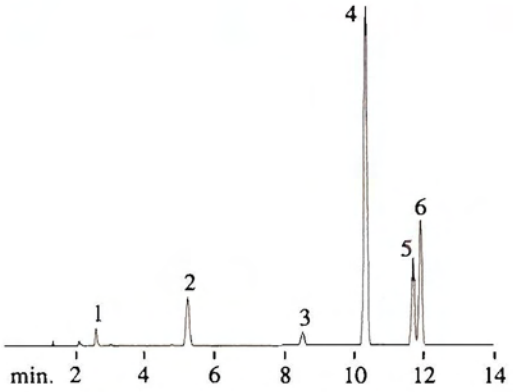
Poor reproducibility of peak area response is also related to the amount of analyte reaching the column. The response for chloroform on an FID is very poor. At the concentrations originally specified for the Standard Solutions, the chloroform response cannot be reproducibly detected above the baseline noise. Krasowski[2] et al. proposed two solutions that had been previously mentioned in the In Process Revision[3] comments in the Pharmacopeial Forum. The first was to double the concentration of OVIs in the Standard Solution and the second was to double the amount of bulk pharmaceutical used to produce the test solution. Both of these modifications increased the on-column concentration and produced a more consistent peak area response for each analyte. USP also increased the allowable relative standard deviation to 15% after finding that 10% was too demanding.

USP has also simplified the standard preparation procedure. The solubility for the OVI's in water is very poor and direct dissolution of these compounds in water is difficult. Standard stability and lifetime can be improved by using stock solutions of the OVI's prepared in DMSO and then making dilutions of the stock standard to produce working standards. Comments in the September/October 1992 Pharmacopeial Forum[4] propose the use of DMSO as the solvent for the stock standard.

Choosing a capillary column to perform OVI analysis has also been a subject for debate. Method I uses a capillary column interfaced with an FID with sample introduction via direct injection of an aqueous sample. The column specified is a 30 meter, 0.53mm ID, 5µm 5% phenyl/95% methyl polysiloxane (G27). When Method I was originally published, a resolution factor of 1.5 was included in the system suitability parameters. Figure 1 shows the analysis of the target compounds for USP 467 on the Rtx™-5 column. Peaks 5 and 6, trichloroethylene and 1,4-dioxane, are not completely resolved and have a calculated resolution factor (R) of 1.4. The criteria for a resolution factor of 1.5 was reduced to 1.0 in the Fifth Supplement in order to make the system suitability requirements easier to achieve. Method V was also introduced in the Fifth Supplement and incorporated the use of a 6% cyanopropylphenyl/94% dimethylpolysiloxane (G43) stationary phase as an alternative to the 5% phenyl/95% methyl polysiloxane stationary phase. Figure 2 shows the use of an Rtx™-1301 column, 6% cyanopropylphenyl stationary phase, for the analysis of organic volatile impurities. Baseline resolution of all of the compounds is obtained and the resolution criteria of 3.0 is easily met.

(**Clinical Corner** is continued on page 11.)

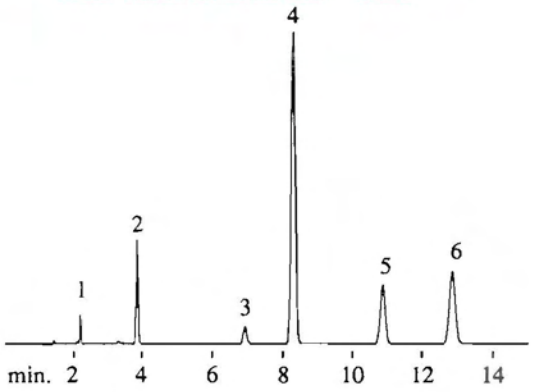
Figure 1 - USP 467 target compounds run on an Rtx™-5.



COMPOUNDS	
1	ethylene oxide
2	methylene chloride
3	chloroform
4	benzene
5	trichloroethylene
6	1,4-dioxane

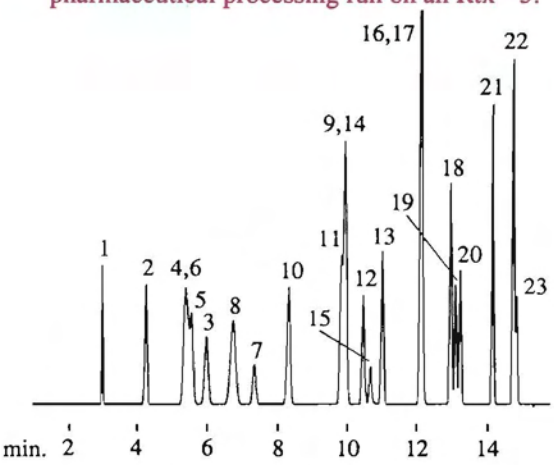
30m, 0.53mm ID, 5.0µm, Rtx™-5 (cat.# 10279)
1.0µl split injection of USP 467 Calibration Mix
Oven temp.: 35°C (hold 5 min.) to 175°C @ 8°C/min., then to 260°C @ 35°C/min.
Inj./ det. temp.: 180°C/260°C
FID sensitivity: 1.28 x 10^{-10}AFS
Carrier gas: helium
Linear velocity: 35cm/sec. @ 35°C
Split ratio: 10:1

Figure 2 - USP 467 target compounds are better resolved on an Rtx™-1301.



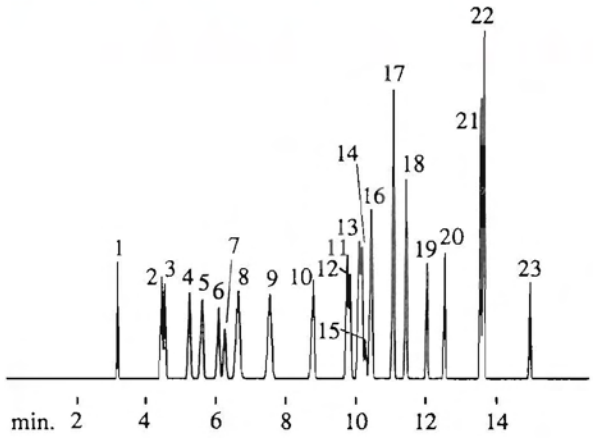
30m, 0.53mm ID, 3.0µm, Rtx™-1301 (cat.# 16085)
1.0µl split injection of USP 467 Calibration Mix
Oven temp.: 40°C (hold 20 min.) to 240°C @ 35°C/min. (hold 10 min.)
Inj./ det. temp.: 180°C/260°C
FID sensitivity: 1.28 x 10^{-10}AFS
Carrier gas: helium
Linear velocity: 35cm/sec. @ 40°C
Split ratio: 10:1

Figure 3 - Solvents commonly used in pharmaceutical processing run on an Rtx™-5.



30m, 0.53mm ID, 5.0µm, Rtx™-5 (cat.# 10279)
0.5µl split injection of residual solvents from pharmaceutical processing
Oven temp.: 35°C (hold 7 min.) to 240°C @ 15°C/min
Inj./ det. temp.: 180°C/260°C
FID sensitivity: 1.28 x 10^{-10}AFS
Carrier gas: helium
Linear velocity: 25cm/sec. @ 35°C
Split ratio: 30:1

Figure 4 - Better resolution is observed on an Rtx™-1301 for solvents commonly uses in pharmaceutical processing.



30m, 0.53mm ID, 3.0µm, Rtx™-1301 (cat.# 16085)
0.5µl split injection of residual solvents from pharmaceutical processing
Oven temp.: 35°C (hold 8 min.) to 240°C @ 20°C/min.
Inj./ det. temp.: 180°C/260°C
FID sensitivity: 1.28 x 10^{-10} AFS
Carrier gas: helium
Linear velocity: 25cm/sec. @ 35°C
Split ratio: 30:1

Peak List for Figures 3 and 4.

COMPOUNDS					
1	methanol	9	hexane	17	benzene
2	ethanol	10	n-propanol	18	heptane
3	diethyl ether	11	methyl ethyl ketone	19	trichloroethylene
4	acetone	12	ethyl acetate	20	1,4-dioxane
5	isopropanol	13	tetrahydrofuran	21	pyridine
6	acetonitrile	14	sec-butanol	22	toluene
7	methylene chloride	15	chloroform	23	dimethylformamide
8	tert-butanol	16	cylcohexane		

Product Listing

30m, 0.53mm ID, 5.0µm **Rtx™-5**	cat.# 10279, $455
30m, 0.53mm ID, 3.0µm **Rtx™-1301**	cat.# 16085, $445
USP 467 Calibration Mix	cat.# 36000, $20 each cat.# 36100, $180/10pk.
5m, 0.53mm ID Phenyl Methyl Deactivated Guard Columns	cat.# 10045, $60 each cat.# 10045-600, $300/6pk.

Standards Spotlight

Additional Standard for Underground Storage Tank Monitoring

• Latest protocol revisions • Important product changes • Full data packs available •

The State of Wisconsin, Department of Natural Resources has modified the composition of the Gasoline Range Organics (GRO) standard enabling laboratories to calibrate for both the Petroleum Volatile Organic Compound (PVOC) and the GRO simultaneously (1). This new standard may be used by laboratories to increase lab efficiency and decrease overall cost by streamlining the analytical method calibration sequence.

Restek's commitment has been to provide quality standards for all method modifications as quickly and cost effectively as possible. In addition, unlike other companies, full data pack documentation is available for every environmental standard we produce.

Wisconsin PVOC/GRO Mix

1000µg/ml each in 1ml purge & trap grade methanol.

methyl-t-butyl ether	benzene
toluene	ethylbenzene
o-xylene	m-xylene
naphthalene	1,2,4-trimethylbenzene
1,3,5-trimethylbenzene	

Cat.# 30085 $25 ea.
30085-500 $55 ea. w/data pack
30185 $225 10pk. w/data pack

1) LUST RELEASE, Vol. 2, No. 3, April 1992, Wisconsin Department of Natural Resources, Leaking Underground Storage Tank Program, P.O. Box 7921, Madison, WI 53707, Phone: (608)266-2172.

LUST Retention Time Standard Changes

In *The Restek Advantage*, Vol. 3 No. 6, November 1992, we introduced a product called the LUST Retention Time Standard. This mixture initially contained the normal paraffins C5, C6, C10, C12, C24, C28, C30, and C40 in methylene chloride at 2500µg/ml each (cat.#'s 31099, 31099-500, and 31199). After further method review and discussion with several analytical laboratories, it became evident that this mixture had several problems associated with its use.

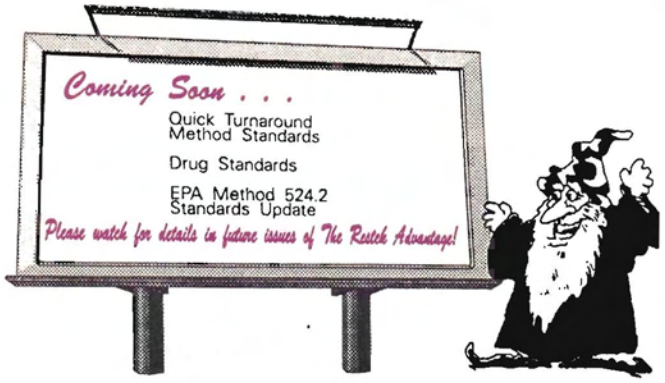
To circumvent these analytical problems, we are discontinuing this mixture (cat.#'s 31099, 31099-500, 31199) and replacing it with a new mixture. This new mixture is designed to determine the retention time windows for elution of gasoline, diesel, and other heavy oils during initial sample screening.

LUST Retention Time Standard

25µg/ml each in 1ml methylene chloride.

hexane (C6)
decane (C10)
dodecane (C12)
tetracosane (C24)
octacosane (C28)
triacontane (C30)
tetracontane (C40)

Cat.# 31200 $28 ea.
31200-500 $58 ea. w/data pack
31300 $252 10pk. w/data pack



Improved Surrogate Mixture for SW-846 Method 8021

We have carefully reviewed EPA Method 8021 and found several problems with the specified standards; in particular, the surrogate mixture. As written, the internal standard mixture specified is to contain fluorobenzene and 2-bromo-1-chloropropane. The surrogate mixture specified contains bromochloromethane, 2-bromo-1-chloropropane, 1,4-dichlorobutane, and bromochlorobenzene.

During our initial review, we inadvertently missed the duplication of 2-bromo-1-chloropropane in both mixtures, and the inclusion of bromochloromethane (which is also a target analyte). Also, the EPA had failed to specify which bromochlorobenzene (2-, 3-, or 4-) should be used. Our testing has determined that the 2-bromochlorobenzene should be used since it can be easily resolved from other analytes. As a result, the 8021 Volatile Organics Kit (cat.#'s 30072 and 30072-500) is being discontinued. We are now offering a replacement 8021 Volatile Organics Kit which includes the new 8021 Surrogate Mix.

8021 Surrogate Mix

1500µg/ml each in 1ml purge & trap grade methanol.

1,4-dichlorobutane 2-bromochlorobenzene

Cat.#	**30086**	**$25 ea.**
	30086-500	**$35 ea. w/data pack**
	30186	**$225 10pk. w/data pack**

8021 Volatile Organics Kit

Contains one ampul (1ml) each of the following mixtures:

30041	502.2 Internal Standards Mix (IS)
30042	502.2 Calibration Mix #1
30043	502.2 Calibration Mix #2
30044	502.2 Calibration Mix #3
30045	502.2 Calibration Mix #4
30046	502.2 Calibration Mix #5
30047	502.2 Calibration Mix #6
30086	8021 Surrogate Mix

Cat.#	**30087**	**$210 ea.**
	30087-500	**$360 ea. w/data pack**

Attention VOA Lab Analysts . . .

We're Working Harder to Make Your Job Easier

Restek has acquired pure individual cis- and trans-1,3-dichloropropene. All mixtures made after July 1992 that contain these two compounds will be exactly the concentration specified in our literature. Analysts using these mixtures won't have to change computer calibration files to adjust concentration for these compounds with each lot of material purchased. Verify the exact mixture composition on the certificate of analysis supplied with each ampul.

Restek mixtures that contain cis- and trans-1,3-dichloropropene:

VOA Calibration Mix #4 (cat.#'s 30009, 30009-500, 30109)
502.2 Calibration Mix #2 (cat.#'s 30043, 30043-500, 30143)
624 Calibration Mix #3 (cat.#'s 30022, 30022-500, 30122)
8010A Calibration Mix #2 (cat.#'s 30056, 30056-500, 30156)

Clinical Corner (continued from page 8)

In addition to giving superior resolution for the organic volatile impurities listed in USP 467, the Rtx™-1301 column also shows improved performance for analyzing other solvents as well. Figures 3 and 4 show the analysis of a set of commonly used solvents in pharmaceutical processing. The Rtx™-1301 is able to at least partially resolve all of the solvents in the mixture while the Rtx™-5 has three complete coelutions.

In a review[5] of the current status of USP 467, the USP is advocating the conversion of monograph requirements from Method I to Method V to allow for the use of the cyanopropyl stationary phase for better resolution. They are also anticipating that more laboratories will begin to use the static headspace technique as a means of replacing the direct injection of aqueous samples and its associated problems. These changes, along with the current revisions to USP 467, should result in an easier to use and more reproducible method for the future. ■

References

1. Bergren, M.S., Foust, D.W., Comments on USP General Chapter, Organic Volatile Impurities <467>, and Associated Monograph Proposals. *Pharmacopeial Forum*, May/June 1991, Volume 17 Number 3, pp. 1963-1968.

2. Krasowski, J.A., Dinh, H., O'Hanlon, T.J., Lindauer, R.F., Comments on Organic Volatile Impurities, Method I, <467>. *Pharmacopeial Forum*, May/June 1991, Volume 17 Number 3, pp. 1969-1972.

3. *Pharmacopeial Forum*, March/April 1991, Volume 17 Number 2, p. 1653.

4. *Pharmacopeial Forum*, September/October 1992, Volume 18 Number 5, p.4028.

5. Gray, V., Organic Volatile Impurities Testing Initiative: An Update. *Pharmacopeial Forum*, March/April 1992, Volume 18 Number 2, p. 3205.

Hints for the Capillary Chromatographer

Solvent Loss from Snap-Cap Autosampler Vials

What are snap-cap autosampler vials?

Snap-cap vials and closures are being marketed as an alternative to crimp type aluminum seals used with autosampler vials. The plastic seals are typically made of polypropylene or polyethylene. Snap-caps can be pushed on or pried off by hand, without the use of tools like crimpers and decappers. The snap-cap stays on by either gripping under the lip of a standard autosampler vial, or the snap-cap may have teeth that pop into a special slot-lipped autosampler vial.

Determining the suitability for storing organic solvents

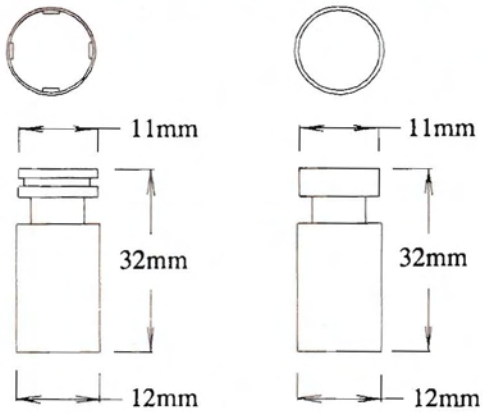
Three brands of 12 x 32mm vials with snap-caps were evaluated against standard vials with aluminum crimp-tops, to determine if the snap-caps could prevent solvent loss at room temperature. The products evaluated were: Brand X and Brand Y snap-caps using standard autosampler vials, and Brand Z snap-caps using special slot-lipped autosampler vials. The reference, used for comparison, were aluminum crimp-top vials equipped with Teflon/red rubber septum.

A slot-lipped autosampler vial and a bottom view of its snap-cap are shown on the left side of Figure 1. This is the design of the Brand Z snap-cap vials. Its snap-cap is held down by its four "teeth" that pop into the vial's slotted lip. A standard autosampler vial and a bottom view of its toothless snap-cap are shown on the right side of Figure 1. This is the design of the Brand X and Y snap-caps. This snap-cap has a ridge around the bottom inside of the cap that grips under the lip of a standard vial.

The two solvents used in this study were methylene chloride (40°C boiling point) and methanol (65°C boiling point). These are two of the most common solvents used in gas chromatography. One milliliter of solvent was volumetrically pipetted into each vial to ensure equal headspace. The vials were then carefully capped and the absolute weight of the vial, solvent, and cap was then determined to the nearest tenth of a milligram. The samples were then stored at room temperature (22 +/- 2°C) and reweighed at selected time intervals over a period of 6 days.

The test results are shown in Figures 2 and 3. The reference crimp-top vials showed the least solvent loss. The Brand X vials were the best performing snap-cap vials and their solvent loss was slightly greater than the crimp-top vials. Figure 2 shows the results for the crimp-top vials compared to Brand X snap-cap vials using a magnified scale. While the solvent loss with Brand X snap-cap vials reached approximately 0.7% with

Figure 1 - Design of Snap-Cap Autosampler Vials



Note: While the vials alone are identical in their overall dimensions, the assembled snap-cap autosampler vials may differ significantly from standard crimp-top vials. The snap-cap vials may be taller, wider, or have shorter necks than assembled crimp-top vials.

methylene chloride over a 135 hour period, this may not be significant enought to be of concern.

Figure 3 shows results for crimp-top vials compared to Brand Y and Z plotted on a larger scale. The scale had to be significantly expanded to show the extreme loss of solvent with these two types of snap-cap vials. Brand Y and Z closures performed so poorly that by comparison, the crimp-top vials show no apparent loss of solvent.

As shown in both figures, the more volatile the solvent being stored in the snap-cap vial, the greater the loss of solvent over a period of time. The snap-cap vials tested did not hold the septum as firmly in place against the top of the vial as the crimp-top vials did. This may be the most significant factor for the solvent losses measured. There is some speculation that chlorinated solvents may also cause swelling of the plastic caps, further aggravating the problem. This idea is currently being evaluated.

How much does solvent loss effect sample concentration?

The loss of solvent with Brand Y and Z closures would cause a significant increase in the concentration of non-volatile analytes stored in a volatile solvent such as methylene chloride. Since all calibration standards are prepared on a weight per volume basis, any loss of volume will cause an increase in effective concentration of a non-volatile analyte.

Figure 2 - Solvent loss over time from Brand X snap-cap vials compared to crimp-top vials

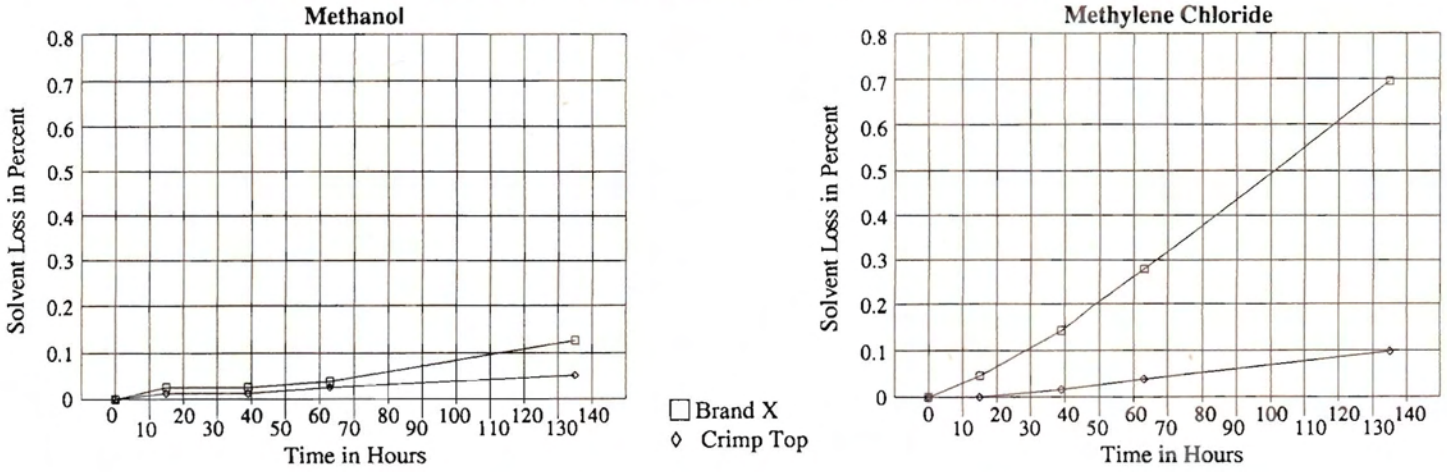
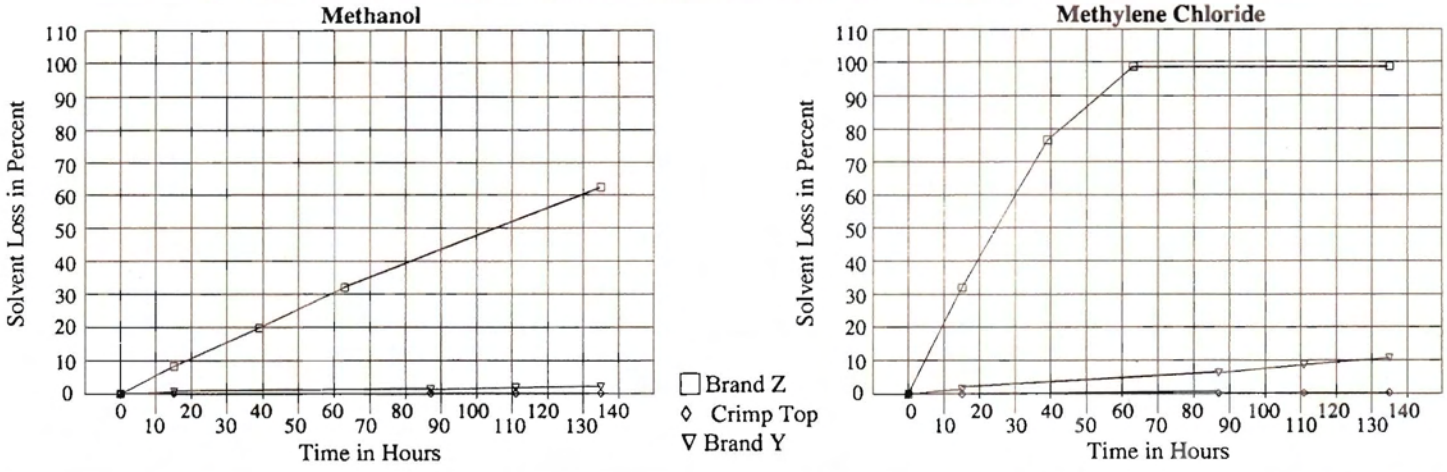


Figure 3 - Solvent loss over time from Brands Y and Z snap-cap vials compared to crimp-top vials.



This increase can be calculated if the amount of solvent loss is known, as shown in this example. Brand Z snap-cap vials containing methylene chloride lost 32% during a 15 hour time period. If the initial concentration of the non-volatile analyte was 100µg/ml, the loss of 32% of the solvent in a 1ml sample would increase the concentration to 147µg/ml.

$$\frac{\text{weight of the analyte in vial}}{\text{(1ml - volume lost)}} = \frac{100\mu g}{(1ml - 0.32ml)} = \frac{100\mu g}{0.68ml} = 147\mu/ml$$

If volatile analytes dissolved in a less volatile solvent are stored in a snap-cap system, the concentration change would be greatly dependant upon the partial vapor pressure and solubility partition coefficient of each material. The change in concentration would have to be determined experimentally with each compound of interest being tested.

What to check before using snap-cap vials

Thoroughly testing a snap-cap autosampler vial system before you use it with your valuable samples can save you lost time and money. First, a test to determine solvent or volatile analyte loss to confirm the quality of the closure. Next, a test should be conducted to determine if the snap-cap vials will feed reliably through your autosampler without jamming. This is especially important with snap-cap vials since the overall assembled dimensions of a snap-cap vial may vary compared to crimp-top vials. The final test should be to evaluate an entire sampling sequence to insure trouble-free unattended operation. Taking some time to fully evaluate a snap-cap vial can save you the loss of your valuable samples.

How to get the best performance out of crimp-top vials

A few simple steps is all it takes to protect your valuable samples in a crimp-top autosampler vial. Fill the vial to the very top if possible. Limiting the vial headspace is essential with volatile gases dissolved in solution. Always crimp the cap tight enough that the cap does not move freely around the vial lip. This ensures that the septum is being held firmly against the top of the vial. Store samples at reduced temperatures in deactivated vials with an unpierced, Teflon® faced septum. This will reduce sample evaporation and prevent analyte degradation and adsorption.

While snap-cap autosampler vials offer ease of use over crimp-top vials, they may not be suitable for volatile solvents or analytes. Snap-cap vials should be thoroughly tested against crimp-top vials with representative samples under the same analysis conditions to ensure that they work properly. ■

Peak Performers

Visually Confirm Proper Column Installation with Vu-Tight™ Direct Injection Sleeves

- Visually confirm connection between the column end and sleeve
- Fits HP, Varian, and most 1/4-inch packed column injection ports with maximum insertion depths of 4 inches
- Converts packed column inlets for use with 0.32 and 0.53mm ID fused silica capillary columns (tubing OD must be 0.4mm or greater)
- Deactivated, strong, and extremely inert
- Slotted top prevents obstruction of carrier gas flow
- Low cost

Convert packed column inlets for use with 0.32 and 0.53mm ID high resolution capillary columns using the new Vu-Tight™ Direct Injection Sleeve. To install the Vu-Tight™ sleeve, simply insert the 1/4-inch inlet sleeve into the packed column injection port and tighten it with a 1/4-inch nut and ferrule. Then connect the column to the outlet of the sleeve with a 1/4 to 1/16-inch stainless steel reducing fitting. The press-tight taper is positioned for easy observation of a proper seal between the column end and the direct injection sleeve.

Vu-Tight™ sleeve designs are also available for use with dirty samples. A Cyclo Vu-Tight™ or a Vu-Tight™ packed with wool or beads effectively traps non-volatile sample residue and can prevent column contamination.

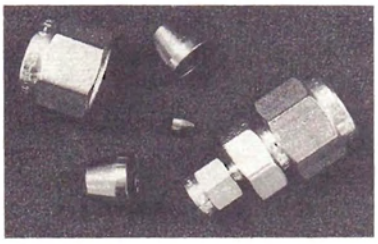
Vu-Tight™ Direct Injection Sleeves (1/4″ OD)

cat.# 20342, $28 each
cat.# 20343, $115/5-pk.
cat.# 20344, $480/25-pk.

Cyclo Vu-Tight™ Direct Injection Sleeves

cat.# 20787, $55 each
cat.# 20788, $220/5-pk.

Vu-Tight™ Installation Fittings



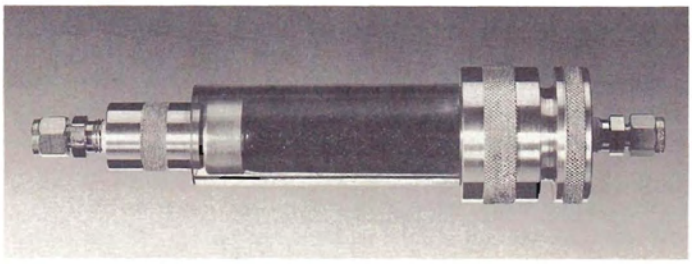
Includes a 1/4-inch SS nut and 1/4-inch graphite ferrule for attaching the sleeve to the GC inlet and a 1/4 to 1/16-inch SS reducing nut plus a 1/4-inch and 0.5mm ID graphite ferrule for attaching the column to the Vu-Tight™ direct injection sleeve.

cat.# 20504, $30 each

High Capacity Indicating Oxygen Trap

- Indicator changes color as O_2 & H_2O are trapped.
- Heavy duty body design virtually eliminates breakage.
- High capacity (>300 s.c.f.) design lasts longer than three smaller traps.
- Economic replacement cartridges change easily.
- Usable with multiple GC systems.
- Removes impurities at flow rates up to 230 s.c.f./hour.
- Removes O_2, H_2O, and trace contaminants.
- Usable with all carrier gases.
- Ambient operating temperature, 100psig operating pressure.
- Built-in microparticulate frit.
- Discharge Gas Purity:
  O_2 < 0.1 ppm where inlet does not exceed 15 ppm
  H_2O < 0.5 ppm where inlet does not exceed 10 ppm

Includes cartridge housing and one cartridge.



1/4″ tube compression fittings: cat.# 20623, $170 each
1/8″ tube compression fittings: cat.# 20624, $170 each
Refill cartridge (fits 1/4 or 1/8″): cat.# 20625, $55 each

Now Available - Untreated Autosampler Vials

Screw-top or crimp-top styles • Clear or amber borosilicate glass
Buy pieces separately or as kits • 100 or 1000 packs available

We now offer untreated vials for most GC autosamplers. These high quality vials are available in either screw-top or crimp-top styles. We offer vials made from both clear or amber borosilicate glass. Both designs offer a Teflon® lined septa in the closure to prevent sample contamination during storage. They can be purchased as complete kits including closures or as vials or closures only. Available in standard 100 packs or economical 1000 packs, these vials are guaranteed to work perfectly with your autosampler.

Screw-top vials	100 Pack		1000 Pack	
Clear, complete kit	21192	$26	21193	$225
Amber, complete kit	21194	$28	21195	$245
Clear, vial only	21184	$15	21185	$125
Amber, vial only	21186	$16	21187	$140
8-425 hole cap	21176	$6	21177	$50
8mm PTFE/silicone septa	21178	$12	21179	$85

Crimp-top vials	100 Pack		1000 Pack	
Clear, complete kit	21196	$20	21197	$175
Amber, complete kit	21198	$20	21199	$175
Clear, vial only	21188	$15	21189	$125
Amber, vial only	21190	$16	21191	$140
11mm aluminum seals w/PTFE-Rubber septa	21174	$9	21175	$75

Use the Screw-top vials for these autosamplers:
- Varian (all models)
- Shimadzu AOC-14
- Hewlett-Packard (7670A, 7671A)
- Perkin-Elmer ISS-100

Use the Crimp-top vials for these autosamplers:
- Antek
- Hewlett-Packard (all models)
- Perkin-Elmer (models: LC-600, 420, 600, 4900, & ISS-100)
- Spectra Physics (7110, 8110)
- Tracor 770
- Dynatech Precision Sampling

new! Deactivated Inlet Sleeves for Fisons/Carlo Erba GCs

- Fits new 8000 series GC
- Fully deactivated for exceptional inertness
- Available for split and splitless injection ports in 3 and 5mm IDs

Restek now offers deactivated inlet sleeves for the Fisons/Carlo Erba 8000 series GC. Both 3 and 5mm ID split and splitless designs are available. Sold individually or in economical 5 and 25 packs, these sleeves are guaranteed to meet the original equipment specifications. (OD 8mm, length 105mm)

Let Restek prepack your sleeves with deactivated fused silica wool or beads. To order prepacked sleeves, simply add the correct suffix to any sleeve catalog number. For sleeves prepacked with wool, add the suffix "200.1" per single sleeve, "200.5" for 5-packs, and "200.25" for 25-packs. For sleeves prepacked with beads, add suffix "201.1" per single sleeve, "201.5" for 5-packs, and "201.25" for 25-packs. When ordering prepacked sleeves, add $10 to the price per single sleeve, $25 for 5-packs, and $75 for 25-packs.

3mm Split
cat.# 20936, $20 each
cat.# 20937, $80/5-pk.
cat.# 20938, $370/25-pk.

3mm Splitless
cat.# 20942, $29 each
cat.# 20943, $115/5-pk.
cat.# 20944, $465/25-pk.

5mm Split
cat.# 20939, $18 each
cat.# 20940, $75/5-pk.
cat.# 20941, $345/25-pk.

5mm Splitless
cat.# 20945, $25 each
cat.# 20946, $100/5-pk.
cat.# 20947, $400/25-pk.

News from Restek

Good News for our Neighbors to the North

Restek is very pleased to announce that, effective immediately, our products will be available in Canada through Chromatographic Specialities, in Brockville, Ontario. They can be reached at (612) 342-4678. We have also expanded our toll-free number for technical support into Canada. Restek's technical support number for the U.S. and Canada is 1-800-356-1688.

Restek Acquires Amchro, Expands to Germany

To improve service to our European customers and expand our distribution network in Europe, Restek Corporation has acquired Amchro in Frankfurt, Germany.

Amchro is an established chromatography products distributor that has successfully distributed Restek products for 5 years. Under its new name, Restek Europa, Amchro will continue selling chromatography products to increase Restek's market share in Europe.

Dr. Johann Vasgyura, Amchro's president, has been appointed as managing director of the new Restek Europa.

We're Sorry,
No More Multipliers

Due to circumstances beyond our control, we no longer offer electron multipliers for Hewlett-Packard 5971 MSD systems. With very limited notification, our supplier has chosen to offer these multipliers only through HP. We regret any inconvenience this may have caused our customers.

No More Saturday Service

For almost a full year, Restek has offered our customers the convenience of Saturday ordering, shipping, and technical support. Because response to these services has been very minimal, effective January 23 we will no longer offer Saturday hours. You can still place orders Monday through Friday from 8am to 7pm Eastern Standard Time (EST). Technical Support will be available Monday through Thursday from 8am to 8pm EST and Friday from 8am to 5pm EST.

Look Out! The Wizard Dollars are Coming

Starting January 4, for every $50 you spend on Restek products you will receive one Wizard Dollar. These Wizard Dollars can be redeemed for valuable gifts ranging from coffee mugs to color televisions. The more products you buy, the more dollars you accumulate, to get bigger and bigger prizes. Look for details about the Wizard Dollar promotion in our new 1993-1994 catalog.

Upcoming Restek Exhibits:

American Academy of Forensic Science Meeting
February 15-19 in Boston, MA (Booth #235)

Paper Presentation: "Evaluation of an Open Tubular Solid Phase Extraction System for Drugs of Abuse".

The 1993 Pittsburgh Conference
March 8-11 in Atlanta, GA (Booth #'s 2009 and 4542).

See back cover for a complete list of our Pittsburgh Conference technical presentations and a coupon for a free gift.

Phone: (814)353-1300
FAX: (814)353-1309
Orders: (800)356-1688

THE RESTEK ADVANTAGE

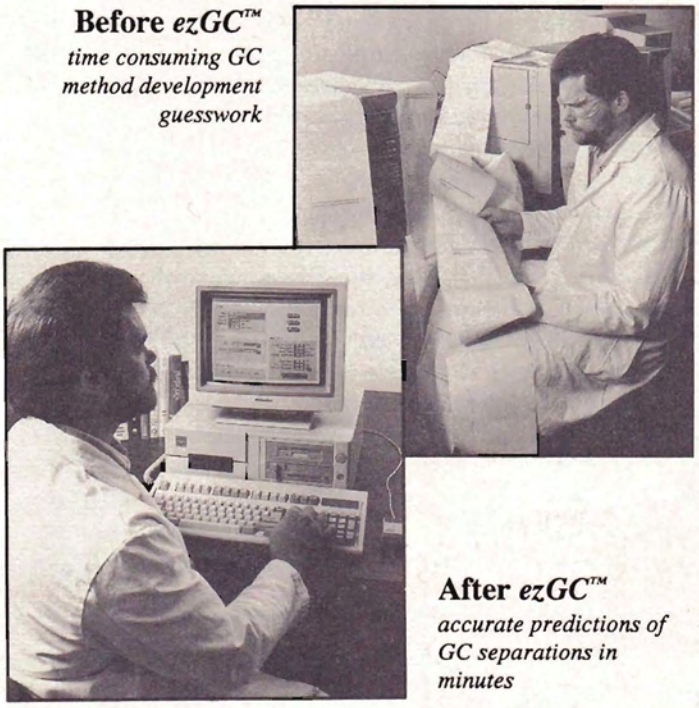
Sneak Preview! *ezGC™* Software

Simplifies GC Method Development

- Saves time and money by reducing analysis times and improving sample resolution.
- Automatically determines optimum temperature program rates and column flow rates.
- Works with constant flow, constant pressure, or electronic pressure/ flow programming.
- Visually demonstrates changes in resolution when the column parameters and operating conditions are changed.
- Easy to use, mouse driven software with built in help menus.
- Takes the guesswork out of capillary column selection.
- Easy to install and works on all DOS operating systems with 512K of free RAM.
- Costs about the same as a 30-meter column.

Did you ever work with a chromatographer who seems to know how to pick the best temperature program and flow conditions? After years and years of experience they seem to inherently know which GC parameters work best. They have learned how parameters such as temperature, flow, and distribution coefficients affect a separation. Why wait years? Use *ezGC™* and quickly become a master at capillary column selection and optimization.

Before *ezGC™*
time consuming GC method development guesswork



After *ezGC™*
accurate predictions of GC separations in minutes

in this issue...

Even experienced chromatographers will benefit by using *ezGC™*. Restek's applications department was hard at work trying to optimize the temperature program rate for the 60 compounds in EPA Method 502.2. They tried 4, 10, 12, and 16°C/min., but there were so many compounds that new coelutions occurred at each temperature program ramp. The separations were so complex that they couldn't figure out whether faster or slower program rates were better. After several frustrating days of working on the project, they tried *ezGC™*. They entered the retention times into the *ezGC™* program and let the software do the optimization. *ezGC™* predicted 7.5°C/min. as the optimum temperature program rate and printed a simulated chromatogram illustrating the expected separations. They were impressed but still not convinced. Actual chromatograms were then generated at 7 and 8°C/min., but only 7.5°C gave the best separation, just as the program predicted. Now our applications department is so convinced of the power of *ezGC™* that they use it for all optimization work.

You can save time and money in your laboratory by using *ezGC™* to optimize all your analyses. If you have a simple analysis with no coelutions, you can use the software to predict the fastest temperature program and flow conditions while

maintaining baseline resolution (R≥1.5). And, if your sample contains compounds which may switch elution orders at the new optimized conditions, *ezGC™* will list the new elution order.

Did you ever wonder how your sample would look on a different film thickness? If you are using a 0.25µm film and you suspect that a 0.5µm film would improve resolution, use *ezGC™* to print a simulated chromatogram with the 0.5µm film. In fact, you can try any other film thickness and *ezGC™* will provide simulated chromatograms at optimized run conditions. How about a longer length or different inside diameter? Enter the desired column dimensions into the *ezGC™* program and it will provide a simulated chromatogram for visual examination. Now you don't have to waste your time or money buying experimental columns to optimize your analysis, *ezGC™* can do it for you.

How does *ezGC™* work?

In the past 20 years, several attempts have been made to predict retention and elution in gas chromatography. Initially, elution order was predicted by Kováts indices (1). However, Kováts indices are restricted to isothermal conditions. With the increasing use of temperature programming, Kováts indices were not applicable in many situations. A modified retention index equation was developed by Van den Dool and Kratz[2] that incorporated Kováts indices into temperature programming. This modified retention index works relatively well, as was demonstrated in *The Restek Advantage* (January 1992). However, neither the Kováts or Van den Dool and Kratz methods account for changes in carrier gas viscosity, linear velocity, film thickness, etc. Recently, advances have been made in developing a more sophisticated method to predict GC behavior. Several researchers, Dose[3]; Curvers and Rijks[4]; and Snow and McNair[5] have contributed to a method for calculating temperature programmed or isothermal retention from thermodynamic parameters. The distribution coefficient K_D is related to the Gibbs free energy of gases in solution by the following equation:

$$\Delta G = RT \ln K_D \text{ and since } \Delta G = \Delta H - T\Delta S$$

substituting $K_D = k * \beta$, the following equation can be derived:

$$\ln k = \left(\frac{\Delta H}{R}\right) * \left(\frac{1}{T}\right) + \ln\left(\frac{a}{\beta}\right)$$

where

$$a = \left(\frac{\Delta S}{R}\right)$$

This new equation is in the form of $y = mx + b$ where $\frac{\Delta H}{R}$ is the slope of the line and the quantity $\ln(a/\beta)$ is the y intercept. The *ezGC™* software incorporates these fundamental concepts into a computer algorithm that makes it possible to accurately predict GC retention times routinely to within 2%.

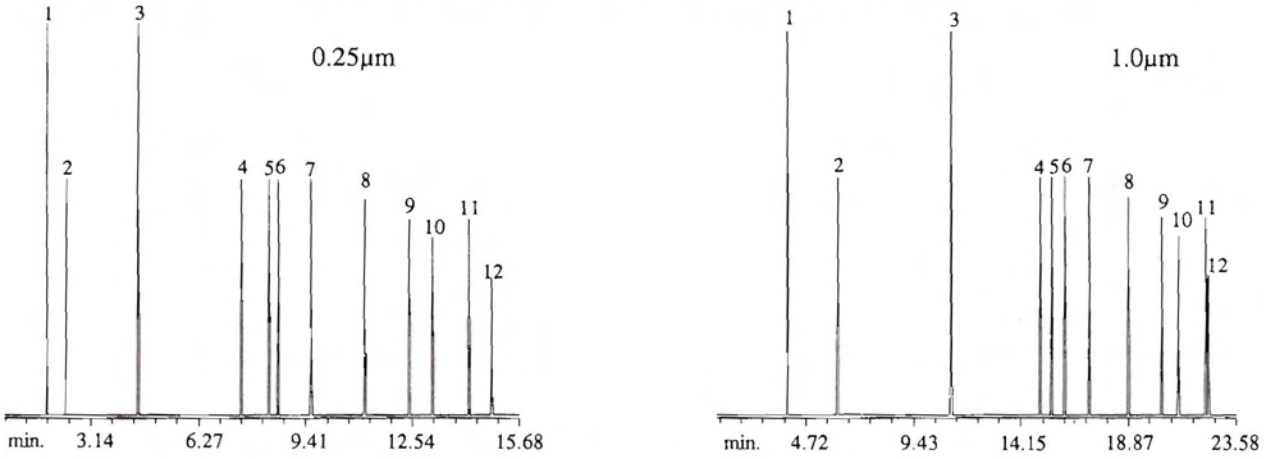
How hard is it to use *ezGC™*?

By following a few simple steps, optimum operating conditions can easily be predicted for any analysis. To utilize *ezGC™*, simply obtain an accurate dead time and run your sample at fast and slow temperature program ramps. Enter the retention times for both runs in the program and you are ready to try new temperature program rates, flow rates, column IDs, film thicknesses, or column lengths. An on-line help manual is available at any time to answer questions, and in those rare cases when you need extra help, experienced Restek technical service chemists will be available to assist you with your more detailed questions.

Ways to generate optimum conditions

Optimum temperature programmed run conditions can be generated two ways. In one case, a specific set of GC conditions is entered and under those conditions, the *ezGC™* program will predict the retention times of the components.

Figure 1 - *ezGC™* quickly predicts actual peak resolution when increasing the film thickness from 0.25 to 1.0µm when using the same temperature program.



30m, 0.25mm ID Rtx®-5
Oven temp.: 35°C (hold 5 min.) @4°C/min. to 280°C **Linear velocity:** 43.0cm/sec. @ 35°C

Table I - Comparison of Experimental vs. Calculated Retention Times

#	Component Name	Exp. tR (min.)	Calc. tR (min.)	Exp. Calc. Error (min.)	(Exp.-Calc.) /Exp. % Error (min.)
1	hexane	3.891	3.900	-0.009	-0.2
2	benzene	6.032	6.117	-0.085	-1.4
3	toluene	11.001	11.076	-0.075	-0.7
4	chlorobenzene	15.002	14.991	0.011	0.1
5	ethylbenzene	15.500	15.495	0.005	0.0
6	*m*-xylene	16.184	16.059	0.125	0.8
7	styrene	17.395	17.129	0.266	1.5
8	isopropylbenzene	19.082	18.861	0.221	1.2
9	n-propylbenzene	20.517	20.345	0.172	0.8
10	1,3,5-trimethylbenzene	21.202	21.071	0.131	0.6
11	tert-butylbenzene	22.385	22.259	0.126	0.6
12	decane	22.501	22.364	0.137	0.6
				Average error	0.7

Predicted results can be viewed in either a table format or a computer simulated chromatogram. Figure 1 shows simulated chromatograms demonstrating how the analysis would look if the stationary phase film thickness was increased from 0.25 to 1.0µm with the same program conditions. The 30m, 1.0µm film thickness increases the analysis times from approximately 14 to 22 minutes. Figure 2 shows the predicted optimum temperature program ramp for the 5m, 1.0µm column to maximize resolution and minimize analysis times. Baseline resolution is obtained in under 6 minutes with the 5m column.

Another way to generate the optimum conditions is by entering a range of desirable temperature program conditions into the program. The optimum conditions, yielding the shortest analysis time with the best resolution, will be listed first with other possibilities listed sequentially. Computing time varies with the number of permutations requested.*

Quickly compare differences in analysis and resolution changes when varying linear velocity, ID, film thickness, length, or theoretical plates

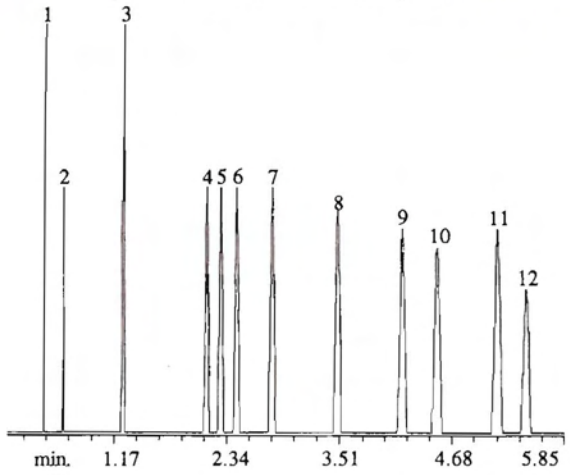
ezGC™ permits a visual comparison of analysis times and resolution when column parameters such as linear velocity (including electronic pressure or flow programming), column diameter, theoretical plates, film thickness, and/or the column length are varied. Table I shows the predicted vs. actual retention times for a 1.0µm Rtx®-5 using data generated on a 0.25µm capillary column. The absolute error is approximately 2%.

ezGC™ simplifies method development

ezGC™ greatly reduces the workload of GC method development. It also insures the best resolution and analysis time conditions for existing methods. This versatile program allows any parameter or combination of parameters to be changed and quickly viewed in either a table format or simulated chromatogram. *ezGC™* can be installed on any IBM PC or compatible system with a hard drive and 512K of free memory.

** A 386SX-25 without a coprocessor was able to evaluate 350 temperature programs for 12 components in under 1 minute.*

Figure 2 - *ezGC™* predicts the optimum resolution and fastest analysis times with a 5.0m, 1.0µm column.

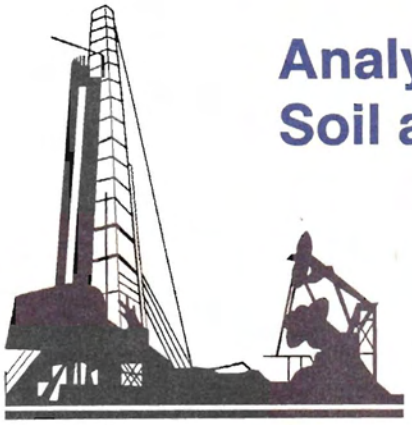


5.0m, 0.25mm ID, 1.0µm Rtx®-5
Oven temp.: 50°C @4°C/min. to 80°C
Linear velocity: 40.9cm/sec. @ 60°C

After reading about *ezGC™*, you may ask, "How could method development be easier?" The answer is, by having Restek generate thousands of thermodynamic retention index libraries on volatile organics, industrial solvents, pharmaceutical compounds, and flavors/fragrances using a wide variety of bonded phases. Restek has dedicated a large portion of our application chemists' time towards generating extensive libraries that interface to *ezGC™*. See the July 1993 issue of *The Restek Advantage* for information on Restek's thermodynamic retention indice libraries. ■

References

(1) Kováts, E., Giddings, J.C., and Keller, R.A., *Advances in Chromatography*, Volume 1, Chapter 7. New York: Marcel Dekker (1965).
(2) Van den Dool, H. and Kratz, P.D., *Journal of Chromatography*, Volume 11, pp.463-471, (1963).
(3) Dose, E.V., *Anal. Chem.*, 1987, 59, 2414-2419.
(4) Curvers, J., Rijks, J., Cramers, C., Knauss, K., Larson, P., *HRC & CC*, Vol. 8, Sept. 1985.
(5) Snow, N.H. and McNair, H.M., *J. of Chrom. Sci.*, Vol. 30, July 1992.



Analysis of Gasoline Range Organics in Soil and Water

Individual states have adopted analytical methods for measuring hydrocarbon contamination in soil and water (1) resulting from leaking underground storage tanks (LUST). The following article addresses some of the more common questions regarding the determination of benzene, toluene, ethylbenzene and total xylenes (BTEX), and total petroleum hydrocarbons (TPH) from gasoline range organics (GRO). Future articles will address the analysis of diesel range organics (DRO) and heavy petroleum products such as lubrication oil.

In general, GRO methods for analysis of TPH and BTEX use a purge and trap sampler, a wide bore capillary column, and both photo ionization (PID) and flame ionization (FID) detectors. The purge and trap sampler (2) is used to extract and concentrate the more volatile gasoline components from water and soil (methanol extract) prior to introduction into the gas chromatograph. The sampling procedure for water is as specified in EPA Method 602 (3). For soils, a methanol extract is added to the purge vessel containing a volume of water as specified in EPA Method 8020 (4). The FID responds to all hydrocarbon species in the complex gasoline sample and is used to detect the total volatile hydrocarbons. A PID, when operated with a 10.0 eV lamp, yields more specific response to aromatic and other unsaturated hydrocarbons present in gasoline and is used to quantitate BTEX. A wide range of columns can be used for GRO analysis, depending upon the requirements specified in each state's analytical procedure. In general, the column, operating under the conditions of the method, must meet some minimum requirements for retention and resolution.

Gasoline is a complex mixture, containing in excess of 400 individual hydrocarbon compounds; so if BTEX is to be determined, the column must resolve these aromatics. Since xylenes are reported as a total, it is not necessary to separate the ortho, meta and para isomers. The resolution between ethyl benzene and *m*-, *p*-xylene is typically the most difficult separation to obtain. Since the same chromatographic method is normally used for both water and soils, the column must also resolve gasoline from the methanol solvent peak. States may differ on which hydrocarbon is used to define the beginning and end of the gasoline compounds to be measured, so the requirements of the column will vary. The 105-meter Rtx®-502.2 column is a good choice for most methods because it resolves 3-methyl pentane from methanol without subambient oven temperatures and provides baseline resolution of ethyl benzene from *m*-, *p*-xylene.

Determining gasoline retention range and calibrating response

Hydrocarbon calibration standards serve two purposes in TPH/BTEX analysis. Since the reporting of TPH requires the summation of the total gasoline area, the standard must contain the first and last components defining the retention time range. Individual states differ on the compounds defining the retention time range for gasoline. Figure 1 shows a chromatogram

Figure 1 - The GRO Mix can be used to establish the start/stop times of the gasoline range and to calibrate FID/PID detector response.

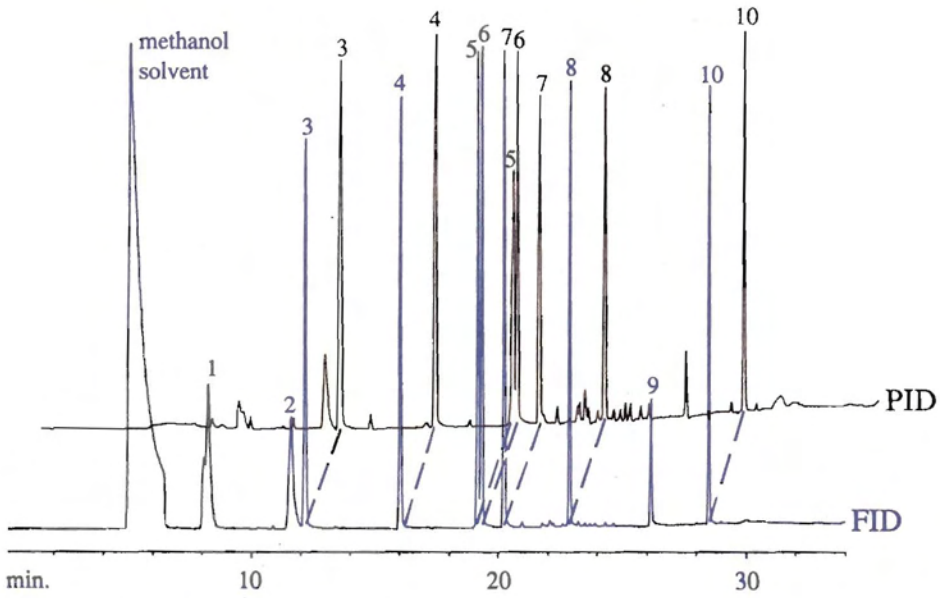
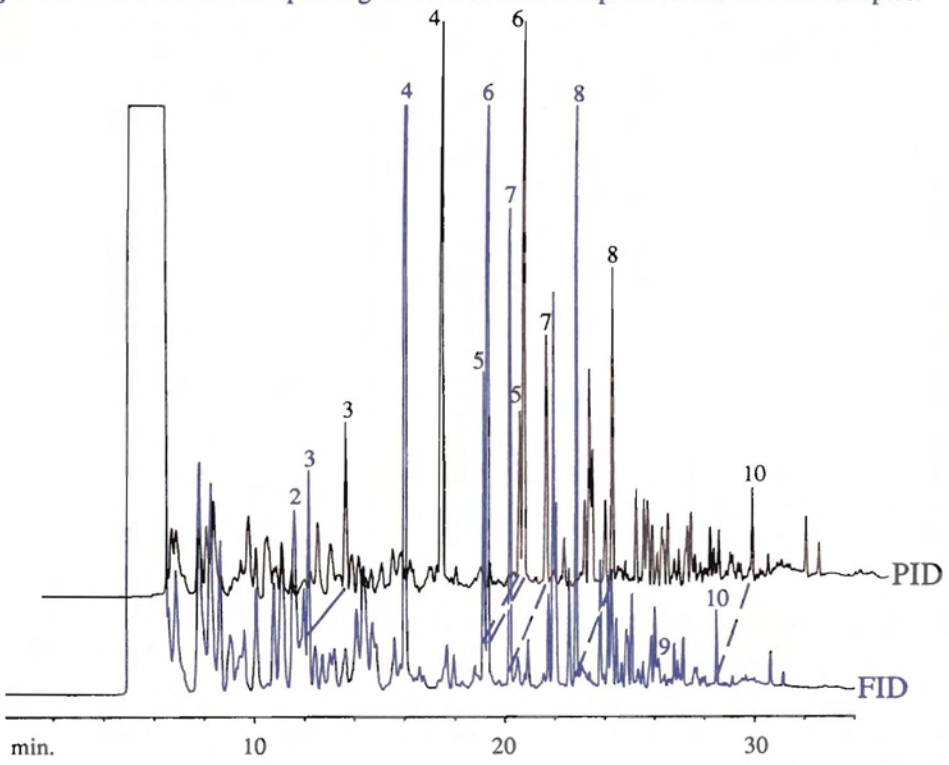


Figure 2 - The unleaded composite gasoline standard is representative of GRO samples.



Sample analysis and evaluating method performance

Once the retention time range and response factors are determined, it is good practice to perform an analysis of a spiked soil or water to determine the analyte recovery and method repeatability. For TPH, a typical gasoline such as the Restek composite gasoline can be used. To calculate BTEX recovery, an individual aromatic standard should be used. This is because the exact concentration of BTEX in the gasoline standard is not easily determined. The addition of an internal standard and surrogate to the samples prior to analysis will usually increase the precision of the results, especially for BTEX. Internal and surrogate standards that have been used successfully include α,α,α-triflourotoluene, 1-chloro-4-fluorobenzene, and 4-bromoflourobenzene.

of Restek's GRO Mix plus dodecane.* The second step is to calibrate the detector for the aromatic hydrocarbons (BTEX) and for the entire gasoline range (TPH). For BTEX, the calibration is straightforward, but for TPH there are two possible procedures. One procedure is to analyze a mixture of individual hydrocarbons covering the gasoline range (Figure 1) and calculate an average response factor from the response factors of each individual component. This calibration standard should be representative of the different types of hydrocarbons in gasoline. States recommending this method of calibration will specify the hydrocarbon components to be used. The other procedure for calibrating TPH response is to analyze a quantitative standard containing one or more gasolines. In theory, a composite should be more representative of the gasoline present in a wide range of samples to be analyzed. An example of a chromatogram generated from Restek's composite gasoline standard appears in Figure 2.

Avoiding some of the common pitfalls

The most common problem encountered in TPH/BTEX analysis is the presence of interfering compounds in the chromatographic analysis. Interferences can be caused by organic solvents present in the samples, background organic

Peak List and Run Conditions for Figures 1 - 2

	COMPOUNDS
1	3-methylpentane
2	2,2,4-trimethylpentane (isooctane)
3	benzene
4	toluene
5	ethylbenzene
6	*m*-xylene
7	*o*-xylene
8	1,2,4-trimethylbenzene
9	dodecane
10	naphtalene

Fig. 1) 105m, 0.53mm ID, 3.0µm Rtx®-502.2 (cat.# 10910)
Sample: GRO Mix (WISC) + dodecane
Concentration: 200ppb each in 5ml of H_2O

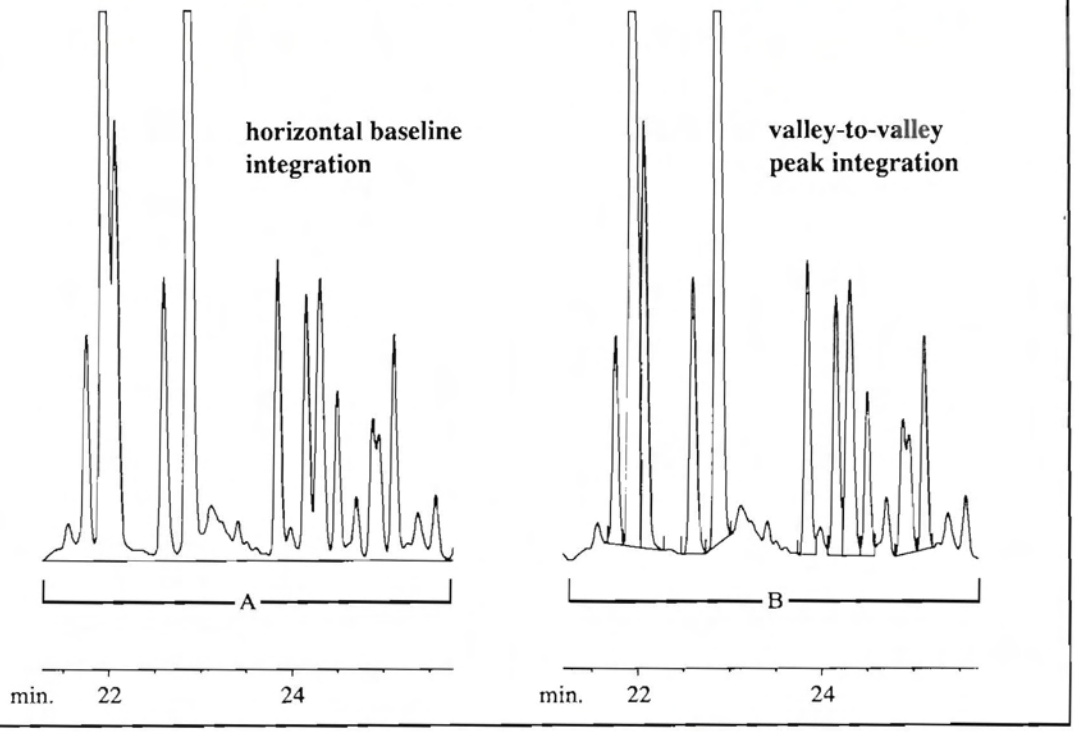
Fig. 2) 105m, 0.53mm ID, 3.0µm Rtx®-502.2 (cat.# 10910)
Sample: Unleaded Gasoline Composite Standard
Concentration: 5ppm in 5ml of H_2O

Oven temp.:	40°C (hold 1 min.) to 100°C @ 5°C/min., then to 240°C @ 8°C/min. (hold 8 min.)		
Inj. / det. temp.:	200°C/250°C		
Carrier gas:	helium (10cc/min.)		
FID sensitivity:	16 x 10^{-11} AFS		
Trap:	Tenax, Silica Gel, Charcoal		
Purge:	12 min. @ 40cc/min.		
Desorb preheat:	175°C	**Desorb temp.:**	180°C
Desorb time:	2 min.	**Desorb flow:**	10cc/min.

* *Some states specify dodecane as the end of gasoline.*

contamination, or carryover of hydrocarbons from previous chromatographic analyses. Each of these problems can result in reporting higher concentrations especially for TPH. To avoid contamination, prescreening the samples on a separate GC, prior to sample preparation is recommended. Overloading the instrument with hydrocarbon contaminants can be minimized by adjusting the sample amount, keeping it within the linear range of the method.

Figure 3 - Horizontal baseline integration provides best results for TPH analysis.



Another column problem encountered with this analysis is low TPH recoveries when response factor calibration is based upon a hydrocarbon component standard as opposed to a composite gasoline standard. A likely explanation for this is that the start and stop integration for gasoline is often well inside the gasoline range, depending upon the hydrocarbons used to set the range. Furthermore, low recoveries are often obtained due to errors in integrating the gasoline area. Figure 3 shows the difference between baselines obtained using horizontal baseline integration (A) and valley-to-valley peak integration (B) modes. The area resulting from the peak integration will give low recoveries because part of the gasoline area is excluded from the calculation. For best results with GRO samples, the baseline obtained should be determined at the beginning and end of the analysis, and a horizontal hold applied between these two points.

Although GRO methods differ between states, the basic procedures are similar. The capillary column frequently recommended for TPH and BTEX analysis is a 105m, 0.53mm ID, 3.0µm Rtx®-502.2. System calibration can be accomplished with either mixtures of individual hydrocarbons or composite gasoline standards. Analysts should refer to their specific methods for analytical and calibration procedures. ■

References

1) Tamlyn Oliver and Paul Kostecki, *Soils Magazine*, December 1992.
2) USEPA, *SW-846 Test Methods for Evaluating Solid Waste*, 3rd Edition; Method 5030, "Purge and Trap".
3) Federal Register 1984 Vol. 49, No. 209; USEPA Method 602 (Purgeable Aromatics).
4) USEPA, *SW-846 Test Methods for Evaluating Solid Waste*, 3rd Edition; Method 8020, "Aromatic Volatile Organics by Gas Chromatography".

Product Listing

Rtx®-502.2 105m, 0.53mm ID, 3.0µm cat.# 10910, $1200

Unleaded Gasoline Composite Standard
cat.# 30081, $25 each
cat.# 30081-500, $35 ea. w/data pack
cat.# 30181, $225 10pk. w/data pack

GRO Mix (WISC) cat# 30069, $25 each
cat.# 30069-500, $55 ea. w/data pack
cat.# 30169, $225 10pk. w/data pack

GRO Mix (EPA) cat.# 30065, $25 ea.
cat.# 30065-500, $55 ea. w/data pack
cat.# 30165, 10pk. w/data pack

1-chloro-4-fluorobenzene Standard
cat.# 30066, $25 each
cat.# 30066-500, $35 ea. w/data pack
cat.# 30166, $225 10pk. w/data pack

4-bromofluorobenzene Standard
cat.# 30067, $25 each
cat.# 30067-500, $35 ea. w/data pack
cat.# 30167, $225 10pk. w/data pack

α,α,α-trifluorotoluene Standard
cat.# 30068, $25 each
cat.# 30068-500, $35 ea. w/data pack
cat.# 30168, $225 10pk. w/data pack

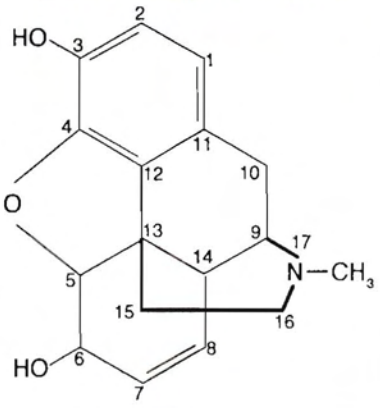
Additional calibration and internal standards/surrogate mixtures are available, including the modified Wisconsin PVOC/GRO Mix. Please call 800-356-1688 for information.

Clinical Corner

Opiate Analysis

Opiates or opioids are terms that classify a group of compounds with morphine-like actions. Their pharmacological properties include analgesia or pain relief, drowsiness and respiratory depression. Figure 1 shows the structure for morphine. Substitutions at the 3, 6, and 17 positions produce compounds with varying degrees of potency and pharmacological activity. The National Institute for Drug Abuse (NIDA) has targeted opiates as a class to be monitored in urine for detection of drug abuse. Testing guidelines have been established with a limit of detection of 0.3µg/ml for morphine. Screening of opiates is commonly done by using enzyme immunoassays. Enzyme immunoassays have the ability to cross react with a number of structurally similar opiates including codeine, hydromorphone, hydrocodone, levorphanol, and oxycodone. In order to differentiate between all of the possible substances being detected by enzyme immunoassay, confirmational analysis by GC/MS should be performed.

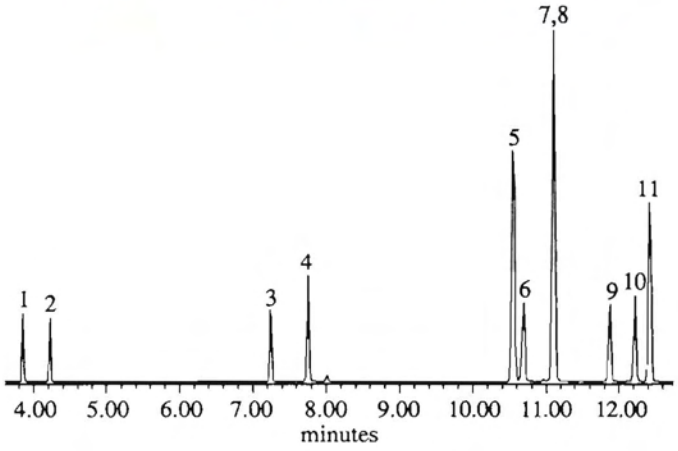
Figure 1 - Morphine structure



Chromatographic performance of the opiates is significantly affected by small changes in their chemical structure. The presence of hydroxyl groups at the 3 and 6 positions produce compounds that are more polar and reactive. Compounds with reactive hydroxyl groups in their chemical structure can suffer from adsorption and peak tailing, leading to diminished response in chromatographic systems that contain active sites. Sample preparation of sensitive compounds, like opiates, should take place in silanized glassware and samples should be stored in deactivated sample vials. Derivatization of reactive hydroxyl groups can improve chromatographic performance and detection limits and prevent sample loss on glassware and sample vials. Both trimethylsilyl and fluoroacyl derivatives of the opiates yield end products that are less polar and/or more volatile than the underivatized compound.

For this analysis, trimethylsilyl derivatives were prepared using BSTFA with 1% TMCS. Derivatizing the reactive hydroxyl group with a less polar trimethylsilyl group eliminates the tailing peaks commonly seen with compounds like morphine. Figure 2 shows the analysis of a selection of opiates on an Rtx®-5 column. Compounds that have been derivatized prior to analysis are designated as TMS in the peak list. The TMS derivatized opiates chromatograph well on a low polarity (Rtx®-5) column with good resolution and peak shape.

Figure 2 - Opiates analysis on an Rtx®-5 column.



30m, 0.25mm ID, 0.25µm Rtx™-5 (cat.# 10223)
2µl split injection of Opiates

Oven temp.: 200°C to 325°C @ 7°C/min.
Inj. temp.: 250°C
Det. type: HP MSD 5971A **Det. temp.:** 300°C
Carrier gas: helium **Linear velocity:** 30cm/sec. set @ 200°C
Split ratio: 50:1 **Ionization:** EI **Mode:** SIM

	COMPOUNDS	IONS MONITORED
1	meperidine	71, 246
2	alphaprodine	172, 187
3	methadone	72
4	levorphanol (TMS)	150, 270, 271, 328
5	codeine (TMS)	178, 196, 234, 371
6	hydrocodone	242, 299
7	morphine (TMS)	234, 429
8	hydromorphone (TMS)	356
9	oxycodone (TMS)	371, 386
10	oxymorphone (TMS)	444, 445
11	nalorphine (TMS)	414, 455

Sensitivity and specificity in confirming the presence of opiates in different samples can be enhanced by selectively choosing certain ions to monitor. Identification based upon the presence of distinctive, high mass ions is preferred, especially when analyzing derivatized compounds. Trimethylsilyl derivatives will add 72 amu for every hydroxyl group derivatized.

(**Clinical Corner** is continued on page 9.)

New! Rt-βDEXm™ Columns

Designed for the Separation of Optical Isomers

- Highly selective for the separation of enantiomers
- Inert and efficient
- Available in both 0.25 and 0.32mm ID
- Equivalent pricing to conventional liquid phase columns
- Individually tested with a chiral mix
- Permethylated β cyclodextrin derivative

Cyclodextrins Provide Unique Selectivity

The importance of chiral molecules and the role which enantiomers play concerning biological activity has escalated efforts in the production of optically pure isomers. High resolution gas chromatography is an exceptional analytical tool in the determination of optical purity of both natural and synthetic molecules.

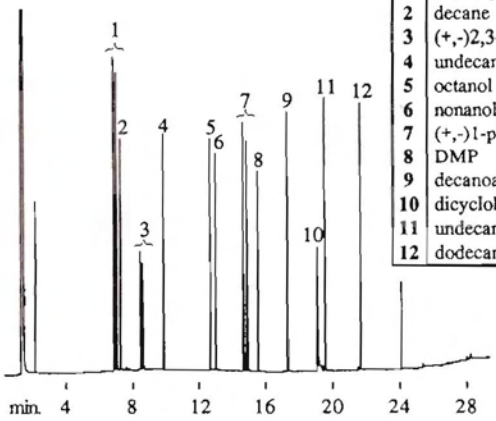
Alkylated cyclodextrin materials can be mixed with common liquid stationary phases to produce capillary columns with the ability to separate volatile enantiomers. The permethylated derivative of beta cyclodextrin is especially selective for a wide variety of chiral separations (1). Optical isomers or enantiomers are non-superimposable mirror images of one another differing only in their interaction with plane polarized light. They have identical physical properties such as boiling point, melting point, and spectroscopic features. Therefore, common liquid phases used in gas chromatography do not possess adequate selectivity for enantiomer separation. The actual mechanism by which cyclodextrin macromolecules (host) and enantiomers (guest) interact is not completely understood (2). Several forces may be involved in relation to "host-guest complexing" but the final result is chiral recognition.

Restek now offers chiral columns to meet the needs of enantiomeric separations. The Rt-βDEXm™ chiral column is a permethylated beta cyclodextrin material doped into the Rtx®-1701 (14% cyanopropyl/86% dimethyl polysiloxane) stationary phase. The Rt-βDEXm™ columns are available in 30-meter lengths with 0.25 and 0.32mm IDs. A 0.25µm stationary phase film thickness provides maximum efficiency and yields optimal resolution of enantiomeric pairs.

Restek's new Rt-βDEXm™ chiral columns are specially tested to ensure reproducibility and selectivity

To assure column-to-column reproducibility, Restek has designed a special test mix for Rt-βDEXm™ columns. The test mix includes three pairs of enantiomers: (+,-)α-pinene, (+,-)2,3-butanediol, and (+,-)1-phenylethanol. The 2,3-butanediol also serves as a test probe for inertness and selectivity. The 2,6-dimethyl phenol and dicyclohexylamine are included to insure acid/base compatibility of the stationary phase. A series of methyl esters is included for total retention and column efficiency measurements. Figure 1 shows the Chiral test mixture analyzed on a 30m, 0.25mmID, 0.25µm Rt-βDEXm™. The symmetrical peak shape and complete resolution of the racemic mixture of the enantiomers indicates both excellent column inertness and selectivity.

Figure 1 - The Rt-βDEXm™ column demonstrates excellent column inertness and resolution of test enantiomers.



Chiral Column Test Mix		
1	(+,-)α-pinene	1.0mg/ml
2	decane	.28
3	(+,-)2,3-butanediol	1.0
4	undecane	.29
5	octanol	.36
6	nonanol	.40
7	(+,-)1-phenylethanol	1.0
8	DMP	.32
9	decanoate	.42
10	dicyclohexylamine	.42
11	undecanoate	.41
12	dodecanoate	.31

30m, 0.25mm ID, 0.25µm Rt-βDEXm™(cat.# 13100)
1µl split injection of Chiral Test Mix (cat.# 35001)
Oven temp.: 40°C to 200°C @ 6°C/min.
Inj. & det. temp.: 200°C **Carrier gas:** hydrogen
Linear velocity: 50cm/sec. set @ 40°C **FID sensitivity:** 8 x 10^{-11} AFS
Split ratio: 42:1

Although the Rtx®-1701 siloxane stationary phase in the Rt-βDEXm™ column is immobilized, the cyclodextrin material can be rinsed out with many common solvents. Therefore, column rinsing is not recommended.

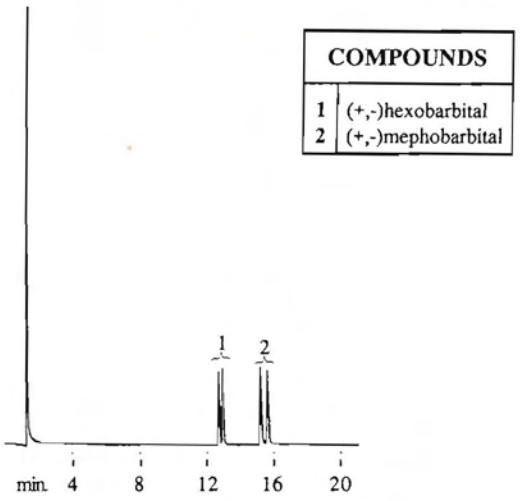
FDA recommends pharmokinetic and toxicity testing for individual enantiomers of new chiral drugs

Stereochemical properties of chiral drugs have been found in many instances to be the controlling factor concerning activity. For example, one enantiomer may be involved in a biological function while its isomeric partner is inactive or exhibits a different functionality. Metabolism of enantiomers may differ significantly, allowing for different rates of reaction for a particular biological process. In some cases one optical isomer may be harmful. Therefore, the Food and Drug Administration (FDA) has recently required drug manufacturers to test individual enantiomers of new chiral drugs for toxicity (3). Figure 2 shows a chromatogram of two common barbiturates analyzed on the Rt-βDEXm™. Resolution of the hexobarbital and mephobarbital enantiomers is obtained in 16 minutes.

Enantiometric separation is highly useful in identification and quality control of many flavors and essential oils

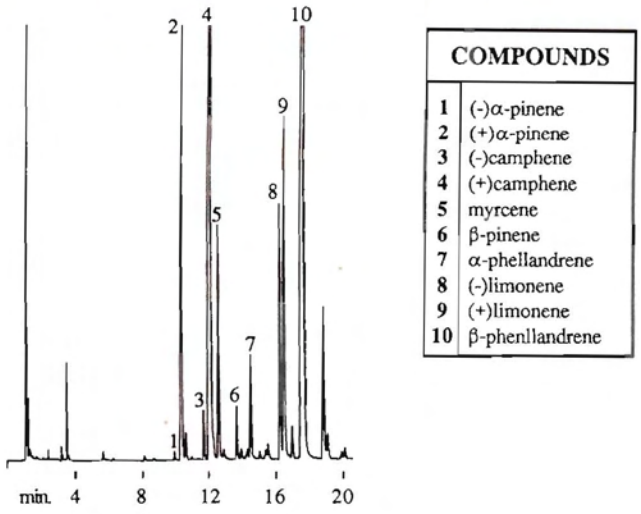
Enantiomeric recognition of compounds contained in natural products has enhanced our level of understanding in many

Figure 2 - The Rt-βDEXm™ column permits enantiomeric separation of common barbiturates.



30m, 0.25mm ID, 0.25µm Rt-βDEXm™(cat.# 13100)
0.5µl split injection of hexobarbital & mephobarbital, concentration=1mg/ml
Oven temp.: 205°C isothermal **Inj. & det. temp.:** 200°C
Carrier gas: hydrogen **Linear velocity:** 50cm/sec. set @ 40°C
FID sensitivity: 8 x 10^{-11} AFS **Split ratio:** 42:1

Figure 3 - Enantiomeric components of ginger oil can be resolved with the Rt-βDEXm™ column.



30m, 0.25mm ID, 0.25µm Rt-βDEXm™(cat.# 13100)
wet needle split injection of ginger oil, concentration=neat
Oven temp.: 50°C (hold 2 min.) to 190°C @ 1°C/min.
Inj. & det. temp.: 200°C **Carrier gas:** hydrogen
Linear velocity: 50cm/sec. set @ 50°C
FID sensitivity: 8 x 10^{-11} AFS **Split ratio:** 71:1 (Split flow: 100cc/min.)

disciplines of scientific research. Natural products often differ from source-to-source, so a thorough analysis of each batch and blend is necessary. Pheromone production in insects has been linked to chiral components in essential oils which are injested while feeding on plant life. The volatility of these compounds makes gas chromatography the ideal analytical tool.

Many cyclic ketones, known as flavor compounds, occur as constituents of essential oils. In some cases, the enantiomers may be distinctly different in flavor and physiological activity. Classes of natural essential oils can also differ in volatile constituents from one another depending on geographic location. Adulteration of natural flavors and fragrances by synthetic additives may also be pinpointed if one can discriminate between ratios of enantiomeric pairs. Figure 3 shows the analysis of ginger oil on a Rt-βDEXm™ column. The enantiomeric selectivity of the Rt-βDEXm™ aids in the identification of the essential oil.

The Restek Rt-βDEXm™ column is highly selective for a wide variety of chiral separations. These chiral columns provide maximum efficiency and resolution between enantiomeric pairs, while the special test mix ensures high column-to-column reproducibility and inertness. ■

References
(1) Keim et. al., "Enantiomer Separation by Gas Chromatography on Cyclodextrin Chiral Stationary Phases", *HRC&CC*, Volume 14, August 1991, 507-529.
(2) Wilfried A. Konig, *Gas Chromatographic Enantiomer Separation with Modified Cyclodextrins*, Huethig, 1992, 137-139.
(3) FDA's Policy Statement for the Development of New Stereoisomeric Drugs, May 1992.

Product Listing

Rt-βDEXm™ 30m, 0.25mm ID, 0.25µm cat.# 13100, $425
Rt-βDEXm™ 30m, 0.32mm ID, 0.25µm cat.# 13101, $460
Chiral Column Test Mix, in methylene chloride, 1ml per ampul, cat.# 35001, $25

Clinical Corner (continued from page 7)

Effective protocols for opiate analysis include extensive sample preparation and optimized instrument parameters. Derivative formation and the use of deactivated glassware, sample vials, and inlet liners will ensure maximum recoveries and response. Optimized detector parameters using selected ions for detection will aid in the identification and of different compounds. ■

Product Listing

Rtx®-5 30m, 0.25mm ID, 0.25µm cat.# 10223, $370

Restek offers a large variety of inlet sleeves for numerous manufacturer's GCs. Please refer to our General Catalog or call customer service at 800-356-1688, ext. 3.

Coming Soon . . . Chemical Standards for Drug Analysis!

Standards Spotlight

EPA Quick Turnaround Method SOW Standards

• *High concentration for maximum value* • *Meet EPA specified quality criteria* • *Full data packs available* •

Restek now has stock chemical standards for all of the EPA Quick Turnaround Methods (QTM) specified in the most recent Statement of Work (SOW). These standards are prepared using precise gravimetric techniques, with concentration verification performed using state-of-the-art capillary chromatography methods.

Quick Turnaround Methods are designed to provide timely data to EPA project officers in several crucial situations: 1) Where field sampling teams have limited knowledge of a waste site and need to focus samples being taken from a particular area, and 2) Where remediation is being performed with heavy equipment on site waiting for sample analysis before proceeding. In all cases, QTM methods require laboratories to submit data to the EPA project officer within 24 hours.

System Monitoring Compounds (SMC) are included in the calibration mixtures at the specified level. Each SMC is also available in an appropriate solvent for matrix spike solution preparation.

QTM Volatiles Method

The QTM volatiles are available in two calibration solutions. Calibration Mix #1 contains all of the components except vinyl chloride. A separate solution containing just vinyl chloride is offered, since this compound is extremely volatile, allowing laboratories to replace vinyl chloride regularly without replacing the less volatile components in Calibration Mix #1.

QTM VOA Calibration Mix #1

2000µg/ml each in 1ml purge & trap grade methanol.
Packaged 1ml per ampul.

benzene
bromodichloromethane
bromoform
carbon tetrachloride
chloroform
chlorobenzene
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethene
trans-1,2-dichloroethene
ethylbenzene
1,1,2,2-tetrachloroethene
tetrachloroethene
toluene
o-xylene
p-xylene
1,1,1-trichloroethane
trichloroethene
cis-1,2-dichloroethene
4-bromofluorobenzene (SMC)

Cat.# 30088 $30 ea.
30088-500 $70 ea. w/data pack
30188 $270 10 pk. w/data pack

QTM VOA Calibration Mix #2

Contains vinyl chloride at 2000µg/ml in 1ml purge & trap grade methanol.
Packaged 1ml per ampul.

Cat.# 30089 $30 ea.
30089-500 $40 ea. w/data pack
30189 $270 10 pk. w/data pack

QTM VOA SMC Mix

4-bromofluorobenzene is the specified SMC in this method. The recommended working solution is to be prepared at a concentration of 50µg/ml in methanol. The following products may be used to prepare the SMC working solution with their respective dilution ratio.

conc. µg/ml	dilution ratio	each	each w/data pk.	10pk. w/data pk.
10,000	1:200	30082/$25	30082-500/$35	30182/$225
5,000	1:100	30003/$25	30003-500/$35	30103/$225
2,500	1:50	30067/$25	30067-500/$35	30167/$225

QTM Phenols Method

This method allows the laboratory to select one of two sample extraction procedures. Because the calibration mix should be prepared in the same solvent as the final sample extract, two different calibration mixes are offered. Use the QTM Phenols Calibration Mix A (in acetonitrile) when extracting samples using solid phase extraction (SPE), and the QTM Phenols Calibration Mix B (in methylene chloride) when using the liquid/liquid extraction procedure.

QTM Phenols Calibration Mix A

Contains 2500µg/ml of each compound in 1ml acetonitrile.
Packaged 1ml per ampul.

phenol
2-methylphenol
2-nitrophenol
2,4-dichlorophenol
2,4,6-trichlorophenol
4-nitrophenol
pentachlorophenol
2-bromophenol (SMC)
2-chlorophenol
3-methylphenol
2,4-dimethylphenol
4-chloro-3-methylphenol
2,4-dinitrophenol
2-methyl-4,6-dinitrophenol
2,3,4,6-tetrachlorophenol

Cat.# 31201 $30 ea.
31201-500 $70 ea. w/data pack
31301 $270 10 pk. w/data pack

QTM Phenols Method (cont.)

QTM Phenols Calibration Mix B

Contains 2500µg/ml of each compound in 1ml methylene chloride
Packaged 1ml per ampul.

phenol	2-chlorophenol
2-methylphenol	3-methylphenol
2-nitrophenol	2,4-dimethylphenol
2,4-dichlorophenol	4-chloro-3-methylphenol
2,4,6-trichlorophenol	2,4-dinitrophenol
4-nitrophenol	2-methyl-4,6-dinitrophenol
pentachlorophenol	2,3,4,6-tetrachlorophenol
2-bromophenol (SMC)	

Cat.# 31205 $30 ea.
31205-500 $70 ea. w/data pack
31305 $270 10 pk. w/data pack

QTM Phenol SMC Mix

Contains 2-bromophenol at 20,000µg/ml in 1ml methanol.
Cat.# 31202 $25 ea.
31202-500 $35 ea. w/data pack
31302 $225 10 pk. w/data pack

QTM Polynuclear Aromatic Hydrocarbons Method

QTM PAH Calibration Mix

Contains 1000µg/ml of each compound in 1ml methylene chloride.
Packaged 1ml per ampul.

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

Cat.# 31203 $45 ea.
31203-500 $85 ea. w/data pack
31303 $405 10 pk. w/data pack

QTM PAH SMC Mix

Contains 2-bromonaphthalene at 20,000µg/ml in 1ml methanol.
Cat.# 31204 $25 ea.
31204-500 $35 ea. w/data pack
31304 $225 10 pk. w/data pack

QTM Pesticides Method

QTM Pesticide Calibration Mix

Contains 25µg/ml of each compound in 1ml hexane
Packaged 1ml per ampul.

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

Cat.# 32036 $30 ea.
32036-500 $70 ea. w/data pack
32136 $270 10 pk. w/data pack

QTM Pesticide SMC Mix

This method specifies preparing a working solution at 5µg/ml.
Contains decachlorobiphenyl at 125µg/ml in 1ml acetone.
Cat.# 32037 $25 ea.
32037-500 $35 ea.
32137 $225 10 pk. w/data pack

QTM PCB Method

This method requires the use of individual Aroclors® in solution with the exception of Aroclor® 1016 and 1260, which are analyzed together. The Aroclor® 1016/1260 mixture, along with the System Monitoring Compound (decachlorobiphenyl) are calibrated at three concentration levels. All other Aroclors® and toxaphene are calibrated at a single concentration.

Aroclor® 1016/1260 Mixture

Contains Aroclor® 1016 and Aroclor® 1260 at 1000µg/ml each in 1ml hexane.
Packaged 1ml per ampul.
Cat.# 32039 $25 ea.
32039-500 $35 ea. w/data pack
32139 $225 10 pk. w/data pack

(Please See Aroclor® & Toxaphene Product Listing Table Below.)

QTM PCB SMC Mix

The method specifies preparing a working solution at 2µg/ml.
Contains decachlorobiphenyl at 200µg/ml in 1ml acetone.
Cat.# 32029 $25 ea.
32029-500 $35 ea. w/data pack
32129 $225 10 pk. w/data pack

Aroclors® & Toxaphene

1000µg/ml in 1 ml hexane	Individual		Individual w/data pack		10pk. w/data pack	
Aroclor® 1221	32007	$25	32007-500	$35	32107	$225
Aroclor® 1232	32008	$25	32008-500	$35	32108	$225
Aroclor® 1242	32009	$25	32009-500	$35	32109	$225
Aroclor® 1248	32010	$25	32010-500	$35	32110	$225
Aroclor® 1254	32011	$25	32011-500	$35	32111	$225
Toxaphene	32005	$25	32005-500	$35	32105	$225

To order any Restek product, call 800-356-1688 (ext.3).

Hints for the Capillary Chromatographer

Selecting the Proper Ferrule for Capillary Columns

Proper ferrule selection is critical for capillary column installation. Characteristics such as thermal stability, ruggedness, and compressibility are determined by the different materials used to make ferrules. It is important to choose the right ferrule type and size to ensure proper column installation. The wrong ferrule type could cause damage to sensitive detectors such as ECDs, ELCDs, and MSDs. The wrong ferrule size or type can cause system leaks that result in decreased sensitivity and deterioration.

Ferrule Materials

Since metal ferrules would damage fused silica tubing, softer materials are used for capillary column ferrules. The two most common materials for capillary column ferrules are graphite and Vespel®. These materials can also be combined to form hybrid ferrules with the benefits of each material. Other ferrule materials, such as Teflon® and silicone, are commonly used with packed columns, but because of their limited thermal stability they are not typically used with capillary columns. Table I lists the maximum operating temperatures and the characteristics of common capillary ferrule materials.

Table I - Common Characteristics of Capillary Ferrules

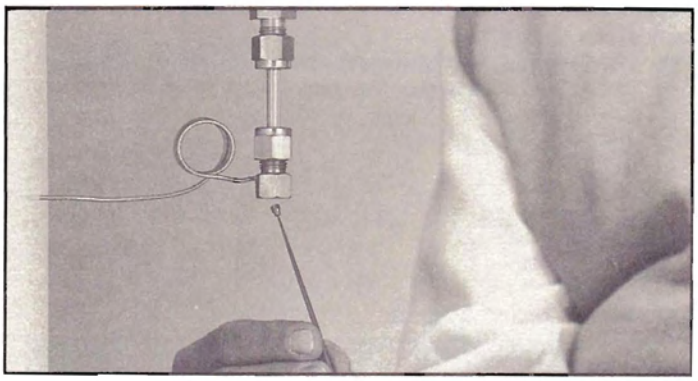
Material	*Max Temp.*	*Characteristics*
Graphite	450°C	Soft, easily conforms to all column sizes. Excellent for high temperature applications. Can flake or deposit particles in inlet & detector fittings. Easily deforms, resulting in limited reusability. Not recommended for vacuum interfaces.
Vespel®/Graphite	400°C	Hard, must be sized to exact column OD. Contracts when cooled causing leakage if not retightened after several thermal cycles. Excellent reusability.

Properties of Graphite Ferrules

Many chromatographers prefer graphite ferrules because they are soft and easily conform to any fitting dimension. Most graphite ferrules are made by tightly winding graphite ribbon around a pin and compressing it into a mold. The graphite ribbon increases ferrule pliability and allows it to deform easily. Increased pliability makes it possible to seal a 0.4mm OD (0.25mm ID) fused silica column with a 0.8mm ID ferrule. In addition, the ferrule can accomodate larger columns if the graphite bore is cored out. These features allow chromatographers to always have the right size ferrule on hand.

Graphite ferrules should be tightened using minimal force. Usually ¼-turn past finger-tight is sufficient to form a leak-tight seal. If a graphite ferrule is over-tightened, it will extrude out of the bottom of the nut, deform into the fitting cavity, and create ferrule fragments. These particles can be driven further into the inlet or make-up gas fitting, causing adsorption or peak tailing when a column is reinstalled. Graphite ferrules can also flake or abrade and emit particles that can clog small orifices. Because graphite is porous, graphite ferrules leak under vacuum. Therefore, graphite ferrules are not recommended for detectors operated under vacuum, such as MSDs.

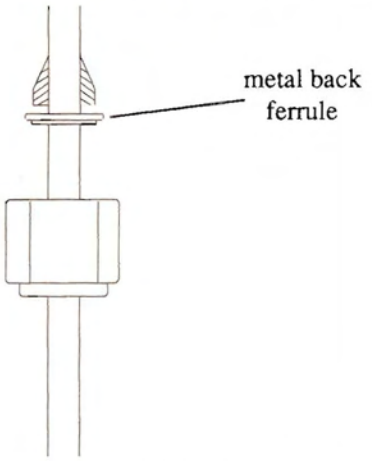
Graphite ferrules must be carefully removed, otherwise fragments and flakes remaining in the fitting can contaminate the GC system. Ferrules are easily dislodged by inserting a tapered needle file into the bore and moving it side-to-side. If the graphite ferrule does not come out in one piece, the inlet or detector fitting should be completely disassembled to ensure that no ferrule fragments remain.



Needle files easily remove graphite ferrules from injector and detector fittings or nuts. Gently insert the file into the ferrule bore and move it from side-to-side to dislodge the ferrule.

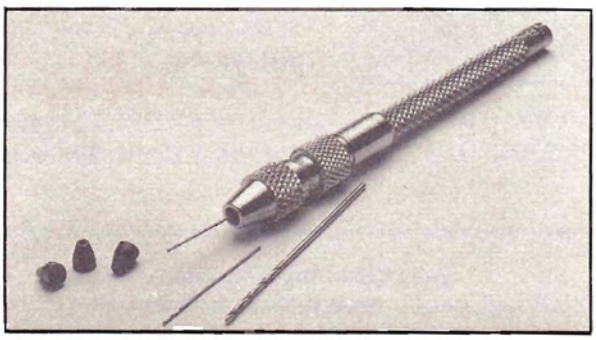
The life of a graphite ferrule is limited because they compress so easily. Some chromatographers obtain new life from a crushed ferrule by installing a reversed Swagelok®-type back ferrule between the fitting and the ferrule (Figure 1). The back ferrule raises the graphite ferrule higher in the fitting, allowing it to seal again.

Figure 1 - Give a used graphite ferrule new life by installing a reversed metal back ferrule in the fitting.



Both 100% Vespel® and Vespel®/graphite ferrules are available. Vespel®-type ferrules are often preferred because they do not flake, deposit particles, or fall apart in a fitting. Most chromatographers choose the Vespel®/graphite ferrule combination. These ferrules are made by compressing a graphite/polyimide powder under high pressure in a heated mold. They retain their shape and can easily be removed intact. Vespel®/graphite has a higher thermal stability than Vespel® (400°C vs. 350°C) and the graphite impregnation makes the ferrule feel softer and seal with less torque. Vespel®/graphite ferrules are currently available in combinations ranging from 85% Vespel®/15% graphite to 60% Vespel®/40% graphite. The 60/40 Vespel®/graphite combinations are preferred by most chromatographers because they seal with the least amount of torque.

Unlike graphite, the inside diameter of Vespel®-type ferrules must be very close to the column OD in order to seal properly. If the ID of a Vespel®-type ferrule is too large for the column OD, it will not compress properly and allow a leak. Usually, the ferrule forms an oval shape, gripping the tubing but not sealing at the ends of the oval. If the ID of a Vespel®-type ferrule is too small to fit over the column, the bore must be enlarged with a small drill.



If the Vespel®/graphite ferrule's ID is too small to fit over the column, a pin vise drill can be used to enlarge the bore.

Vespel®/graphite ferrules will deform to the exact fitting dimension when heated. Usually this deformation process causes the ferrule to become loose and leak during the cool down cycle of a GC oven. Therefore, they must be subsequently retightened after several thermal cycles or carrier gas leakage will occur. No additional shrinkage or loosening occurs once the ferrule has conformed to the internal dimensions of the fitting cavity.

Vespel® ferrules can be removed from a fitting using a tapered needle file in the same manner as a graphite ferrule. Vespel® ferrules sometimes stick to the fitting and column after they have been in use for a prolonged period. Stuck ferrules can be removed by tapping the fitting with a solid object such as a wrench and gently pulling outward on the column. This problem is greatly minimized by using Vespel®/graphite combination ferrules.

What are common ferrule sizes?

Most column connections in the GC inlet and detector are made using $^1/_{16}$" Swagelok®-type fittings. The ID or opening of the ferrule depends on the outside diameter of the column. Table II lists common fused silica capillary column IDs, ODs, and recommended ferrule sizes.

Table II - Common Ferrule Sizes for Fused Silica Capillary Columns

Column ID	*Column OD*	*Ferrule Opening*
0.18 to 0.25mm	0.35 to 0.40mm	0.4mm
0.32mm	0.45 to 0.48mm	0.5mm
0.53mm	0.69 to 0.72mm	0.8mm

The choice of ferrule material is often personal preference. If you are installing a capillary column for the first time, we suggest using a graphite ferrule. Graphite easily forms a leak-tight seal and conforms to any column OD. If you frequently install new columns, Vespel®/graphite is recommended to eliminate particle evolution and minimize maintenance downtime. However, when connecting columns to MSDs or Mass Spectrometer transfer lines, Vespel®/graphite is the only ferrule you should use to ensure a leak-free seal under vacuum. We recommend trying both ferrule types to choose a ferrule that best fits your needs. ■

Suggestions?

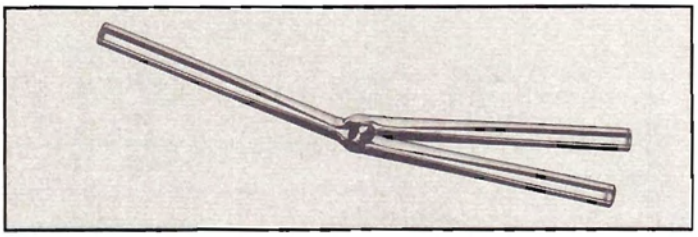
Is there a topic you would like to see covered in "Hints for the Capillary Chromatographer"? If so, please call our technical service department toll-free at 800-356-1688, ext. 4.

Peak Performers

Universal Angled "Y" Press-Tight® Connector

- Made from inert fused silica
- Fabricated at an angle approximating the radius of a capillary column.
- Does not place a strain on column end connections.

Universal "Y" Press-Tights® have become popular for splitting the sample between two columns for simultaneous confirmational analysis. They are also used for splitting the column effluent onto two different detectors. Our analysts had difficulty keeping the column ends sealed in the Press-Tight® because the standard straight "Y" creates strain on the fused silica tubing. To correct this problem, we have designed a "Y" connector bent at the appropriate angle to reduce the strain when connecting two columns or attaching a guard column or transfer line to an analytical column. Now both the inlet and outlet ends of the "Y" conform to the column radius. Fits fused silica tubing with ODs ranging from 0.3 to 0.8mm.



Universal Angled "Y" Press-Tight® Connector
cat.# 20403, $65 each
cat.# 20404, $175/3-pack

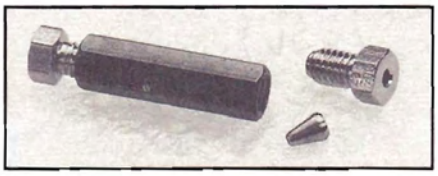
MXT® Low Dead Volume Connectors

In response to customer requests, we have developed metal connectors to join two MXT® columns, attach an MXT® guard column to an analytical column, or perform confirmational analysis with two MXT® columns.

These low dead volume connectors are Silcosteel®-treated, just like our MXT® columns, to make them inert to active compounds. We chose a 1/32" body size to minimize thermal mass and manufactured special metal ferrules that fit the OD of our 0.28 and 0.53mm ID MXT® columns perfectly.

The union connects two pieces of MXT® tubing and the "Y" connects two columns to a guard column or one column to two different detectors. These connectors will not cause peak tailing or affect system inertness and can be used up to 400°C without degrading the deactivation layer.

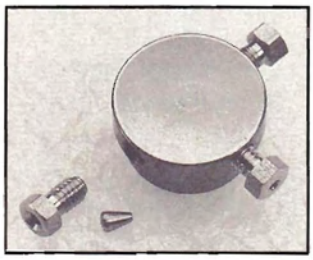
To connect a 0.53mm ID guard column to a 0.28mm ID MXT® analytical column, simply buy the appropriate ferrule sizes. The bodies of both the union and "Y" connectors are the same. A connector for 0.28mm ID MXT® columns will work for 0.53mm ID MXT® columns if the correct ferrules are used. See the chart below to determine what ferrule internal diameter fits the appropriate MXT® column.



MXT® Low Dead Volume Connector

- Connect guard columns/transfer lines to MXT® columns.
- Low thermal mass tracks rapid oven temperature programming.

for 0.28mm ID MXT® columns: cat.# 20397, $50 each
for 0.53mm ID MXT® columns: cat.# 20394, $50 each



MXT® Low Dead Volume "Y" Connector

- Connect two MXT® columns to one inlet.
- Connect one MXT® column to two detectors.

for 0.28mm ID MXT® columns: cat.# 20396, $90 each
for 0.53mm ID MXT® columns: cat.# 20395, $90 each

MXT® Connector Replacement Ferrules

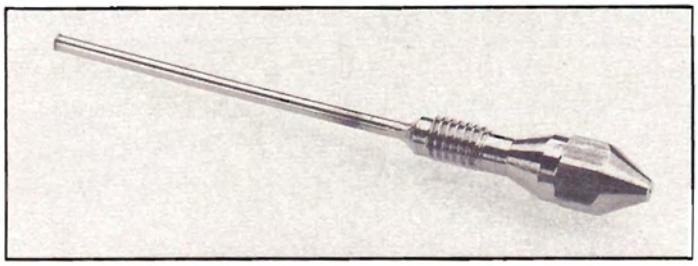
Ferrule ID	Fits column ID	cat.#	price
0.59mm	0.28mm	20398	$45/10-pk.
0.79mm	0.53mm	20399	$45/10-pk.

1/32" replacement nut: cat.# 20389, $15/5-pk

1/4"-3/16" Open End Wrench
A high quality wrench to use with the MXT® Low Dead Volume Connectors.
cat.# 20388, $20/2-pk.

FID Replacement Jet for Hewlett-Packard 5890 GCs

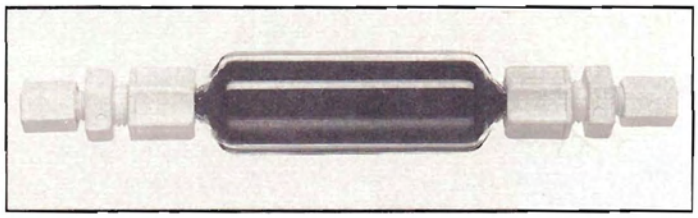
- Fluted jet tip easily guides capillary column into jet bore.
- High performance, Silcosteel® version eliminates adsorption of active compounds.
- Engineered to exceed original equipment specifications.
- Priced lower than HP replacement.



Restek has developed two versions of an HP 5890 FID jet. The standard version (replaces HP part# 19244-80560) is engineered with a fluted jet tip to guide the capillary column into the jet. This design prevents the fused silica column end from hitting the jet tip during installation. The high performance version is the same as the standard version, except that it has been treated with the Silcosteel® process to create an inert interior and exterior. This process coats the entire jet with a micron layer of silica and then further passivates the metal surface by deactivating it in the same manner as our MXT® columns. The high performance jet is extremely inert to active environmental or pharmaceutical compounds. Both versions are precisely machined and undergo stringent quality control to ensure the performance meets or exceeds the original specifications.

Standard HP 5890 Capillary Replacement FID Jet
cat.# 20670, $36 each
cat.# 20671, $95/3-pack
High Performance HP 5890 Capillary Replacement FID Jet
(treated with Silcosteel®, use with active compounds)
cat.# 20672, $48 each
cat.# 20673, $125/3-pack

New High Capacity Split Vent Trap



Potentially hazardous or carcinogenic chemicals can enter the lab atmosphere through the split vent in a capillary GC. As much as 99% of the sample injected vents to the air where chemists working nearby breath these pollutants. This problem is further magnified when multiple GCs are used in the same lab. Split vent traps, packed with charcoal, reduce the uncontrolled release of hazardous materials into the lab.

After examining many trapping materials, we chose a special type of activated coconut charcoal due to its tenacious trapping ability. Several trap designs were also evaluated. Narrow 1/4" trap bodies cause increased back pressure on the inlet system and severely retard retention times. In addition, the excessive backpressure on the split vent outlet can cause the back pressure regulator to perform erratically when the solvent expansion pulse occurs. Therefore, a large trap body design maximizes the quantitiy of charcoal that comes in contact with the sample vapor stream without causing unreasonable backpressure. Trap bodies made from solvent resistant plastics were investigated but continuous solvent exposure caused either cracking or leakage. A glass trap body provided the best resistance and longevity from repeated solvent injections.

When compared to other designs, the new high capacity split vent trap more than quadruples the number of injections that can be performed before solvent breakthrough occurs when compared to other designs (1300 vs. 300 injections). The trap provides protection for thirteen hundred injections or 50 days if one analysis is performed per hour. We recommend trap replacement every 1300 injections or at least every two months. The 1/8" female fittings accomodate most GCs and allow easy installation.

High Capacity Split Vent Trap Kit
includes 1/8" copper connecting tubing, Velcro® mounting strip, and 1/8" ferrule fittings
cat.# 20698, $25 each
cat.# 20699, $100/5-pack

To order any Restek product, call 800-356-1688 (ext.3).
For direct technical service, call 800-356-1688 (ext. 4).

THE RESTEK ADVANTAGE

A Thin Film (0.25µm) 0.53mm ID Rtx®-1701 Provides the Best Overall Resolution of Pesticides in the Least Amount of Time

In March of 1990, the EPA's Contract Lab Program (CLP) Statement of Work was revised to allow the use of 0.53mm ID capillary columns for the analysis of pesticides and Aroclors®. All subsequent statements of work have included this option including the 12/90 and 2/91 revisions. This method makes recommendations for the use of 1701 (cyanopropylphenyl polysiloxanes) and 608 (methyl phenyl polysiloxane) capillary columns, and has a provision for allowing equivalent columns if they meet the resolution and calibration requirements. The method also makes recommendations for run conditions to be used with both columns. However, many labs following these recommendations find it difficult to meet the resolution criteria specified in the method. To reduce analysis time, many labs prefer to run both columns simultaneously using the same run conditions. This is difficult because the optimum run conditions for one column may not be the optimum for the other column.

Selecting a single set of run conditions that will be appropriate for both columns can be a time consuming task. Using *ezGC™*, a new methods development software program, can simplify the job of selecting the optimum run conditions that will work for both capillary columns. In addition, *ezGC™* will insure that the analysis can be achieved in the fastest possible time, thereby reducing analysis costs even further.

in this issue...

EPA CLP pesticide method requirements

The EPA's CLP Statement of Work for pesticides and Aroclors® specifies very strict calibration, resolution, and analytical sequence requirements for the method. Resolution requirements state that all components in the Resolution Check Mixture must be greater than or equal to 60% resolved, all components in the Performance Evaluation Mixture must be 100% resolved, and components in the individual pesticide mixtures A or B must be greater than or equal to 90% resolved. Tables I, II, and III list the components in the resolution check mixture, performance evaluation mixture, and the individual pesticide mixtures, respectively.

Table I - Resolution Check Mixtures, must be ≥ 60% resolved

γ-chlordane	endrin ketone	endosulfan I
methoxychlor	p,p'-DDE	tetrachloro-m-xylene
dieldrin	decachlorobiphenyl	endosulfan sulfate

Table II - Performance Evaluation Mixture, must be 100% resolved

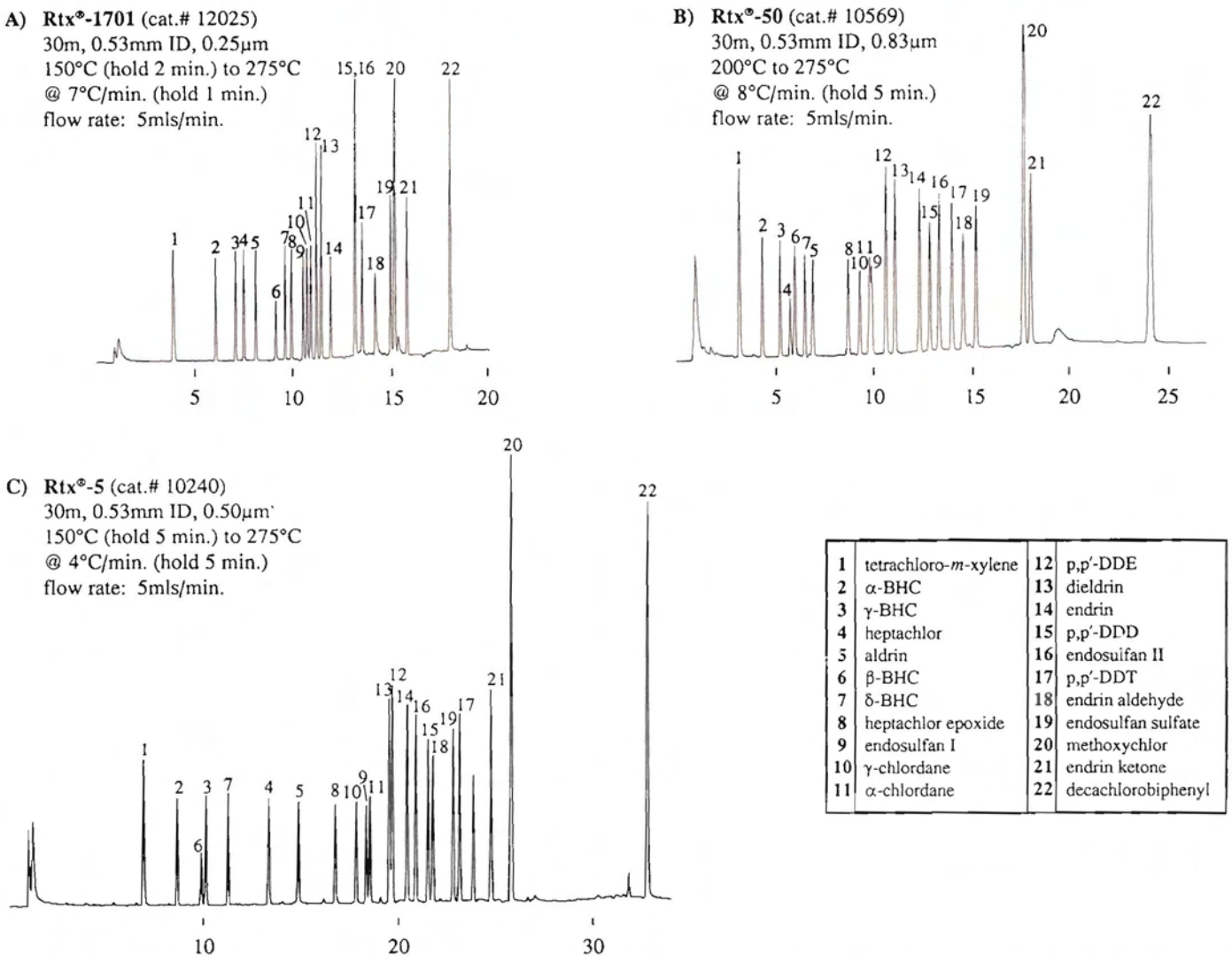
γ-BHC	endrin	α-BHC
methoxychlor	p,p'-DDT	tetrachloro-m-xylene
β-BHC	decachlorobiphenyl	

Table III - Individual Standard Mixtures A or B, must be ≥ 90% resolved

Mixture A	Mixture B	
α-BHC	β-BHC	p,p'-DDE
heptachlor	δ-BHC	endosulfan sulfate
γ-BHC	aldrin	endrin aldehyde
endosulfan I	heptachlor epoxide	endrin ketone
dieldrin	α-chlordane	endosulfan II
endrin	γ-chlordane	tetrachloro-m-xylene
p,p'-DDD	decachlorobiphenyl	
p,p'-DDT		
methoxychlor		
tetrachloro-m-xylene		
decachlorobiphenyl		

The resolution criteria specified in the method must be met by both columns used in the analysis. The method recommends the use of 0.53mm ID capillary columns with 1701 (cyanopropyl phenyl polysiloxane) and 608 (phenyl methyl polysiloxane) stationary phases, but equivalent columns can be used if they meet the requirements for resolution and initial calibration. In order to meet the resolution criteria, *ezGC™* software

Figure 1 - Resolution criteria of CLP pesticides are exceeded using optimized temperature program conditions for each column as predicted by *ezGC™*.



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

was used to optimize column selection and run conditions for the analysis of chlorinated pesticides specified in the EPA's CLP.

How does *ezGC™* work?

The first step in optimizing the run conditions is to determine the retention of CLP pesticides using both a slow (4°C/min.) and a fast (12°C/min.) temperature program ramp on each column. The retention data for the two analyses on each column type is entered into *ezGC™* along with the peak identifications. The software will then optimize the operating parameters such as initial temperature, initial hold time, program rate, final temperature, and final hold time. The software will list the 50 best run parameters in order of shortest analysis time. The list generated for each column type can be compared to determine what set of run parameters, with the shortest analysis time, will work for both columns. Other parameters such as flow rate, column length, ID, and film thickness can also be varied to determine if changing these parameters will improve resolution or reduce analysis time. A listing of elution order, retention time, and resolution for the components can be obtained for any of the 50 conditions. In addition, a simulated chromatogram can also be printed.

The results of the *ezGC™* optimization of the CLP pesticide analysis shows that the three columns and optimized temperature program conditions in Figure 1 **exceed** the resolution requirements of the method.

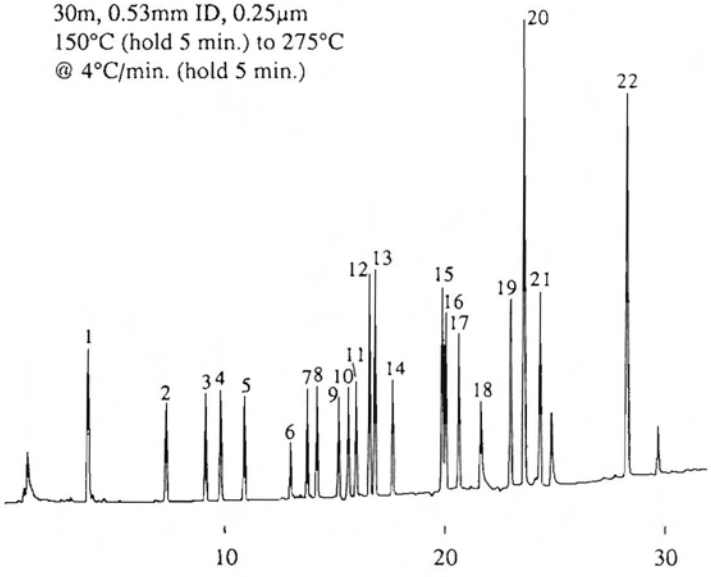
Of the three column choices, the Rtx®-1701 gives the fastest analysis time of the pesticides in 18 minutes, but yields a complete coelution of p,p'-DDD/endosulfan II (Figure 1A). Originally a 0.5µm Rtx®-1701 was used but a computer analysis using *ezGC™* demonstrated that the 0.25µm Rtx®-1701 column would provide a faster analysis time and better resolution than the 0.5µm film. In addition, a 0.25µm column will exhibit better thermal stability and less bleed than the 0.5µm Rtx®-1701, making it clearly the column of choice. The

Rtx®-50 also meets and exceeds the resolution criteria with analysis times of 24 minutes, but gives only 20% resolution of α-chlordane/endosulfan I (Figure 1B). The Rtx®-5 gives the best resolution of the CLP pesticides, but has an analysis time of 30 minutes (Figure 1C).

Column choice and recommended run conditions for the simultaneous analysis of CLP pesticides

The CLP Statement of Work also makes recommendations for simultaneous dual column analysis. In dual column confirmational analysis, a sample is run on two columns of differing polarity, yielding two separate chromatograms from one injection. Simultaneous dual column analysis both improves qualitative analysis and sample throughput in the lab. However, finding the optimum temperature program to use on both columns is a difficult, time consuming task. Since the optimum temperature program conditions for the Rtx®-1701, -50, and -5 were all different as seen in Figure 1, we used *ezGC™* to predict a simultaneous optimum temperature program for a dual column configuration. Figure 2 shows the results of the simultaneous optimization. The optimized dual column system is made up of the Rtx®-5 (30m, 0.53mm ID, 0.50µm) and the Rtx®-1701 (30m, 0.53mm ID, 0.25µm) and can be run simultaneously at 150°C (initial hold 5 min.) to 275°C @ 4°C/min. (hold 5 min.). All resolution criteria are maintained in the simultaneous dual column confirmational system and analysis is complete in 30 minutes. In addition, neither column shows coelutions under the simultaneous optimum temperature program chosen.

Figure 2 - Simultaneous dual column confirmation of CLP pesticides can be obtained using an Rtx®-5/Rtx®-1701 column combination.



Please refer to Figure 1C for the Rtx®-5 chromatogram and peak identifications.

Problems associated with pesticide analyses

The analytical sequence outlined in the CLP protocol specifies that an injection of the instrument blank and individual standard mixtures A & B be run after every 12 hours to maintain GC calibration. After 24 hours, an instrument blank and performance evaluation mixture must be run to verify calibration. These two steps are repeated at 12 and 24 hour intervals. Additionally, the breakdown of DDT and endrin in the performance evaluation mixtures must be less than 20% and the combined breakdown of DDT and endrin must be less than 30% to maintain system inertness.

Typically, system calibration and inertness are not met when the GC system and column become contaminated from dirty samples. In the *ezGC™* optimization of CLP pesticides, the resolution criteria within the software was set to exceed the method requirements (100 vs 60%) to permit longer analytical sequences before contamination affects resolution and inertness and GC maintenance is required. An Rtx®-1701 (30m, 0.53mm ID, 0.25µm), Rtx®-50 (30m, 0.53mm ID,0.83µm), or Rtx®-5 (30m, 0.53mm ID, 0.50µm) column can be used to analyze CLP pesticides. The resolution criteria specified in the method is met on any one of these three columns. However, we found that the Rtx®-1701/Rtx®-5 combination offers the best resolution in the least amount of time for dual column simultaneous analysis. (Note - for further information about dual column simultaneous analysis or using 0.53mm ID columns in the direct injection mode, request Restek's *Guide to Direct/On-column Flash Vaporization Injection.*) ■

Product Listing

Rtx®-5	30m, 0.53mm ID, 0.50µm, cat.# 10240, $445
Rtx®-1701	30m, 0.53mm ID, 0.25µm, cat.# 12025, $445
Rtx®-50	30m, 0.53mm ID, 0.83µm, cat.# 10569, $495

Resolution Check Mix,
cat.# 32001, $30 each
32001-500, $60 ea. w/data pack
32101, $270 10pk. w/data pack

Performance Evaluation Mix,
cat.# 32002, $30 each
32002-500, $60 ea. w/data pack
32102, $270 10pk. w/data pack

Pesticide Mix A,
cat.# 32003, $30 each
32003-500, $60 ea. w/data pack
32103, $270 10pk. w/data pack

Pesticide Mix B,
cat.# 32004, $30 each
32004-500, $70 ea. w/data pack
32104, $270 10pk. w/data pack

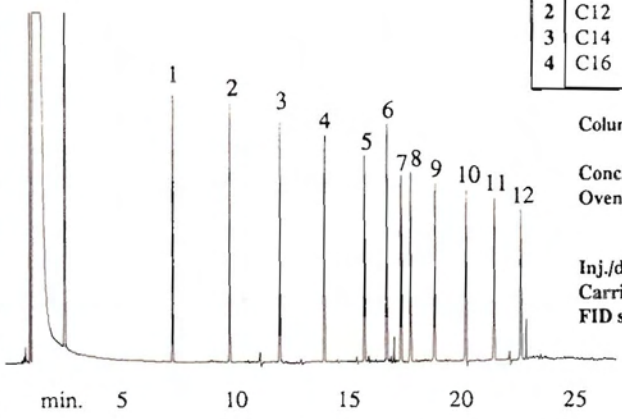
Pesticide Surrogate Mix,
cat.# 32000, $25 each
32000-500, $45 ea. w/data pack
32100, $225 10pk. w/data pack

For more information on ezGC™ Method Development Software, please call technical service at 1-800-356-1688, ext. 4.

Analysis of Diesel Range Organics in Soil and Water

In the March 1993 issue of *The Restek Advantage*, we published an article on the analysis of Gasoline Range Organics (GRO). This is a follow up article that focuses specifically on the analysis of Diesel Range Organics (DRO) and discusses how to avoid the common problems associated with this analysis.

Figure 1 - DRO individual component standards define the hydrocarbon retention time range and calibrate the instrument for DRO analysis.



1	C10	5	C18	9	C22
2	C12	6	*o*-terphenyl	10	C24
3	C14	7	C20	11	C26
4	C16	8	5-α-androstane	12	C28

Column:	30m, 0.53mm ID, 1.0µm Rtx®-5
Conc.:	50ng/µl
Oven temp.:	40°C (hold 2 min.) to 310°C @ 12°C/min. (hold 10 min.)
Inj./det. temp.:	280°C/310°C
Carrier gas:	helium @ 65cm/sec.
FID sens.:	8 x 10^{-11} AFS

As in GRO analysis, many states have adopted their own testing methodology for measuring DRO. In general, DRO methods measure the concentration of diesel fuel organics ranging from C10 - C28 in water and soil. DRO methods are designed to measure mid-range petroleum products such as diesel fuel, home heating oil, gas oil, jet fuel and kerosene. The typical DRO analytical method involves a liquid/liquid extraction with methylene chloride and Kuderna-Danish concentration of the extract. One microliter of the extract is then injected onto a megabore capillary column using a flame ionization detector (FID)[1]. Water samples are extracted as specified in SW-846 Method 3510 (separatory funnel liquid/liquid extraction) or Method 3520 (continuous liquid/liquid extraction). Soil samples are extracted using Method 3550 (sonication). The FID responds to all hydrocarbon species and is used to detect the total petroleum hydrocarbons (TPH). A wide range of columns can be used for DRO analysis, depending upon the requirements specified in each state's analytical procedure. In general, the column, operating under the conditions of the method, must meet some minimum requirements for retention and resolution.

To determine TPH, the sum of the area under a range of hydrocarbons is used. States may differ on which hydrocarbons are used to define the beginning and end of the diesel compounds to be measured, but typically begin with C10 or C12 and end with C24 or C28. Integration begins 0.1 minutes before the retention time of the first hydrocarbon marker and ends 0.1 minutes after the last hydrocarbon marker in the component standard. Most methods have specific performance requirements. Examples of method performance requirements include: 1) the beginning hydrocarbon must be completely resolved from the solvent peak or, 2) internal and surrogate standards must be resolved from the hydrocarbons. Most methods also employ a discrimination check to test for molecular weight discrimination. When analyzing such a wide range of hydrocarbons, molecular weight discrimination of the later eluting hydrocarbons is a common problem. Discrimination can occur from poor volatility in the injection port and/or syringe needle discrimination. Due to the higher hydrocarbon range and GC oven temperatures required for DRO analysis, a 30 meter, 0.53mm ID, 1.0µm Rtx®-5 is an excellent column to meet the resolution and performance requirements of the methods.

Determining diesel retention range and calibrating response

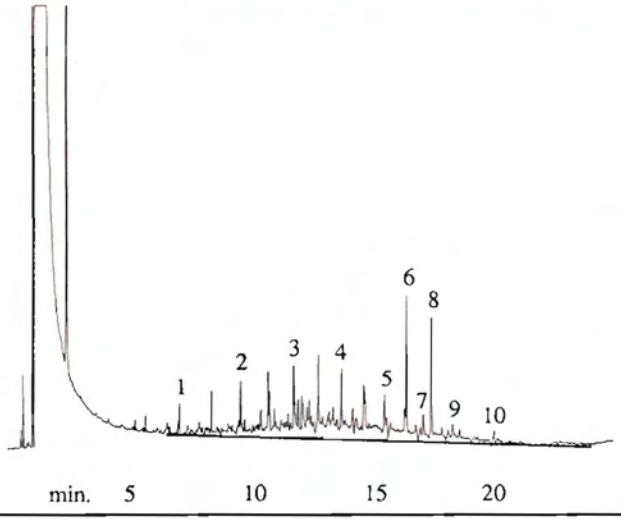
Since reporting TPH requires the summation of all chromatographic peaks eluting between the first and last component specified in the method, the calibration standard must contain the components defining the retention time range. Individual states differ on which compounds to use in defining the retention time range for diesel. Figure 1 shows a chromatogram of Restek's DRO individual component standard. This standard is used to determine the beginning and ending retention times of the diesel range. This standard is also used to calculate an average response factor by dividing the total area of all components by the total amount injected.

Confirmation of TPH response is achieved by analyzing a quantitative standard containing one or more diesel fuels. This composite standard was designed to be representative of diesels present in a wide range of samples to be analyzed. An example of a chromatogram generated from Restek's DRO composite diesel standard is shown in Figure 2. Once the beginning and ending retention times and response factors from the individual components have been defined, the total area of the composite standard is used to calculate the recovery for a spiked standard.

Improper integration can cause low DRO recoveries

Low recoveries, one common problem associated with DRO analysis, is usually due to errors in integration. The chromatographic profile of diesel oils does not give baseline resolution of all components, which results in a characteristic rising baseline underneath the resolved hydrocarbon components as shown in Figure 2. When calculating the recovery of DRO samples, integration should be accomplished using the baseline hold method. Baseline to baseline integration is performed by

Figure 2 - Composite diesel standards can be used to calculate TPH recovery.



1	C10
2	C12
3	C14
4	C16
5	C18
6	*o*-terphenyl
7	C20
8	5-α-androstane
9	C22
10	C24

Conc.: 500ng/µl
(same conditions as Figure 1)

obtaining the baseline at the beginning and end diesel retention markers and applying a horizontal hold between these two points. If valley to valley integration is used, low recoveries will result since a large portion of the total diesel response will be excluded from the integrated area.

High DRO recoveries can be caused by many factors

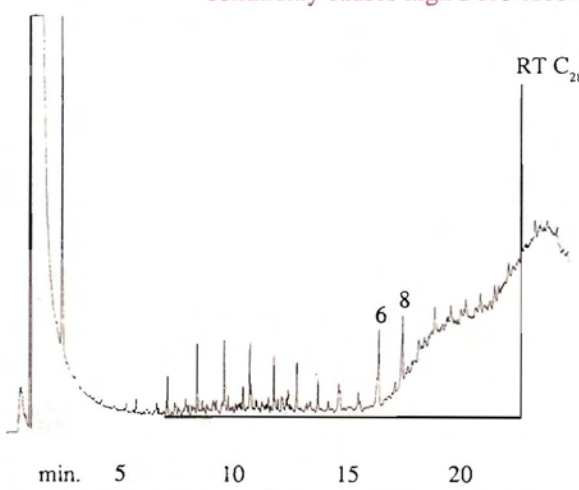
Another problem commonly seen in DRO analysis is erroneously high recoveries (>100%). One cause of high recovery is mass discrimination of high molecular weight hydrocarbons causing a low average response factor value. High recoveries can also be caused by changes in the baseline due to column bleed. Column bleed creates high DRO recovery since the background area under the diesel is increased when programming the GC oven to the higher temperatures required for DRO analysis. To minimize high recoveries from column bleed, perform a blank run prior to analysis to determine the true baseline. This true baseline can be subtracted from the DRO analysis to compensate for the additional area caused from normal column bleed.

High molecular weight interferences can create high DRO recoveries

Another source for reported high DRO recovery is from contamination. Contamination can occur from the presence of organic or high molecular weight contaminants, such as phenols, phthalates, lubricating oils or motor oils that were also extracted during the liquid/liquid extraction step. Figure 3 shows a chromatogram of a DRO sample that contains motor oil contamination. Most diesel range organics range from C10 to C28. In Figure 3, it appears that motor oil contamination is present with a hydrocarbon range beginning near C20 and overlapping beyond C28. If an analyst was to sum the area from the defined retention time marker of C10 to C28, an erroneously high DRO value would result. In the case of motor oil contamination, the analyst is required to make a judgment call, recognize the patterns of diesel fuel and motor oil, and determine a "cut point" for the DRO range.

Another common problem creating high DRO recovery or false positives is memory effect. Memory effects can be caused by poor sample volatility, backflash, or carryover. Poor sample volatility is caused when high molecular weight compounds are not completely vaporized and condense in the injection port. Figure 4 (page 6) shows an example of memory effect from a previous injection of high molecular weight hydrocarbons. To increase sample volatility, an injection port temperature of 280°C to 300°C is recommended. Backflash is created when the sample vaporization cloud exceeds the sample expansion volume of the inlet sleeve. Backflash can be minimized by increasing the inlet pressure, decreasing sample size, or diluting the sample. (Refer to our *Guide to Direct/On-column Flash Vaporization Injection* for more information on backflash.) Carryover is typically caused when sample residue is left behind after injecting a highly concentrated sample or from high molecular weight contaminants extracted during sample preparation. When running samples, always establish a sequence that injects from low to high concentration. If a sample has an unknown concentration, make a blank run after the sample to bake-out any potential contaminants. To minimize memory effects, prescreen extracts on a separate GC using a 15-meter Rtx®-5 column, prior to final sample preparation. If there is a high concentration of contaminants, the sample should be diluted before injecting it onto the column used for final quantitation.

Figure 3 - High molecular weight motor oil contamination commonly causes high DRO recovery.



1	C10
2	C12
3	C14
4	C16
5	C18
6	*o*-terphenyl
7	C20
8	5-α-androstane
9	C22
10	C24

Conc.: 500ng/µl
(same conditions as Figure 1)

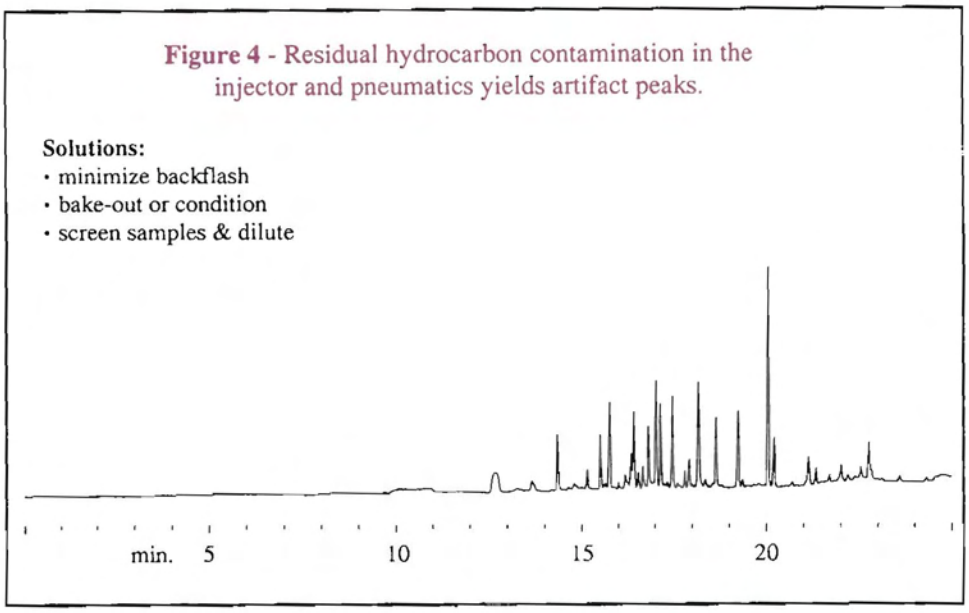


Figure 4 - Residual hydrocarbon contamination in the injector and pneumatics yields artifact peaks.

Although DRO methods may differ between states, the basic analytical procedures are similar. The recommended column for DRO analysis is a 30 meter, 0.53mm ID, 1.0µm Rtx®-5. The Rtx®-5 meets resolution requirements of DRO methods and minimizes baseline interferences from column bleed and mass discrimination. System calibration can be accomplished with mixtures of individual hydrocarbons or composite diesel standards. Baseline hold integration should be applied to yield accurate DRO recoveries and prevent low recovery. Baseline subtraction, sample prescreening, and column bake-out should be performed to minimize the potential of high recoveries. ■

1) EPA UST Workgroup American Petroleum Institute, February 1992.

Product Listing

Rtx®-5 30m, 0.53mm ID, 1.0µm, cat.# 10255, $445

Underground Storage Tank Monitoring Standards

DRO Mix (EPA/WISC)

2000µg/ml each in 1ml methylene chloride.

decane (C10)	dodecane (C12)
tetradecane (C14)	hexadecane (C16)
octadecane (C18)	eicosane (C20)
docosane (C22)	tetracosane (C24)
hexacosane (C26)	octacosane (C28)

Cat.# 31064 $25 ea.
31064-500 $55 ea. w/data pack
31164 $225 10 pk. w/data pack

Recommended internal standard: ***5-α-androstane Standard***

2000µg/ml in 1ml methylene chloride.

Cat.# 31065 $25 ea.
31065-500 $35 ea. w/data pack
31165 $225 10 pk. w/data pack

Recommended surrogate: ***o-terphenyl Standard***

2000µg/ml in 1ml acetone.

Cat.# 31066 $25 ea.
31066-500 $35 ea. w/data pack
31166 $225 10 pk. w/data pack

Diesel Surrogate Standards

Individual mixtures at 10,000µg/ml in 1ml methylene chloride.

compound	each	each w/data pack	10pk. w/data pack
p-terphenyl	31095/$25	31095-500/$35	31195/$225
2-fluorobiphenyl	31096/$25	31096-500/$35	31196/$225
o-terphenyl	31097/$25	31097-500/$35	31197/$225
1-chlorooctadecane	31098/$25	31098-500/$35	31198/$225

Diesel Fuel #2 Composite Standard

5000µg/ml in 1ml methylene chloride.

Cat.# 31093 $25 ea.
31093-500 $35 ea. w/data pack
31193 $225 10 pk. w/data pack

Kerosene Fuel Composite Standard

5000µg/ml in 1ml methylene chloride.

Cat.# 31094 $25 ea.
31094-500 $35 ea. w/data pack
31194 $225 10 pk. w/data pack

Jet Fuel A Standard

Commercial Jet Fuel A prepared at 5000µg/ml in 1ml methylene chloride.

Cat.# 31215 $25 ea.
31215-500 $35 ea. w/data pack
31315 $225 10 pk. w/data pack

Fuel Oil #4 Standard

Fuel Oil #4 is typically used in limited applications where preheating of the fuel prior to burning cannot be utilized. The fuel is a blend of distillate (Fuel Oil #2) and residual (Fuel Oil #6) to meet ASTM viscosity specifications. The Fuel Oil #4 standard offered here has a kinematic viscosity of 21.9 at 38°C (100°F). Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.# 31216 $25 ea.
31216-500 $35 ea. w/data pack
31316 $225 10 pk. w/data pack

Fuel Oil #5 Standard

Fuel Oil #5 is also typically used in applications where little or no preheating of the fuel prior to burning is utilized. As with Fuel Oil #4, Fuel Oil #5 is a blend of distillate and residual fuel to meet viscosity criteria. The Fuel Oil #5 standard offered here has a kinematic viscosity of 106.5 at 38°C (100°F). Kinematic viscosity measurement performed using ASTM D-445. Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.# 31217 $25 ea.
31217-500 $35 ea. w/data pack
31317 $225 10 pk. w/data pack

Fuel Oil #6 Standard

This oil, also called Bunker C or residual, is a black viscous fuel. Applications where it may be used all require the ability to preheat the fuel prior to pumping and burning. Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.# 31218 $25 ea.
31218-500 $35 ea. w/data pack
31318 $225 10 pk. w/data pack

(Product Listing is continued on page 7.)

Standards Spotlight

Product Update - USP 467 Calibration Mixture

In September 1991 Restek introduced a calibration mixture for USP Method 467, Organic Volatile Impurities (cat.# 36000). Since the initial introduction, the method has been modified several times to its current approved form (1). There are however, more recent modifications being proposed at this time (2).

In our continuing effort to provide quality chemical standards and meet the ever changing method requirements, we are now discontinuing the original USP 467 Calibration Mix (#36000) and introducing two new calibration mixtures for this protocol.

In the Fifth Supplement, USP-NF (1) the requirement to analyze for trace levels of ethylene oxide was removed from Method 467 and relegated to individual drug monographs. To meet the currently approved version of Method 467, we are offering a Revised USP 467 Mixture, cat.# 36001. Listed below are the components and concentrations of each analyte.

Revised USP 467 Mixture

Prepared in dimethyl sulfoxide, 1ml per ampul.

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

Cat.# 36001 **$20 ea.**
36101 **$180 per pack of 10 ampuls**

In the most recent edition of Pharmacopeial Forum (2), additional method modifications have been proposed. While the changes have not been formally adopted, laboratories may wish to begin evaluation of the new method. These proposed changes impact the calibration mixture required. The concentration of methylene chloride under consideration is five times higher than the original level in the current protocol (500ppm vs. 100ppm). In addition, the solvent used to prepare the stock calibration standard is now methanol instead of dimethyl sulfoxide. The stock calibration standard required to evaluate the proposed method revisions is listed.

Proposed USP 467 Mixture

Prepared in methanol, 1ml per ampul.

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

Cat.# 36002 **$20 ea.**
36102 **$180 per pack of 10 ampuls**

1) Fifth Supplement, USP-NF, Organic Volatile Impurities <467>, page 2706-2708, Nov. 15, 1991.

2) Pharmacopeial Forum, Volume 19, Number 3, May-June 1993, Organic Volatile Impurities <467>, page 5335-5337

(Product Listing continued from page 6.)

JP-4 Military Fuel Standard

Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.# 31219 **$25 ea.**
31219-500 **$35 ea. w/data pack**
31319 **$225 10 pk. w/data pack**

JP-5 Military Fuel Standard

Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.# 31220 **$25 ea.**
31220-500 **$35 ea. w/data pack**
31320 **$225 10 pk. w/data pack**

DRO Mix (Tenn/Miss)

Contains the following compounds at 1000µg/ml each in 1ml methylene chloride.

decane (C10)	undecane (C11)
dodecane (C12)	tridecane (C13)
tetradecane (C14)	pentadecane (C15)
hexadecane (C16)	heptadecane (C17)
octadecane (C18)	nonadecane (C19)
eicosane (C20)	heneicosane (C21)
docosane (C22)	tricosane (C23)
tetracosane (C24)	pentacosane (C25)

Cat.# 31214 **$30 ea.**
31214-500 **$70 ea. w/data pack**
31314 **$270 10 pk. w/data pack**

Clinical Corner

Quantitative Drug Standards

In an effort to continue meeting the varied demands of our customers, Restek introduces the availability of DEA exempt, quantitative drug standards. These standards can be used as qualitative retention time markers, reference material for spectral identification or for spiking samples for quantitative analysis. Each standard is prepared to the same exacting criteria that is used for our environmental standards.

All raw materials and final products are subject to exhaustive QA testing using DSC, GC/FID, GC/NPD, GC/MS, & FT-IR.

Comprehensive purity determinations
Our manufacturing process starts with extensive purity determinations for each raw material that goes into our standards. In order for raw materials to pass our quality assurance specifications, they must have a minimum purity of 98%. Drug raw materials are checked by two separate processes to verify purity. Differential Scanning Calorimetry (DSC) is used to assay the purity of all our crystalline materials. Determinations are accurate within 0.1 mole percent. Purity is also checked by performing GC-FID and GC-NPD analysis on all starting materials. These additional checks ensure that extraneous chromatographic peaks will not be present in the final product.

Final product testing
Identity confirmation is also an important part of our manufacturing process. All raw materials are checked by two separate methods to confirm their identity. Mass spectral identification is accomplished by using GC-MS and infrared spectra are taken using FT-IR. All spectra are compared to reference spectra published in either computer libraries or reference texts.

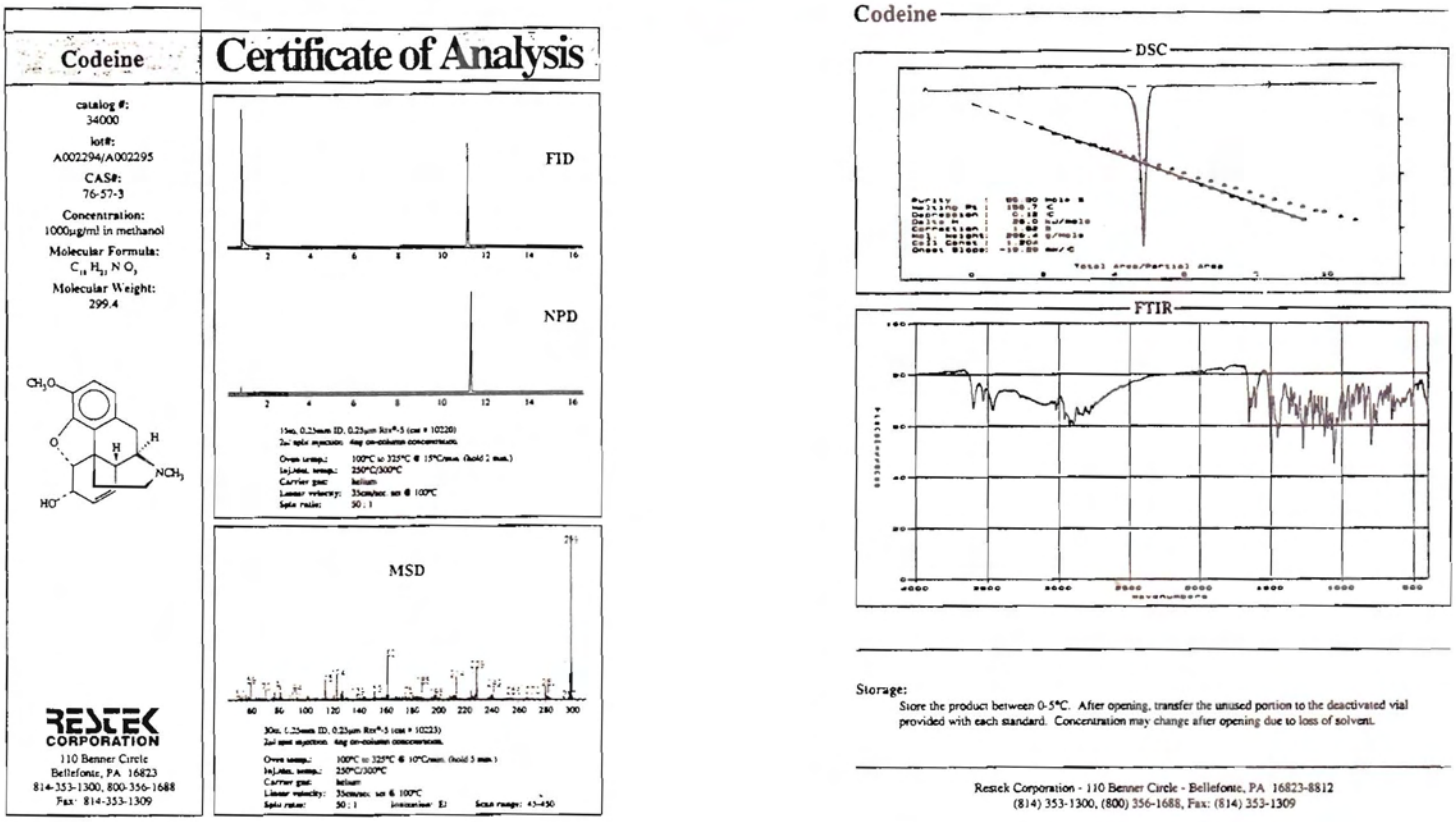
User friendly packaging
All of Restek's drug standards are provided as quantitative methanolic solutions. Every standard is prepared using precise gravimetric techniques after the raw materials have been approved for use. Subsequently, each lot of standard is analyzed by GC-FID using an internal standard and then compared to a reference lot of the same material in order to maintain reproducibility of concentration from lot to lot.

Restek's standards are packaged in flame sealed, silanized amber glass ampuls under nitrogen to enhance product stability prior to opening. Each ampul is supplied with an ampul breaker to safely open the ampule and a silanized amber screw top vial with label for storage of the unused portion of the standard.

Certificate of analysis
A Certificate of Analysis is supplied with each standard. This certificate includes compound structure, molecular weight and formula, DSC purity, GC-FID purity, GC-NPD purity, GC-MS and FTIR identification. The concentration as stated on the certificate is calculated from the gravimetric data. Concentrations reported are based on the weight of the free acid or free base form of the compound, with corrections being made for the salt form of the raw material. A sample of the Certificate of Analysis is shown in Figure 1.

DEA exempt
These standards have been exempted by the Drug Enforcement Administration from the normal requirements for purchasing controlled substances. They can be obtained without a DEA license, phone orders can be accepted, and no additional paperwork is required with your order. ■

To order any Restek product, call 800-356-1688 (ext. 3). For direct technical service, call 800-356-1688 (ext. 4).



All standards are diluted in methanol

Description	Concentration	Cat.#	Price	Description	Concentration	Cat.#	Price
alprazolam	1000µg/ml	34042	$25	lorazepam	1000µg/ml	34051	$25
amobarbital	1000µg/ml	34028	$25	medazepam	1000µg/ml	34052	$25
dextro-amphetamine	1000µg/ml	34020	$25	meperidine	1000µg/ml	34004	$25
aprobarbital	1000µg/ml	34029	$25	mephobarbital	1000µg/ml	34034	$25
barbital	1000µg/ml	34030	$25	meprobamate	1000µg/ml	34059	$25
benzoylecgonine	1000µg/ml	34016	$30	methadone	1000µg/ml	34005	$25
benzphetamine	1000µg/ml	34022	$25	methamphetamine	1000µg/ml	34021	$25
bromazepam	1000µg/ml	34043	$25	methaqualone	1000µg/ml	34064	$25
butabarbital	1000µg/ml	34031	$25	methohexital	1000µg/ml	34035	$25
butalbital	1000µg/ml	34032	$25	methyprylon	1000µg/ml	34060	$25
cannabidiol	1000µg/ml	34011	$25	morphine	1000µg/ml	34006	$25
cannabinol	1000µg/ml	34010	$30	nitrazepam	1000µg/ml	34053	$25
chlordiazepoxide	1000µg/ml	34044	$25	oxazepam	1000µg/ml	34054	$25
clobazam	1000µg/ml	34045	$25	oxycodone	1000µg/ml	34007	$25
clonazepam	1000µg/ml	34046	$25	oxymorphone	1000µg/ml	34065	$25
cocaethylene	1000µg/ml	34019	$35	pentazocine	1000µg/ml	34062	$25
cocaine	1000µg/ml	34015	$25	pentobarbital	1000µg/ml	34036	$25
codeine	1000µg/ml	34000	$25	phencyclidine	1000µg/ml	34027	$30
desmethyl diazepam	1000µg/ml	34048	$30	phendimetrazine	1000µg/ml	34025	$25
diazepam	1000µg/ml	34047	$25	phenmetrazine	1000µg/ml	34026	$25
ecgonine	1000µg/ml	34017	$30	phenobarbital	1000µg/ml	34037	$25
ecgonine methyl ester	1000µg/ml	34018	$35	phentermine	1000µg/ml	34024	$25
fenfluramine	1000µg/ml	34023	$25	prazepam	1000µg/ml	34055	$25
flunitrazepam	1000µg/ml	34049	$25	dextro-propoxyphene	1000µg/ml	34008	$25
flurazepam	1000µg/ml	34050	$25	secobarbital	1000µg/ml	34038	$25
d,l-glutethimide	1000µg/ml	34058	$25	talbutal	1000µg/ml	34039	$25
hexobarbital	1000µg/ml	34033	$25	temazepam	1000µg/ml	34056	$25
hydrocodone	1000µg/ml	34002	$25	thebaine	1000µg/ml	34009	$25
hydromorphone	1000µg/ml	34063	$25	thiamylal	1000µg/ml	34040	$25
levorphanol	1000µg/ml	34003	$25	thiopental	1000µg/ml	34041	$25
				triazolam	1000µg/ml	34057	$25

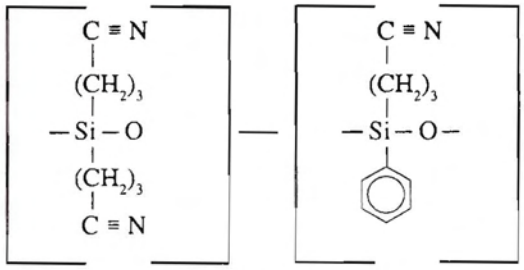
Improved Rtx®-2330 Capillary Columns

New Surface Treatment and Advanced Polymer Technology Improve the Quality and Longevity of Rtx®-2330 Capillary Columns

One of the most polar capillary column stationary phases is a 90% biscyanopropyl/ 10% phenylcyanopropyl polysiloxane or Rtx®-2330. Because of the presence of the cyano groups in the polymer backbone, the Rtx®-2330 stationary phase possesses a strong dipole moment and is highly selective for cis/trans compounds or compounds with conjugated double bonds. The Rtx®-2330 is widely used for analyzing fatty acid methyl esters (FAMEs), cis/trans isomers, rosin acids, and TCDD isomers.

Figure 1 - Rtx®-2330 stationary phase structure and composition.

90% biscyanopropyl - 10% phenylcyanopropyl polysiloxane



Polarity: very polar
Uses: FAMEs, cis/trans & dioxin isomers
Temp. limits: 0°C/275°C

Despite the high utility of biscyanopropyl/ phenylcyanopropyl phases, they typically suffer from poor column efficiencies, higher bleed, and short column lifetimes when thermally cycled. These problems result from difficulties in getting the high cyano content stationary phases to coat uniformly on a smooth fused silica surface. To overcome these limitations, Restek's chemists have developed a new surface treatment that is more compatible with the Rtx®-2330 phase. In addition, they synthesized an advanced polymer that is more compatible with this surface to produce columns with improved column efficiency and lower bleed. The combination of the new surface treatment and advanced polymer technology results in enhanced column lifetimes.

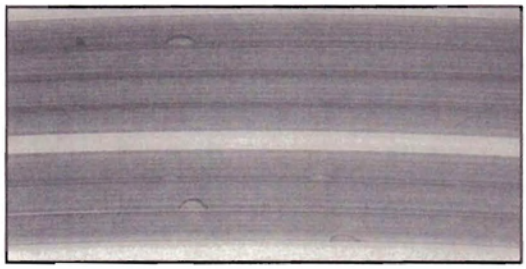
Common problems associated with biscyanopropyl/ phenylcyanopropyl columns

The Rtx®-2330 stationary phase is extremely polar because of the high percentage of cyano groups in the polymer backbone. Figure 1 shows the structure and composition of the Rtx®-2330 phase. The high concentration of cyano groups hinders crosslinking and prevents complete crossbonding of the stationary phase to the fused silica surface. Therefore, Rtx®-2330 columns cannot be solvent rinsed. Because of incomplete crossbonding, high cyano content stationary phases have been known to thermally rearrange (puddle) and lose efficiency rapidly while the oven is being cycled up and down in temperature. At high GC oven temperatures (> 275°C), cyano polymers become very fluid and lose critical viscosity leading to high bleed levels, retention time shifts, and loss of resolution for critical pairs.

Surface chemistry and polymer composition greatly affect column quality and lifetime

Restek's chemists have developed a surface layer that properly matches the critical surface tension of the 2330 phase. If the surface layer is not completely compatible in polarity with the stationary phase, the phase will "puddle"or bead up after repeated thermal cycling. If puddling occurs, a column will exhibit poor efficiency and high bleed. The photo below shows phase puddling due to thermal rearrangement. The dark spot on the tubing indicates where stationary phase has beaded up on the column surface.

Finding the optimum surface layer for a stationary phase is one of the most important steps in capillary column manufacturing. The compatibility of the surface layer with the stationary phase dramatically affects the column's efficiency, thermal stability, and longevity.



An incompatible deactivation layer prevents uniform stationary phase coating and can create phase puddling on the column's surface.

In combination with the new surface layer, we have synthesized an advanced polymer that resists rearrangement and puddling. The new polymer has the same polarity as the original polymer, but crosslinking mechanisms have been enhanced to increase column longevity. The end result is an improved Rtx®-2330 capillary column that exhibits enhanced column efficiency, thermal stability, and column lifetime.

Unique selectivity of the Rtx®-2330 is ideal for a wide variety of applications

Because of the high content of cyano groups in the Rtx®-2330 polymer backbone, it is very selective for a wide variety of applications. In the food industry, many laboratories are involved in the analysis of fatty acids to comply with the new labeling regulations.

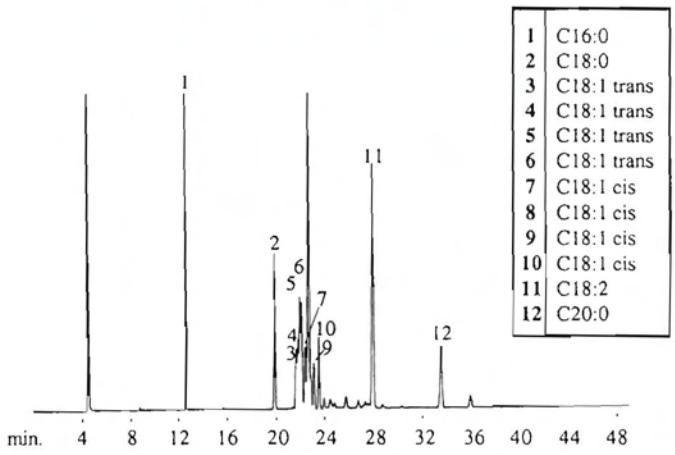
Partially hydrogenated oils contain geometric and positional isomers of unsaturated fatty acids. Food chemists who analyze these complex mixtures of FAME cis/trans isomers often have

difficulty obtaining the resolution needed for this analysis. Figure 2 shows the analysis of commercial margarine on a 105m, 0.25mm ID, 0.20µm Rtx®-2330 and the resolution obtained of the C18:1 cis/trans isomers on this phase.

EPA Methods 513 and 613 are capillary GC/ECD and GC/MS methods developed for monitoring the highly toxic 2,3,7,8 isomer of tetrachlorodibenzo-p-dioxin (TCDD). The Rtx®-2330 phase is the primary column recommended by the EPA for Methods 513 and 613 since it can resolve 2,3,7,8-TCDD from the other isomers present in TCDD. Figure 3 shows the analysis of TCDD on a 60m, 0.25mm ID,0.20µm Rtx®-2330 by GC/ECD. Baseline resolution of the 2,3,7,8-TCDD isomer and low column bleed is achieved on this column.

Improvements in the Rtx®-2330 surface layer and polymer technology yield capillary columns with enhanced column efficiencies and increased longevity. Analysts should expect better peak resolution, lower bleed, and longer column life-times with the improved Rtx®-2330 columns.

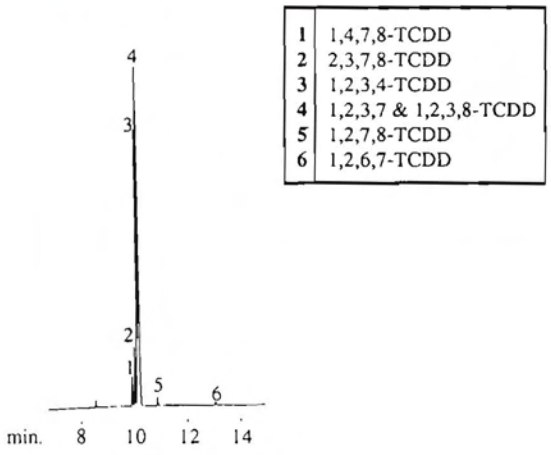
Figure 2 - Resolve C18:1 cis/trans isomers in margarine using the Rtx®-2330.



105m, 0.25mm ID, 0.20µm Rtx®-2330 (cat.# 10729)
1.0µl split injection of a commercial margarine mixture.
Concentration approximately 5µg.

Oven temp.: 165°C isothermal
Inj. & det. temp.: 275°C
Carrier gas: hydrogen
Linear velocity: 40cm/sec.
FID sensitivity: 4 x 10^{-11} AFS
Split ratio: 50:1

Figure 3 - Achieve TCDD isomer separation on a 60m, 0.25mm ID, 0.20µm Rtx®-2330.



60m, 0.25mm ID, 0.20µm Rtx®-2330 (cat.# 10726)
2.0µl splitless injection of TCDD isomers.
Concentration approximately 0.5µg of each isomer.

Splitless hold: 0.45 min.
Oven temp.: 200°C (hold 1 min.) to 250°C @ 8°C/min. (hold 15 min.) to 275°C @ 15°C/min. (hold 5 min.)
Inj. & det. temp.: 275°C **Carrier gas:** hydrogen
Linear velocity: 40cm/sec.
ECD sensitivity: 21kHz full scale

Product Listing

Rtx®-2330* (90% biscyanopropyl - 10% phenylcyanopropyl polysiloxane) Polymer stable to 275°C

	df (µm)	temp. range	15-Meter		30-Meter		60-Meter		105-Meter	
0.25mm ID	0.10	0 275°C	10705	$225	10708	$380	10711	$665	10714	$870
	0.20	0 275°C	10720	$215	10723	$370	10726	$655	10729	$850
0.32mm ID	0.10	0 275°C	10706	$245	10709	$410	10712	$725	10715	$945
	0.20	0 275°C	10721	$235	10724	$400	10727	$715	10730	$925
0.53mm ID	0.10	0 275°C	10707	$250	10710	$455	10713	$820		
	0.20	0 275°C	10722	$240	10725	$445	10728	$795		
0.18mm ID	df (µm)	temp. range	10-Meter		20-Meter		40-Meter			
	0.10	0 275°C	40701	$200	40702	$320	40703	$595		

Caution: As with all high cyano content stationary phases,the analysis of acids, alcohols, and diols may show poor reproducibility due to an interaction of the cyano groups & column surface with these compounds.
*Not solvent rinsable.

Hints for the Capillary Chromatographer

The Importance of Inlet Sleeve Deactivation

Chromatographers frequently call with questions about sleeve deactivation. Due to the popularity of this topic, we have decided to rerun the article on "The Importance of Inlet Sleeve Deactivation" from September 1990's issue of The Restek Advantage.

Your entire sample pathway must be inert

Capillary columns are inherently more inert than packed columns. However, capillary chromatographers still experience breakdown and adsorption of active compounds. Inertness of the sample pathway is particularly important with certain pesticides, herbicides, drugs, amines, and acids. If the column itself is inert, then what is the source of the problem? One major area that most chromatographers overlook is the inlet. The inertness of the inlet sleeve is very important since it is part of the sample pathway and can often be the source of adsorption or breakdown. If the sleeve is not deactivated properly, adsorption can take place in the sleeve and alter the composition of the sample reaching the column. Also, when analyzing compounds at low levels, adsorption or decomposition of some components in the sleeve results in poor quantitation and even misidentification.

Is it important to deactivate the sleeve?

For most samples, the answer to this question is a resounding yes. We tested the adsorptive characteristics of an undeactivated versus a deactivated sleeve (Figure 1). Endrin, an active pesticide, was injected into an untreated and a properly deactivated inlet sleeve. The undeactivated sleeve showed 98% breakdown of a 50pg injection of endrin into its respective degradation products, endrin aldehyde and endrin ketone. The deactivated sleeve showed only 6% breakdown.

In addition to breakdown, an improperly deactivated sleeve can cause irreversible adsorption. To demonstrate this effect, we compared the response factors of three active compounds, 2,4-dinitrophenol, pentachlorophenol, and benzidine on both undeactivated and properly deactivated sleeves. Table I shows that the response factors for all three compounds are lower on the undeactivated sleeve than on the deactivated sleeve.

Table I

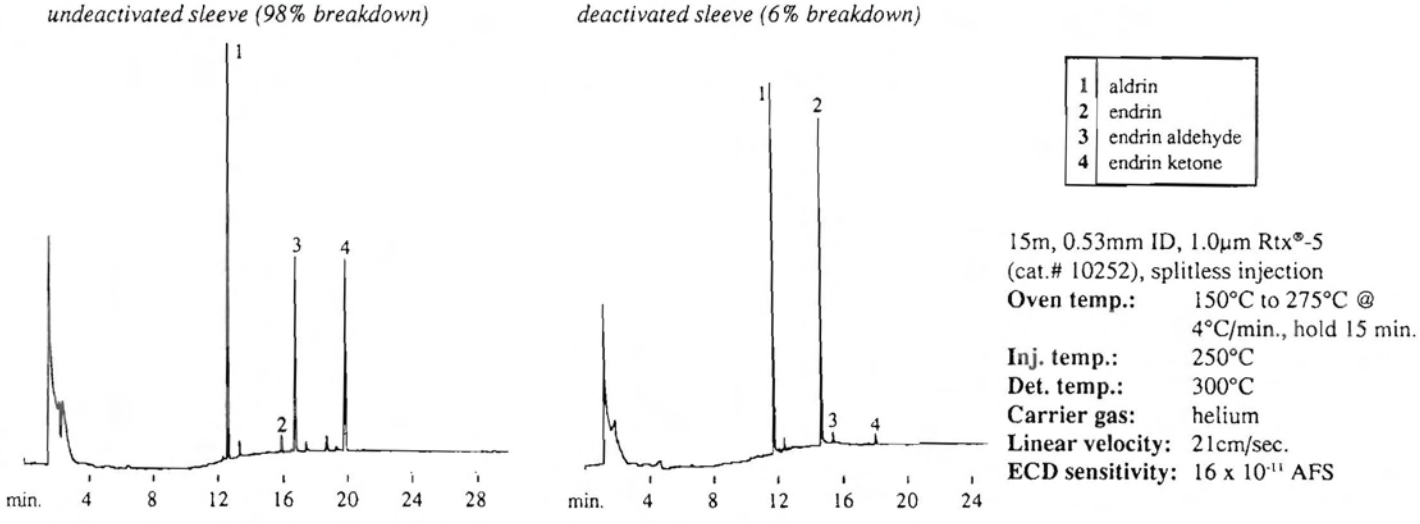
Average response factors relative to naphthalene of 3 injections.

	2,4-dinitrophenol	pentachlorophenol	benzidine
deactivated	0.248	0.240	0.327
undeativated	0.185	0.188	0.234

Why do response factors change for active compounds?

A priming effect occurs when improperly deactivated sleeves are used for adsorptive samples. We injected 1.0µl of an endrin standard onto a column and calculated the percentage of breakdown. This first injection primed the sleeve with endrin. When we injected the endrin standard onto the column again, the result was less endrin breakdown. This procedure can be

Figure 1 - Comparison of endrin breakdown on an undeactivated and a deactivated Cyclosplitter®.



repeated until the percentage breakdown remains constant for three injections. If you are running calibration curves and your numbers are jumping around in this manner, your inlet sleeve may not be adequately deactivated.

How do you know that your sleeve has been properly deactivated?

The only true test of a sleeve's inertness is in its actual performance with active compounds. A visual inspection of the sleeve will not tell you that the sleeve has been deactivated. In fact, most instrument manufacturers do not deactivate their inlet sleeves.

Are you using a home-brewed sleeve deactivation recipe?

Many chromatographers are aware that their sleeves are not deactivated and develop many different recipes to treat their sleeves. The most common treatment is with dimethyldichlorosilane (DMDCS). The use of this material presents several problems. It reacts with moisture to produce HCl vapors, so treatments with DMDCS must be done in a well-ventilated area. Also, DMDCS, if not properly end capped, can react with humidity in air, resulting in weakened effectiveness.

We examined the quality of in-house deactivation procedures by comparing several home-brew recipes commonly used. We looked at acid treatment, DMDCS treatment, acid and DMDCS treatment, and finally our own proprietary deactivation procedure. We evaluated each of these procedures in duplicate using a Cyclosplitter®. The resulting endrin breakdown from these five treatments is shown in Table II. The results clearly show that all deactivation techniques are not equal and a highly inert surface requires a procedure combining both acid and DMDCS treatments.

Table II

% endrin breakdown with various deactivations

Treatment	Run #1	Run #2
Acid only	36%	45%
DMDCS only	20.8%	18.3%
Acid and DMDCS	19.8%	18.5%
Restek's Old Deactivation*	10.0%	15%
Restek's New Deactivation	2.2%	1.2%

% endrin breakdown calculation:

Σ areas = endrin area + endrin aldehyde area + endrin ketone area

$$\% \text{ total breakdown} = 100 \times \frac{\text{aldehyde area + ketone area}}{\Sigma \text{ area}}$$

* Prior to April 1991.

Will glass wool or beads in your sleeve cause adsorption and breakdown?

Yes. Many chromatographers who analyze dirty samples use a plug of glass wool or beads in their sleeve to act as a filter. However, both wool and beads greatly increase the surface area that the sample contacts and can be a source of adsorption or breakdown. It is critical that the wool or beads be properly deactivated. Even if your wool is deactivated, active sites can be created as the fibers break when inserting the wool into the sleeve. If you plan to use wool, be sure that it is thoroughly deactivated and use care when inserting it into the sleeve.

Can sleeves be cleaned without redeactivation?

Sleeves only need to be redeactivated if the deactivation layer is removed during cleaning. Daily GC use deposits septa particles, sample residue, and pyrolizates on the sleeve surface, contaminating your inlet. One way to prolong the lifetime of your deactivated sleeve is to wash out the sleeve with methanol (since methanol does not swell septa or make them sticky). Nylon tube brushes and pipe cleaners are also helpful for removing small septa fragments from dirty sleeves. *However, be careful to avoid scratching the liner with the inner metal core of the brush.* You can then use stronger solvents such as methylene chloride, hexane, or the solvent your sample is diluted in, to remove additional contamination. These mild procedures will not require redeactivation.

If the contamination has been pyrolized onto the sleeve, solvent rinsing or other simple cleaning procedures may still leave a residue. The only effective procedures to remove this residue are strong acids, bases, detergents, or baking in a muffle furnace. If you use these harsh procedures, you will remove the deactivation layer, requiring redeactivation.

Analyzing active compounds is a difficult assignment. If you properly deactivate your inlet sleeve you will find many of your analysis problems solved. For more information, please call our technical service group at 800-356-1688, ext. 4. ■

Suggestions?

Is there a topic you would like to see covered in "Hints for the Capillary Chromatographer"? If so, please call our technical service department toll-free at 800-356-1688 (ext. 4).

Peak Performers

Now available from Restek...

Hoke Toggle, Ball, and Plug Valves

Restek now offers Hoke toggle, ball, and plug valves. These high quality, precision machined valves are available in 1/8" and 1/4" sizes in both brass and stainless steel. The toggle valves are ideal for applications when instant on/off control is necessary. Hoke toggle valves are rated to 200psig at 21°C and have a maximum operating temperature of 149°C. The forged body ball valves are ideal for high pressure applications. Hoke ball valves have a floating ball to assist in sealing and reduce operating torque and dual seats provide leak-tight bidirectional sealing. They are rated to 6000psig at 21°C and have a maximum operating temperature of 249°C. The plug valves are ideal for applications where throttling or on/off operations are required. Hoke plug valves have dual retaining rings to prevent accidental plug removal. They are rated to 3000psig at 21°C and have a maximum operating temperature of 205°C.

	Brass		Stainless Steel	
Valve type	1/8"	1/4"	1/8"	1/4"
Toggle	21885	21886	21985	21986
	$36	$33	$85	$80
Ball	21887	21888	21987	21988
	$50	$50	$90	$88
Plug	21889	21890	21989	21990
	$48	$44	$70	$68

Miniature Leak Detector

Leaks in a gas chromatographic system can cause problems ranging from increased detector noise, baseline instabilities, and short column lifetime, to wasting expensive carrier gases. GL Sciences' new portable Leak Detector LD-223 allows analysts to detect minute gas leaks undetected by liquid leak detectors. This leak detector's compact size is designed for easy transport and hand-held usage. Simple, push button operation allows one-touch zero adjustment, while the low dead volume sampling line provides quick sample response. Trace leaks of both helium and hydrogen* can be detected using the high sensitivity range. Four AA alkaline batteries (not included) provide up to 12 hours of continuous operation.

- Portable size
- Quick response
- High sensitivity
- Simple operation
- Contamination-free leak detection

GL Sciences Leak Detector: cat.# 21605, $950 each

* not designed for use in explosive atmospheres.

Don't miss this new wizard dollar item...

Wizard Pitcher & Glasses Set

We have added another premium gift to our Wizard Dollar promotional program. Now available is a wizard pitcher and glass set. This set contains one 55 ounce glass pitcher and eight 13 ounce beverage glasses for just 25 Wizard Dollars.

If you are not saving your Restek Wizard Dollars, you are missing out on valuable FREE gifts. For more information on this program, call 800-356-1688, ext. 3 to request a copy of the Wizard Dollar Promotional Flyer.

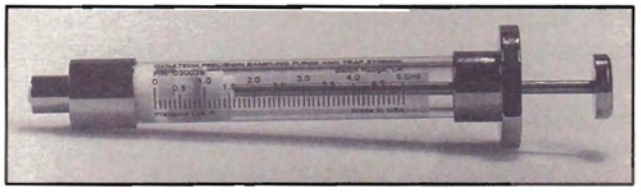
Wizard Pitcher & Glasses set, cat.# 60028, $25 wizard dollars

To order any Restek product, call 800-356-1688 (ext. 3).

Heavy Duty Purge and Trap Syringe

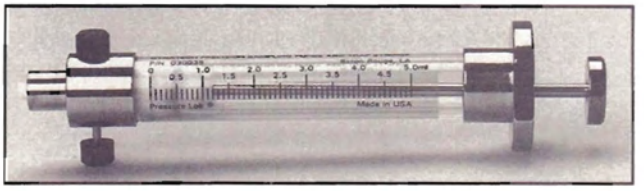
Designed by Dynatech Precision Sampling Corporation for filling and emptying sparge tubes on purge and trap concentrators, these new purge and trap syringes offer the analyst special features for purge and trap.

- Teflon® Luer-lock tip
- Heavy duty glass barrel and metal front and rear flanges reduce likelihood of breakage if dropped.
- Pressure-Lok plunger tip



Without Sample Lok

Size	2ml	5ml	10ml
Catalog number	21205	21206	21209
Price	$60	$60	$60



With Sample Lok

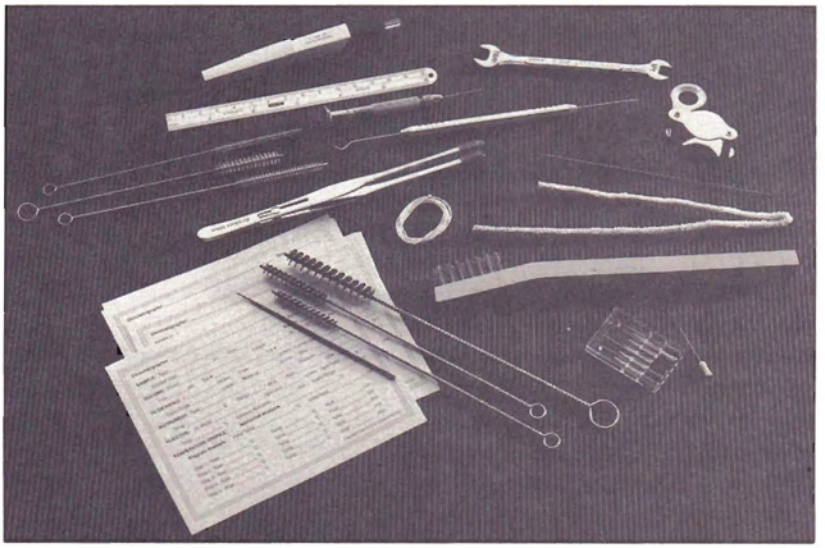
Size	5ml	10ml
Catalog number	21208	21207
Price	$75	$75

The MLE Tool Kit Contains a Variety of Tools to Make Your Life Easier!

This kit contains all the tools necessary for installing and maintaining your capillary columns.

The MLE Kit includes:

- Rubber-tipped Slide-Lok tweezers
- 15cm compact steel ruler
- Sapphire scribe
- Pocket magnifier
- Pin vise with three drills (0.41, 0.51, 0.8mm)
- Four-inch tapered needle file
- Six stainless steel jet reamers (0.25 - 0.65mm)
- Five self-adhesive capillary column labels
- Septum puller
- Three nylon brushes ($^1/_8$", $^3/_{16}$", $^1/_4$")
- Pipe cleaner (one-foot)
- $^1/_4$" - $^5/_{16}$" wrench
- One-meter of high temperature string (400°C)
- Three stainless steel brushes ($^3/_{16}$", $^1/_4$", $^3/_8$")
- Stainless steel toothbrush
- Glass wool puller/inserter
- Fused silica column installation guide
- Capillary reference poster



MLE Tool Kit: cat.# 20118, $119 each

News from Restek

Restek Celebrates 8th Anniversary

October 1993 marks Restek's eighth anniversary. Since 1985, we have grown to over 80 employees and two locations, Bellefonte and Germany. Another major building expansion is planned for next spring to give us more room for our existing departments.

Special thanks go out to our loyal customers, suppliers, and distributors who have helped to make our rapid success possible. The Restek Wizards are looking forward to future challenges and your continued support.

It's not too late to sign up for Restek's Capillary Chromatography Seminars!

Restek's 5th seminar tour begins on September 13 in Sacramento, California and Seattle, Washington. The tour will continue with presentations in 52 cities throughout the United States. This educational course has been updated for 1993 and includes sections on capillary theory & terminology, inlet systems & injection techniques, column selection, column installation, and maintenance & troubleshooting. If you would like more information about our seminar series, call 800-356-1688, ext. 3.

Upcoming Restek Exhibits:

ALEX
October 5-7 in San Francisco, CA (Booth # 115)
Paper Presentations: "Efficient GC Method Development Using Thermodynamic Retention Modeling"

Society of Forensic Toxicologists (SOFT) Meeting
October 13-15 in Pheonix, AZ (Booth # 26)
Paper Presentations: "Optimization of Drug Analysis Using Gas Chromatography & Thermodynamic Retention Times"

Eastern Analytical Symposium (EAS)
November 15-19 in Somerset, NJ (Booth # 233)
Short Course: Advanced Gas Chromatography

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THE RESTEK ADVANTAGE

NEW! Columns for Blood Alcohol Analysis

Faster Analysis Time and Baseline Resolution

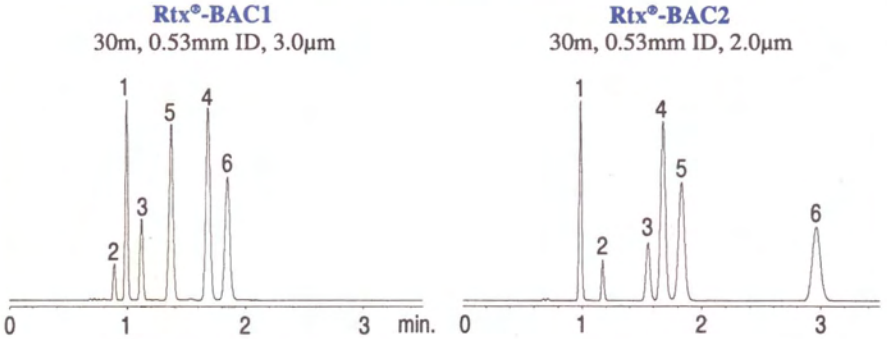
Testing for the presence and subsequent quantitation of ethanol in blood, breath and urine are the highest volume tests performed in forensic laboratories. In addition to ethanol, the detection of several other significant alcohols and their metabolites is necessary. Gas chromatographic assays provide the greatest amount of flexibility and specificity in analyzing for these volatile compounds. Headspace sampling is preferred over direct injection since it eliminates the build-up of non-volatile contamination at the head of the column.

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 maintaining baseline resolution for all analytes. Isothermal analysis is the method of choice because it eliminates the cooling down period between temperature programmed runs. Overall analysis time can be reduced in isothermal analyses by raising the temperature of the analysis or by increasing carrier gas flow rate. However, in attempting to shorten the analysis time, either by increasing the flow rate or by raising the temperature, many traditional capillary stationary phases fail to provide adequate resolution of all of the components commonly tested in blood alcohol analyses.

Figure 1 shows the analysis of low molecular weight alcohols and their metabolites on two new stationary phases developed specifically for blood alcohol analysis. These phases yield baseline resolution with very short analysis times for all of the compounds while providing elution order changes for four out of six compounds. These changes in elution order and retention

in this issue...

Figure 1 - Achieve baseline resolution of blood alcohols using dual columns in less than 3 minutes.

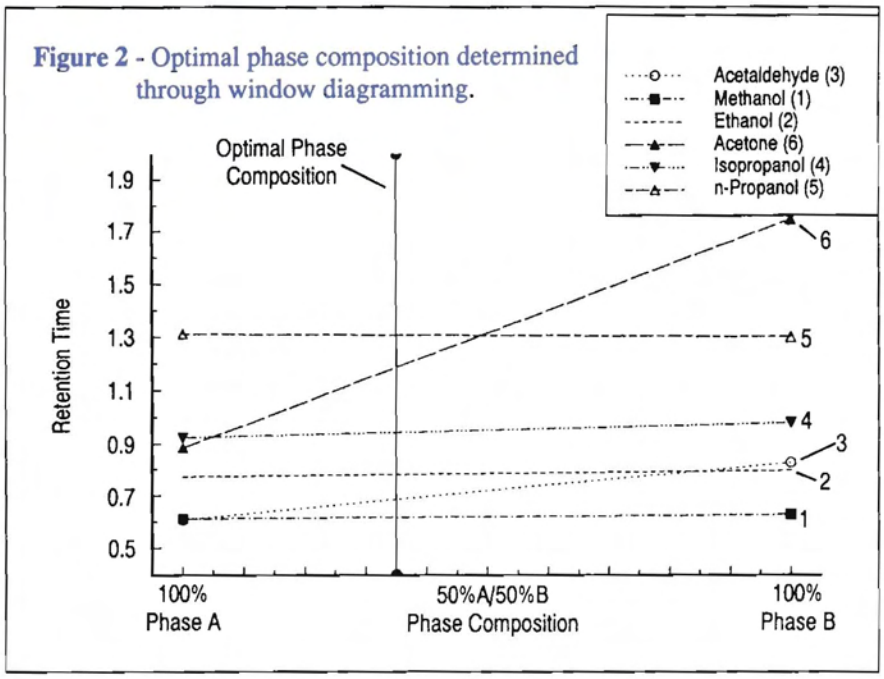


1.0ml headspace sample of Blood Alcohol Mix
Oven temp.: 40°C isothermal
Inj. & det. temp.: 200°C
Carrier gas: helium
Linear velocity: 80cm/sec. set @ 40°C
FID sensitivity: 1.28 x 10^{-10}AFS

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

Chromatograms produced using a model 7000 static headspace autosampler on loan courtesy of The Tekmar Company.

Figure 2 - Optimal phase composition determined through window diagramming.



time can be used as a confirmational tool in identifying the presence or absence of volatile compounds in forensic specimens. Dual column confirmational analyses significantly reduce the chance of false negative or false positive results.

See Hints for the Capillary Chromatographer on Performing Dual Column Analysis (pages 10 and 11)

Resolution between critical components and speed of analysis were finely tuned by altering the molar composition of certain functional groups in the stationary phase. By using the system of column development demonstrated by Mehran et al [1,2], two columns with dissimilar stationary phases were coupled together to evaluate changes in resolution for this group of six compounds. Window diagramming was then used to predict optimal stationary phase composition from a minimal number of experiments. In Figure 2, a plot of retention time versus stationary phase composition shows the phase compositions that should be able to perform the separation. In our experiments, four different functional groups were evaluated for their ability to alter specific separations. By combining two or more of these functional groups into a single, homogeneous stationary phase, complete resolution of all components can be achieved.

Optimal performance of these columns during headspace analysis depends on GC system set up. Band broadening can occur if there is excess dead volume in the sample flow path from the sample valve to the head of the column. Low volume sleeves or interfaces in the injection port significantly reduce the amount of excess volume at the exit end of the transfer line. Carrier gas flow rate through the sample loop and transfer line is also important in maintaining a narrow sample band width prior to the sample reaching the head of the column. Our experiments showed that a high flow rate of 30ml/min. was most effective in transferring the sample from the headspace unit to the column in a tight sample band. This flow was subsequently split between two columns by using a Universal "Y" Press-Tight® connector to provide simultaneous analyses from the same headspace sample.

The inclusion of multiple functional groups in stationary phases can significantly alter the retention characteristics for specific compounds. Restek's BAC columns incorporate functional groups into the stationary phase that have selective retention mechanisms for alcohols, ketones and aldehydes. Baseline resolution and elution order changes as well as short analysis time can be achieved for the analysis of blood alcohols.

References

1. M. F. Mehran, W. J. Cooper and W. Jennings, *Journal of High Resolution Chromatography and Chromatography Communications*, Volume 7 (1984) 215.

2. M. F. Mehran, W. J. Cooper, R. Lautamo, R. R. Freeman, and W. Jennings, *Journal of High Resolution Chromatography and Chromatography Communications*, Volume 8 (1985) 715.

Product Listing

Rtx®-BAC1
30m, 0.53mm ID, 3.0µm
cat.# 18001

Rtx®-BAC2
30m, 0.53mm ID, 2.0µm
cat.# 18000

Universal "Y" Press-Tight® Connector
cat.# 20405 each
cat.# 20406, 3-pack

Universal Angled "Y" Press-Tight® Connector
cat.# 20403 each
cat.# 20404, 3-pack

5m x 0.53mm ID Guard Column
cat.# 10045 each
cat.# 10045-600, 6-pk.

Ambient Air Analysis According to EPA Method TO-14

Since the Clean Air Act was introduced in 1990, much research has been done in developing methods for the analysis of Hazardous Air Pollutants (HAPs). One of the most common analytical methods for HAPs analysis is EPA compendium method TO-14. This method is designed for measuring relatively nonpolar volatile organic compounds. Method TO-14 involves sampling the air with a stainless steel SUMMA® canister followed by cryogenic sample preconcentration. The sample is then transferred to a gas chromatograph, analyzed by high resolution capillary gas chromatography and measured with a sensitive detector such as a mass spectrometer.[1]

There has been an increasing interest in analyzing polar volatile organics, in addition to the TO-14 compound list. Many of these polar compounds are chemically reactive and are commonly found at trace levels in air. Such compounds originate from a variety of industrial processes, automotive emissions, and consumer products, and some may be formed in the atmosphere by photochemical oxidation of hydrocarbons.[2] The measurement and detection of these compounds is very difficult and the analytical methods have not been completely developed.

Figure 1 shows the analysis of a calibration mix of the polar and non-polar volatile organic compounds (VOCs) commonly found in air. The configuration used for this analysis involved sample preconcentration using a Tekmar AEROCAN™ 6000 and a glass bead trap cryogenically cooled to -165°C. The sample was desorbed and secondarily focused at -175°C on the head of a 60m, 0.32mm ID, 1.8µm Rtx®-502.2 capillary column using a cryofocusing module. Due to the volatility of these compounds a long length, thick film capillary column must be used. The Rtx®-502.2 stationary phase is specifically designed for the analysis of volatile organics in water, but is also an excellent choice for the analysis of VOCs in air.

Both polar and nonpolar volatiles can be effectively monitored using a 60m, 0.32mm ID, 1.8µm Rtx®-502.2 capillary column. This long length, thick film column provides the necessary resolution of VOCs in air as described in EPA compendium method TO-14.

References

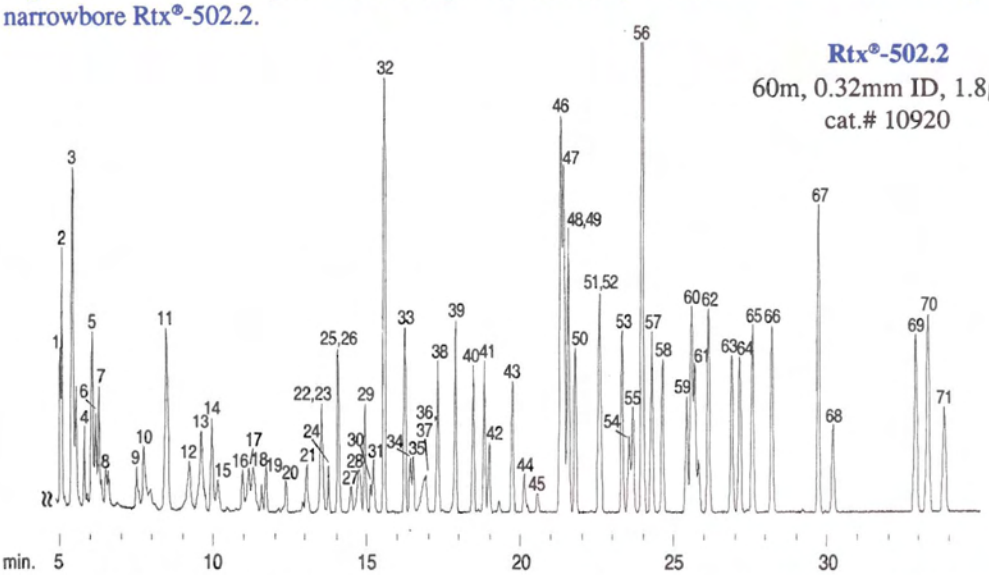
1. Winberry, W.T, Murphy, N.T, Riggan, R.M., *Methods for Determination of Toxic Organics Compounds in Air, EPA Methods, Compendium Method TO-14,* 1990

2. Kelly,T.J, Callahan, P.J., Pliel, J, Evans, G.F., *Method Development and Field Measurements for Polar Volatile Organic Compounds in Ambient Air,* Environ. Sci. Technol. 1993, 27, 1146-1153.

Peak List

1. chlorodifluoromethane
2. dichlorodifluoromethane
3. dichlorotetrafluoroethane
4. chloromethane
5. butane
6. vinyl chloride
7. 1,3-butadiene
8. acetaldehyde
9. bromomethane
10. chloroethane
11. trichlorofluoromethane
12. isopropanol
13. acetone
14. 1,1-dichloroethene
15. acetonitrile
16. dichloromethane
17. acrylonitrile
18. 1-propanol
19. trans-1,2-dichloroethene
20. 1,1-dichloroethane
21. methyl ethyl ketone
22. cis-1,2-dichloroethene
23. methacrylonitrile
24. chloroform
25. bromochloromethane
26. THF
27. 1,1,1-trichloroethane
28. n-butanol
29. heptane
30. 1,2-dichloroethane
31. benzene
32. 1,4-difluorobenzene
33. trichloroethene
34. ethyl methacrylate
35. 1,2-dichloropropane
36. 1,4-dioxane
37. bromodichloromethane
38. MIBK
39. octane
40. toluene
41. 2-hexanone
42. 1,1,2-trichloroethane
43. tetrachloroethene
44. dibromochloromethane
45. 1,2-dibromoethane
46. chlorobenzene-d5
47. chlorobenzene
48. m-xylene
49. p-xylene
50. 2-heptanone
51. styrene
52. o-xylene
53. isopropylbenzene
54. bromoform
55. 1,1,1,2-tetrachloroethane
56. 4-bromofluoromethane
57. n-propylbenzene
58. 1,3,5-trimethylbenzene
59. alpha-methyl styrene
60. t-butylbenzene
61. 1,2,4-trimethylbenzene
62. sec-butylbenzene
63. 1,3-dichlorobenzene
64. 1,4-dichlorobenzene
65. butylbenzene
66. 1,2-dichlorobenzene
67. dodecane
68. dibromochloropropane
69. 1,2,4-trichlorobenzene
70. hexachlorobutadiene
71. naphthalene

Figure 1 - Polar and nonpolar volatile organics commonly found in air can be successfully analyzed on a narrowbore Rtx®-502.2.



60m, 0.32mm ID, 1.8µm Rtx®-502.2 (cat.# 10920)
500ml of 10ppbv standard
Concentrated on an AEROCAN™ 6000 using a glass bead trap at -165°C then desorb at 200°C for 4 min. at 1 ml/min. and cryofocused using a cyrofocusing module at -175°C then desorb at 150°C.

Oven temp.:	35°C (hold 6 min.) to 120°C @ 15°C/min., then to 200°C@ 5°C/min., then to 220°C @ 25°C/min. (hold 10 min.)
Carrier gas:	helium at 1ml/min., linear velocity = 20cm/sec.
Det:	HP-5971A GC/MS
Det. temp:	280°C
Scan Range:	28-260
Solvent delay:	4 min.

Chromatogram courtesy of Allen Madden of The Tekmar Company

Now There are Two Vu-Union™ Designs

• Original design for standard and microbore capillary columns (graphite ferrules) •
• New design for GC/MS and SFC systems (Vespel®/graphite ferrules) •

Restek recently introduced the Vu-Union™ for connecting capillary columns. Although these connectors work well with graphite ferrules under normal GC conditions, an additional connector was needed for applications involving GC/MS and SFC applications that require Vespel® ferrules. Graphite ferrules provide excellent sealing properties under normal GC pressures, but are susceptible to oxygen permeation under vacuum and may leak when excessive pressure is used while Vepsel® maintains a leak-tight seal.

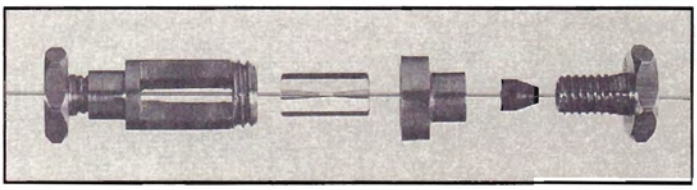
For vacuum and high pressure situations, Restek has developed a new Vu-Union™ for use with Vespel®/graphite ferrules for GC/MS or SFC. Figure 1 shows the disassembled version of the new Vu-Union™. A shortened version of the original Vu-Union™ glass insert allows more torque to be applied to the Vespel®/graphite ferrules without fear of cracking the insert. The addition of hex end nuts allows wrenches to be used for applying high torque to tighten the end fittings.

The new connector also maintains all the benefits of the original Vu-Union™: visual confirmation of the seal, deactivated glass inserts, and a stainless steel high-precision machined body. The new Vu-Union™ combines the benefits of a low dead volume Press-Tight® connector with the confidence of a ferrule seal for the vacuum and high-pressure conditions found in GC/MS or SFC.

The standard Capillary Vu-Union™ is an excellent choice for connections of guard columns to analytical columns, transfer lines at the inlet, or the repair of broken columns. The Capillary Vu-Union™ uses the original glass insert which accepts standard graphite ferrules. The ferrules fit into both ends of the insert, adding a positive seal to the end of the column and connector (Figure 2). The ferrule "seats" or deforms to the shape of the fitting, creating a secondary seal and preventing leaks.

Restek's two Vu-Union™ designs give the chromatographer a number of choices for leak free connections for GC, GC/MS and SFC applications. Vu-Unions™ combine the benefits of a low dead volume connector with the confidence of a ferrule seal. The window allows visual confirmation of the seal between the column and connector. The Capillary and Microbore Vu-Unions™ are designed for graphite ferrules and low pressure conditions. The GC/MS & High Pressure Vu-Unions™ are designed for use with Vespel®/graphite ferrules under vacuum and high-pressure conditions. Deactivated glass inserts are available for all three systems to maintain system inertness.

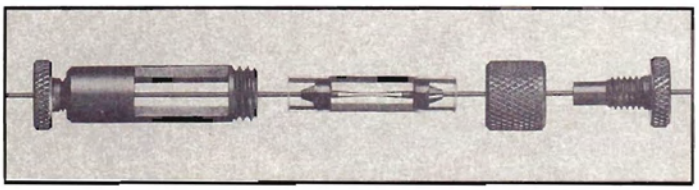
Figure 1 - A disassembled Vacuum/High Pressure Vu-Union™ shows the compact glass insert and hex end nuts.



Features of the Vacuum (GC/MS) and High Pressure (SFC) Vu-Union™:

- Maintains seal under high vacuum used with mass spectrometers.
- Seals under high pressures used in SFC.
- Low thermal mass.
- Uses tough Vespel®/graphite ferruels.
- End fittings incorporate wrench pads for maximum ferrule torque.
- Column seal visually confirmed.
- Universal, fits column IDs from 0.1 to 0.53mm ID.
- Connects columns without peak tailing or loss of efficiency.
- Combines the benefits of Press-Tight® and confidence of ferrule seals.
- Will not unpredictably disconnect.

Figure 2 - A disassembled Capillary/Microbore Vu-Union™ shows the primary and secondary sealing mechanisms.



Features of the Capillary and Microbore Vu-Union™:

- For use with pressures between 0 to 100psi.
- Seals easily without wrenches.
- Uses soft graphite ferrules that conforms easily to all tubing dimensions.
- Column seal visually confirmed.
- Universal, fits column IDs from 0.1 to 0.53mm ID.
- Connects columns without peak tailing or loss of efficiency.
- Combines the benefits of Press-Tight® and confidence of ferrule seals.
- Will not unpredictably disconnect.

Figure 3 - An assembled Capillary/Microbore Vu-Union™ shows the column connection.

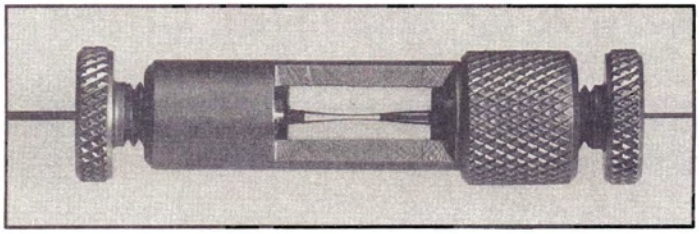
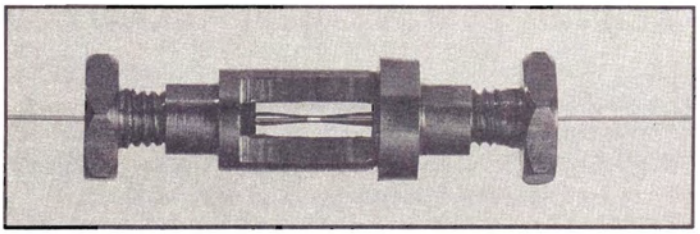


Figure 4 - An assembled Vacuum/High Pressure Vu-Union™ shows the column connection.



Capillary Vu-Union™

- Connect guard columns, transfer lines or repair broken columns.
- Seals easily with minimal torque.
- For use between 0 to 100psi.
- Deactivated, tapered glass insert for column ODs 0.35-0.74mm (IDs from 0.15mm* to 0.530mm).
- For use with standard graphite ferrules (ordered separately).

Capillary Vu-Union™, cat.# 20418 each
Replacement Inserts, cat.# 20419, 3-pk.

Microbore Vu-Union™

- Connect narrow bore tubing under operating conditions from 0-100psi.
- Deactivated, tapered glass insert for column ODs 0.15-0.45mm.
- For use with standard graphite ferrules (ordered separately).

Microbore Vu-Union™, cat.# 20416 each
Replacement Inserts, cat.# 20417, 3-pk.

Vu-Union™ Graphite Ferrules (for Capillary and Microbore Vu-Unions™)

- Easiest ferrules to use with the Vu-Union™.
- Universal, fits Vu-Union™ and connects capillary columns to most GC inlets.
- 450°C maximum operating temperature.

Ferrule ID	Fits Column ID	cat.# (10-pk.)
0.3mm	<0.20mm	20233
0.4mm	0.25mm	20200
0.5mm	0.32mm	20201
0.8mm	0.53mm	20202

Vacuum Vu-Union™

- Connect analytical columns to Mass Spec transfer lines.
- Use under vacuum conditions.
- Deactivated, tapered glass insert for column ODs 0.35-0.74mm (IDs from 0.15mm* to 0.530mm).
- Vespel Graphite ferrules only (ordered separately).

Vacuum Vu-Union™, cat.# 20427 each
Replacement Inserts, cat.# 20428, 3-pk.

High Pressure Vu-Union™

- Will not leak or crack under high-pressure SFC conditions.
- Deactivated, tapered glass insert for column ODs 0.15-0.45mm.
- Vespel®/graphite ferrules only (ordered separately).

High Pressure Vu-Union™, cat.# 20425 each
Replacement Inserts, cat.# 20426, 3-pk

High Pressure and Vacuum Vespel®/Graphite Vu-Union™ Ferrules

- Use only with the High Pressure and Vacuum Vu-Unions™.
- 60% Vespel®/40% graphite.
- 400°C maximum operating temperature.

Ferrule ID	Fits Column	cat.# (10-pk.)
0.3mm	<0.22mm ID - <0.4mm OD	20423
0.4mm	0.25mm ID - 0.4mm OD	20420
0.5mm	0.32mm ID - 0.5mm OD	20421
0.8mm	0.53mm ID - 0.8mm OD	20422

*seals with 0.15mm tubing with a 0.35mm OD

NEW! Hydroguard™
Water Resistant Guard Tubing and Transfer Lines

Dr. Konrad Grob recently published a challenge for chromatographers to develop water resistant guard/transfer line tubing[1]. He found the deactivation layer of a capillary column quickly degrades when it is partially filled with water and refluxed as a vapor (similar to steam cleaning). When transfer lines from purge & traps, air monitoring equipment, or other instruments carry condensed water vapor, the deactivated tubing quickly becomes active due to the creation of free silanol groups. These silanol groups subsequently cause adsorption of active oxygenated compounds such as alcohols and diols.

Restek's chemists investigated this phenomenon and found a solution, our Hydroguard™ deactivation process. By using a unique deactivation chemistry, a high density surface is created that is not readily attacked after an aggressive hydrolysis treatment. The high density surface coverage effectively prevents water vapor from reaching the fused silica surface beneath the Hydroguard™ deactivation layer.

The experiment was conducted by filling deactivated 30m, 0.25mm ID polyimide fused silica columns with 0.75ml of deionized water. One end of the column was flame-sealed, while the other end was evacuated under 0.8mm Hg of vacuum for 15 minutes, then flame-sealed. The columns were subsequently hydrothermally treated by heating at 120°C for one hour and dried with methanol before testing. An evaluation of surface changes was performed by connecting the 30m hydrothermal treated guard tubing to a 30m, 0.25mm ID, 0.25µm Rtx®-5 capillary column using a Vu-Union™ and testing with the Grob test mixture.

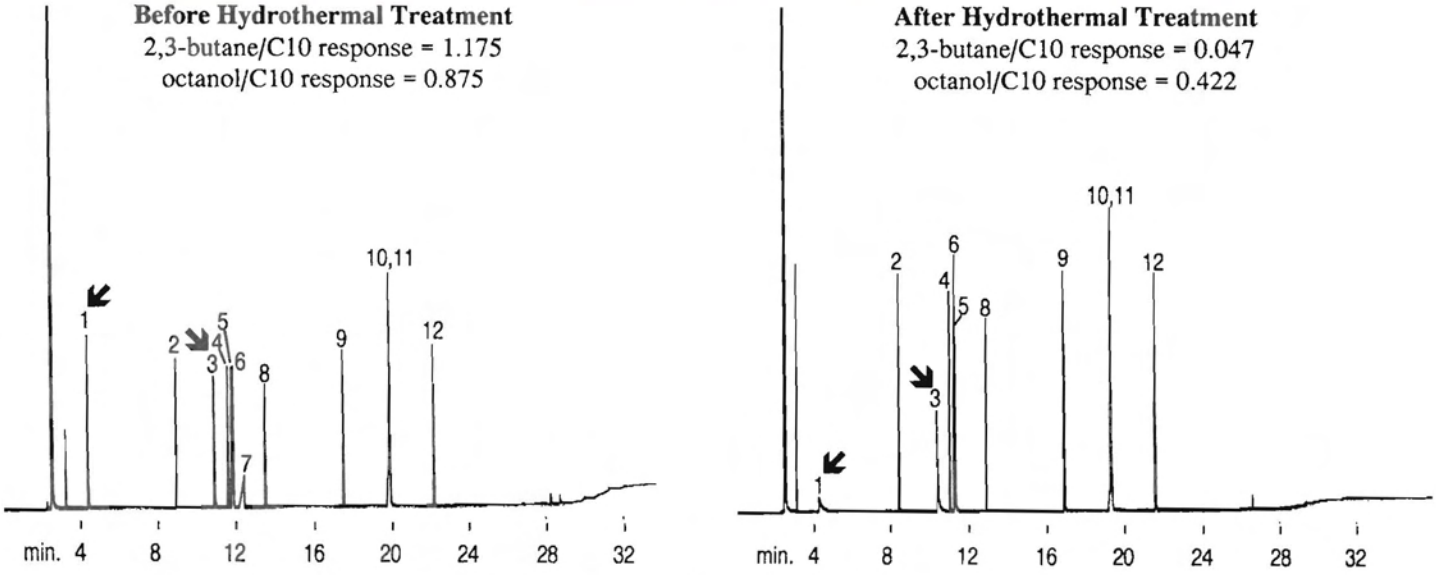
Figure 1 shows the standard Intermediate Polarity (IP) deactivated guard tubing before and after the hydrothermal treatment. Damage to the deactivation layer was indicated by the diminished response and tailing of 2,3-butanediol and octanol peaks. In contrast, the new Hydroguard™ deactivated surface was unaffected by the harsh hydrothermal treatment. Figure 2 shows the Hydroguard™ deactivated guard tubing before and after the hydrothermal treatment. The response of the 2,3-butanediol decreased slightly from 1.225 to 1.087 but the octanol response increased slightly from 0.857 to 0.913. This data shows the resistance of Hydroguard™ deactivated guard/transfer line tubing to aggressive water exposure.

Analysts using guard tubing or transfer line tubing that will be exposed to condensed water vapors should use Hydroguard™ guard tubing and transfer lines. For those analysts injecting organic solvents, Restek's standard IP deactivated guard tubing is preferred. Please call your local distributor if you are unsure which guard tubing to use.

References

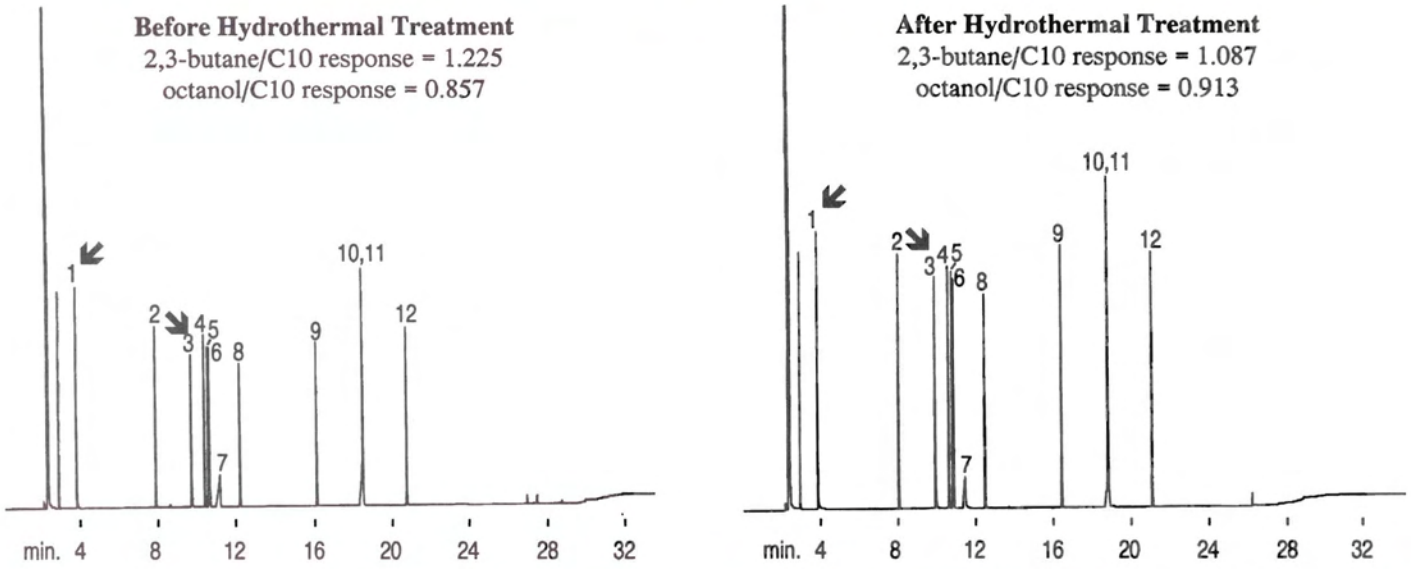
1. K. Grob and B. Schilling, "What Hinders the Further Development of Capillary GC?", *HRC & CC*, Vol 16, June 1993, pg 333-337.

Figure 1 - The Grob Test Mix shows that exposure to water at elevated temperatures causes a loss of tubing inertness with standard guard tubing. (30m, 0.25mm ID IP Guard Tubing)



Peak Identifications and Run Conditions are listed on page 7.

Figure 2 - Grob Test mix shows that the Hydroguard™ guard tubing resists damage from liquid or vaporized water. (30m, 0.25mm ID Hydroguard™ Tubing)



Peak List and Run Conditions for Figures 1 & 2

Grob Test Mix

1. 2,3-butanediol
2. decane
3. 1-octanol
4. undecane
5. nonanal
6. 2,6-dimethylphenol
7. 2-ethylhexanoic acid
8. 2,6-dimethylanaline
9. methyl decanoate
10. dicyclohexylamine
11. methyl undecanoate
12. methyl dodecanoate

30m, 0.25mm ID, 0.25µm Rtx®-5 column (cat.# 10223)
1µl split injection of Grob Test Mix (cat.# 35000)

Oven temp.: 40°C to 185°C @ 6°C/min., then to 325°C @ 15°C/min. (hold 10 min.)
Inj. & det. temp.: 325°C
Carrier gas: hydrogen
Linear velocity: 40cm/sec. set @ 40°C
FID sensitivity: 8×10^{-11} AFS
Split ratio: 40:1

Use Hydroguard™ Tubing for Connecting GCs to:

- Purge & Trap systems
- Headspace analyzers
- Summa canister sampling systems
- Air analysis equipment
- Other instruments that trap and release water vapors to GCs
- Any analytical instrument that needs an inert, water resistant pathway.

Benefits of Hydroguard™ Tubing:

- Resists degradation by water injections or condensation.
- Withstands harsh "steam cleaning" chromatography processes.
- Increases column lifetime.
- Reduces effects of dirty samples on column performance.
- Reduces downtime and maintenance.
- Protects expensive analytical columns.
- Prevents damage from harmful materials.

6-Packs of 5m Hydroguard™ Fused Silica Guard Columns & Transfer lines

Save money when you buy 6-packs!

Nominal ID	Nominal OD	Cat. #
0.25mm	0.40mm	10079-600
0.32mm	0.50mm	10080-600
0.53mm	0.75mm	10081-600

Hydroguard™ Fused Silica Guard Columns & Transfer Lines

- 5-meter lengths for convenient connections
- Copy of Grob test chromatogram included for each tubing lot

Nominal ID	Nominal OD	Cat. #
0.05mm*	0.35mm	10075
0.10mm*	0.35mm	10076
0.15mm	0.35mm	10077
0.18mm	0.40mm	10078
0.25mm	0.40mm	10079
0.32mm	0.50mm	10080
0.53mm	0.75mm	10081

Longer Length Hydroguard™ Fused Silica Guard Columns & Transfer lines

Nominal ID	10-meter cat.#	30-meter cat.#	60-meter** cat.#
0.25mm	10082	10085	10088
0.32mm	10083	10086	10089
0.53mm	10084	10087	10090

*not tested with Grob Mix due to a high pressure drop
** Restek recommends cutting 60m guard columns into shorter lengths. Using them full length may cause peak distortion.

Determining Organic Chemical Purity Using Differential Scanning Calorimetry

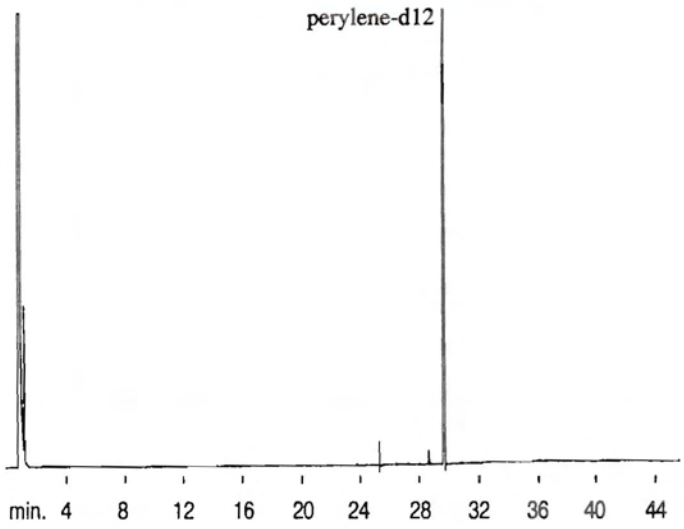
Analytical calibration standards must be made from correctly identified, high purity raw materials. Quantitative standards are prepared gravimetrically and their concentrations are typically assigned from this data. Gravimetric data alone can lead to erroneous results if the material contains varying waters of hydration, salts, or other inorganic impurities. Any impurity may affect the stability and concentration of the standard. Organic chemical purity is most frequently determined by GC/FID or GC/MS. These procedures alone may not yield complete information about the purity of a material. If the impurity is insoluble or does not respond to the detection system used it will go undetected. Since no one analytical technique can provide absolute chemical identification and purity determination, several complimentary techniques must be chosen to ensure the correct identification and purity determination of raw materials used for chemical standards.

Differential Scanning Calorimetry (DSC) has the potential to detect all of the impurities that cannot be identified by GC/FID and GC/MS purity assays including residual catalysts, solvents, water, and inorganic impurities. The extended temperature range of DSC from -170°C to over 600°C allows the determination of accurate melting points not achievable with most melting point equipment. These melting points can be used to help confirm the identity of a chemical.

DSC is an analytical technique where the enthalpy of a sample is measured as a function of its temperature. The instrument is calibrated for temperature, thermal lag, and enthalpy with high purity (99.999+%) metals of known properties. The purity assay is performed on 2 milligrams of the neat raw material, eliminating any possible solvent interferences. The sample is hermetically sealed inside an aluminum sample pan. The assay is run by cooling the sample to 25 degrees below its melting point, ramping the temperature to 25 degrees above the melting point and collecting data on the melting point transition. Any impurity that is soluble in the melt of the main component will cause a melting point depression, or a broadening of the melting transition. Water, residual catalysts, solvents, inorganic, and organic impurities can all be detected by DSC. If the amount of impurities is less than 3 mol/mol percent, the Van't Hoff equation will provide an accurate mol/mol purity determination for the raw material. The mol/mol percent purity is only equal to the weight percent purity when the molecular weights of the impurity and main component are the same. This DSC purity analysis is described in detail in ASTM Method E928-85.

In many cases the DSC data simply confirms the purity determination made by GC/FID. However, in some cases the GC/FID purity data can be misleading. Figure 1 shows a GC/FID analysis of a perylene-d12 sample. When the purity was determined to be 99.5%. However, Figure 2 shows a broad DSC melting point endotherm of this same sample, indicating a major impurity. The impurity was later identified as a residual inorganic catalyst that was not detected by GC/FID. While the exact purity could not be determined, the DSC indicated purity was much less than 97%.

Figure 1 - The GC/FID analysis of perylene-d12 gave a misleading purity value of 99.5 %.



30m, 0.32mm ID, 0.25µm Rtx®-5 (cat.# 10224)
1.0µl split injection of perylene- d12, concentration: 2mg/ml

Oven temp.:	50°C (hold 6 min.) to 300°C @ 10°C/min. (hold 15 min.)
Inj. & det. temp.:	300°C
Carrier gas:	hydrogen
Linear velocity:	40cm/sec. set @ 40°C
FID sensitivity:	1.28 x 10^{-10}AFS

Figure 2 - The DSC detected large amounts of impurities later identified as residual catalyst in the perylene-d12.

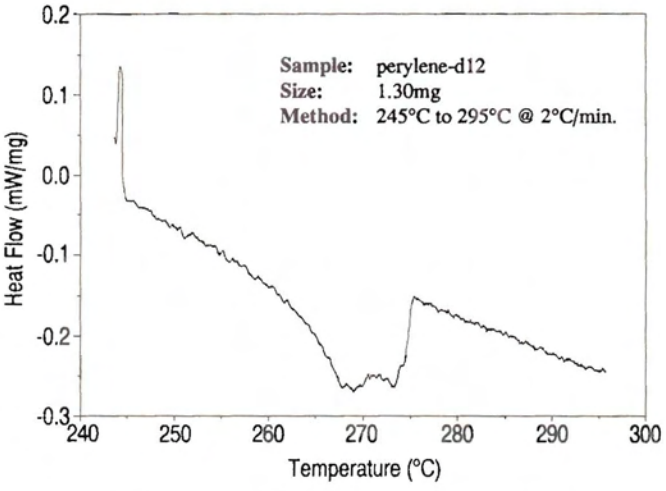
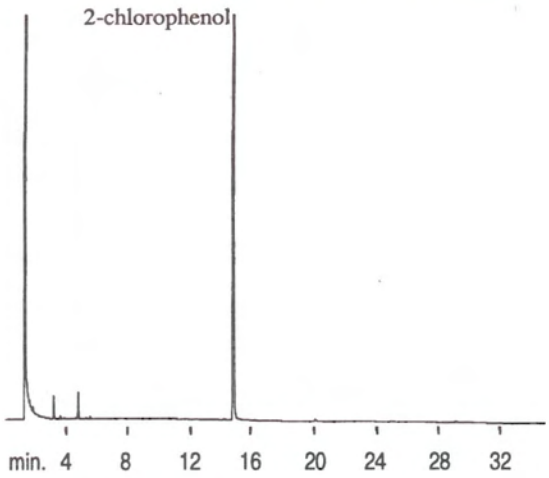


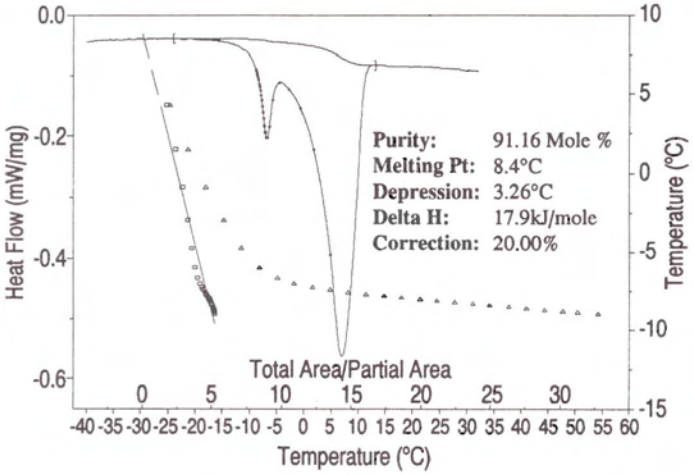
Figure 3 - GC/FID analysis of 2-chlorophenol did not indicate the presence of a major impurity.



30m, 0.53mm ID, 3.0µm Rtx®-1 (cat.# 10185)
2.0µl split injection of 2-chlorophenol, concentration: 2mg/ml

Oven temp.:	40°C (hold 2 min.) to 200°C @ 5°C/min. (hold 30 min.)
Inj./det. temp.:	250°C/290°C
Carrier gas:	hydrogen
Linear velocity:	40cm/sec. set @ 40°C
FID sensitivity:	1.28 x 10^{-10}AFS

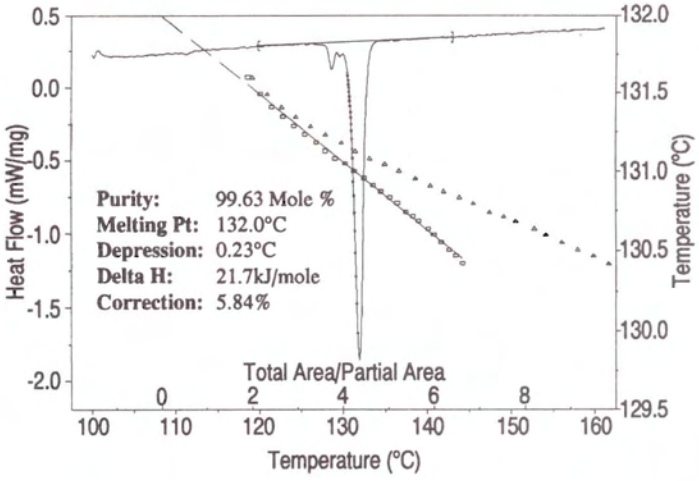
Figure 4 - The two DSC endotherms suggest a large impurity which was later identified as water.



A similar situation occurred with a sample of 2-chlorophenol. The GC/FID analysis indicated a purity of 99.0% (Figure 3). The DSC analysis showed two endotherms, again indicating a major impurity (Figure 4) which was undetected by GC. It was later determined that water, which does not respond on an FID, was the impurity.

All organic compounds are not suitable for purity analysis by DSC. The first constraint is that the raw material must be a single structural isomer with one discreet melting point between -150°C and 600°C. The material must be thermally

Figure 5 - The DSC run on pentobarbital indicated impurities that turned out to be other crystalline states of this material.



stable throughout the experiment while in contact with the aluminum pan and any residual room air inside the pan. Liquid raw materials must be crystallized inside the DSC pan before the experiment starts. Even though the DSC can cool a sample down to -170°C, crystallization is a thermodynamic and kinetic process, thus causing some materials to remain as super cooled liquids for long periods of time.

Figure 5 shows the DSC analysis of a pentobarbital sample. The results would indicate that two impurities existed in the sample. Further investigation revealed that pentobarbital contains three stabile crystalline phases and that the material was actually very pure.

Since no one analytical technique can give absolute chemical identification and purity determination for all organic compounds, multiple techniques must be chosen. DSC is an excellent technique to compliment chromatographic analysis. The combination of DSC, GC/FID, and GC/MS analysis can provide reliable chemical identification and purity determination for most organic compounds. Restek employs DSC in addition to GC-FID and GC-MS for the purity determination of raw materials that are used for our chemical standards.

Call your local distributor to request a copy of our new, 40-page *Chemical Standards Catalog*.

Hints for the Capillary Chromatographer

Techniques for Dual Capillary Column Confirmational Analysis

While capillary columns offer high resolution, they do not necessarily separate all components contained in complex mixtures. Coelutions can occur which decrease the quantitative and qualitative accuracy of an analysis. This is particularly a problem for ECDs, FIDs, NPDs and other detectors which do not give a positive identification for each peak. Even mass spectrometers cannot differentiate between structural isomers and must rely on the column for complete separation. Dual column confirmational analysis using two columns of different polarity can increase the reliability of GC data. If two peaks coelute on the first column, they can usually be separated on a second column of different polarity enhancing qualitative results. Quantitative results can be confirmed since the areas of the coeluting peaks on the first column should equal the combined areas of separated peaks on the second column.

There are three types of single inlet/dual column connection techniques commonly used. The technique chosen will depend on whether split/splitless or direct injections are performed. Only the "Y" Press-Tight® connector/guard column combination can be used with either split/splitless or direct injection techniques. The two-hole ferrule technique works best with split/splitless injections, whereas the direct injection tee is designed to function in a 1/4" packed column injection system operated in the direct injection mode. All three techniques will be described separately.

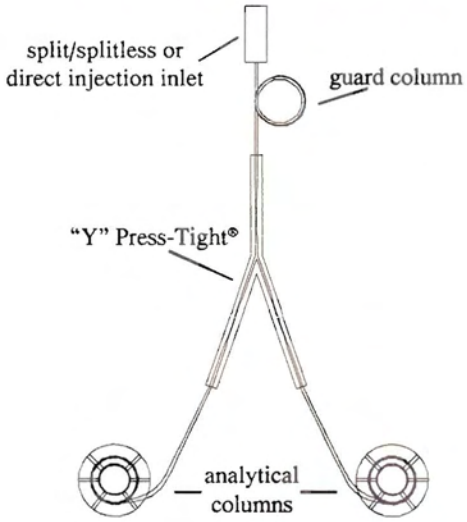
"Y" Press-Tight ® Connector with Guard Tubing

Figure 1 shows the "Y" Press-Tight® configuration for dual column confirmational analysis. A five-meter guard column is connected to the base of the "Y" Press-Tight® with the two analytical columns connected to each outlet leg of the connector. The guard column can be connected to either a split/splitless or direct injection inlet depending on the analyst's preference. The vaporized sample initially travels through the guard column until it reaches the "Y" Press-Tight® where the sample stream splits and a portion travels onto each column. The sample continues to travel through each analytical column until it reaches the detector and provides individual chromatograms.

Press-Tight® "Y"s connect fused silica tubing in the same fashion as a straight Press-Tight® connector. A square cut using a sapphire blade or ceramic scoring wafer is essential to forming a good seal. Examine the column end to make sure it is square and insert it into the Press-Tight® connector, pushing firmly until a uniform brown polyimide "ring" forms. In addition, a small amount of polyimide glue can be used to strengthen the connection.

Usually, the inside diameter of the guard tubing is chosen to match the analytical columns. However, 0.53mm ID guard tubing can be used with two 0.32mm ID analytical columns if the flow rate through the guard tubing is high enough to avoid band broadening. The combined flow rate through each analytical column should equal or exceed the carrier gas optimum flow rate through the larger bore guard tubing.

Figure 1 - The "Y" Press-Tight® configuration allows dual columns to be used in either a split/splitless or direct injection inlet.



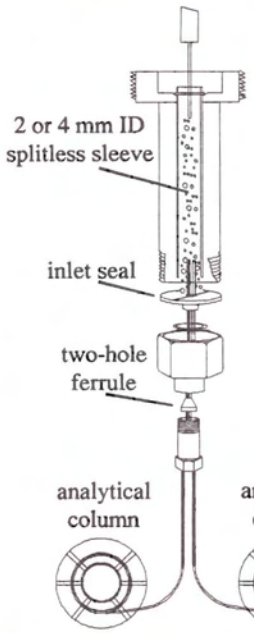
The "Y" Press-Tight® configuration offers versatility since it allows any diameter column or guard column to be connected to any inlet such as split/splitless or direct.

Two Hole Ferrule for Split/Splitless Injectors

Dual column confirmational analysis can also be performed by connecting two columns simultaneously to the same split/splitless inlet via a two-hole ferrule (Figure 2). Most 1/16" capillary inlet fittings will accommodate two 0.25 or 0.32mm ID capillary columns. However, two 0.53mm ID columns are too large to fit a standard 1/16" capillary inlet fitting and require a special 1/8" capillary inlet fitting with a 1/8" two-hole ferrule. Use a split or splitless liner with at least a 4mm ID to ensure that both column ends will fit into the sleeve. If 2mm ID inserts are used, the analyst runs the risk of the column end sitting too close to the sleeve wall which increases split/splitless mass discrimination effects. Standard gooseneck sleeves can not be used because the restriction is less than 1mm

and does not accommodate both columns side by side. Recently, extended goosenecks have become available which are designed with a 4mm internal base to accommodate even two 0.53mm ID columns simultaneously.

Figure 2 - Two hole ferrules can be used to allow dual column confirmational analysis in the same split/splitless inlet.



0.25 and 0.32mm ID column bores can be used with standard 1/16" inlet fittings. However, 0.53mm ID columns require the use of 1/8" fittings to allow both columns to fit side by side in the injector. Either straight or extended gooseneck split/splitless sleeves can be used.

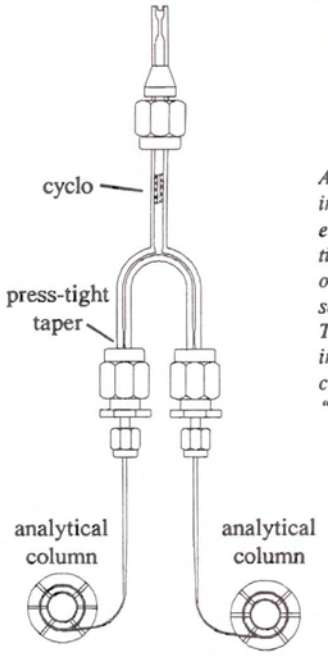
Direct Injection Tee

Many analysts prefer to perform dual column confirmational analysis using direct injection into a 1/4" packed column injection port. Special glass inlet "T"s are available to allow direct connections into two 0.32 or 0.53mm ID columns (Figure 3). The connection from the column inlet to the "T" is made via a Press-Tight® taper as the primary sealing mechanism and a 1/4" to 1/16" reducing fitting as the secondary sealing mechanism. A proper Press-Tight® seal between the column and glass inlet Direct Injection "T" is essential to prevent peak tailing and can be visually observed in Restek's Dual Direct Injection Tee. For the direct injection "T" to function properly, the sample must be thoroughly vaporized prior to the "T" splitting point. Glass wool can be used but may detract from the inertness of the system. Devices such as inverted cups or glass screws (cyclos) can also be incorporated into the inlet leg to ensure complete sample vaporization. These devices also ensure a high degree of inertness since they can be deactivated as a complete unit.

Uniform Sample Splitting

Regardless of which type of dual column system you choose, both column diameters and lengths should be the same. This will ensure that the same amount of sample reaches each column. Slight differences in flow rates between each analytical column are acceptable. However, large flow differences cause an excessive amount of sample to be delivered preferentially onto one column resulting in lower sensitivity for the other column.

Figure 3 - A dual column direct injection "T" allows two 0.32 or 0.53mm ID columns to be securely connected to one 1/4" packed column inlet.



A dual column direct injection "T" incorporates a glass screw to ensure complete sample vaporization prior to splitting the sample onto two columns. The dual sealing mechanisms of a Press-Tight® taper and reducing fitting increase the ease of use and confidence over the Press-Tight® "Y" configuration.

Simultaneous dual column confirmational analysis increases qualitative and quantitative reliability without increasing analysis time. The "Y" Press-Tight® can be used with any injection mode. The two column ferrule technique can only be used for split/splitless injectors, whereas the dual column direct injection "T" must be used in 1/4" packed column injection ports. No conclusive evidence exists that favors one technique over the other when analyzing adsorptive compounds. However, the two-hole ferrule technique used in the splitless injection mode exhibited the highest amount of molecular weight discrimination. Direct Injection is preferred over splitless injection when analyzing high molecular weight compounds, because it minimizes molecular weight discrimination. (For more information on molecular weight discrimination, request Restek's *Guide to Direct/On-column Flash Vaporization Injection.*) Therefore the direct injection tee or the "Y" Press-Tight® in the direct injection mode is recommended over the two-hole ferrule when analyzing high molecular weight compounds or samples with a wide boiling point range. Otherwise, the choice depends on the analyst's personal preference and inlet limitations.

See Restek's *Chromatography Products Catalog* under Dual Column Analysis or call your local distributor for more information. ■

Analyzing Neutral Sterols on an Rtx®-225 for Colon Cancer Research

A special thanks to Dr. Lynne Ausman and Ni Rong of the School of Nutrition, Tufts University, Medford, MA 02155; USDA Human Nutrition Research Center on Aging, Tufts University, Boston, MA 02111, for providing the information for the following article.

Neutral sterols are a class of compounds which include cholesterol and its main degradation products coprostanol and coprostanone. These compounds are formed in the colon with the action of microbial enzymes. Studies of humans that have cancer or exhibit a high risk of colon cancer show that these individuals may have a different amount of cholesterol breakdown than individuals at a lower risk of the disease.

In 1983, Dr. Lynne Ausman of the School of Nutrition at Tufts University, studied a colony of cotton-top tamarins, a new world monkey, exhibiting a high incidence of spontaneous colitis and colon cancer in captivity.[1] In working with these animals, she studied their normal lipid and bile acid metabolism using packed column gas chromatography. The results indicated that these monkeys have a low rate of microbial conversion of cholesterol to secondary products.[1]

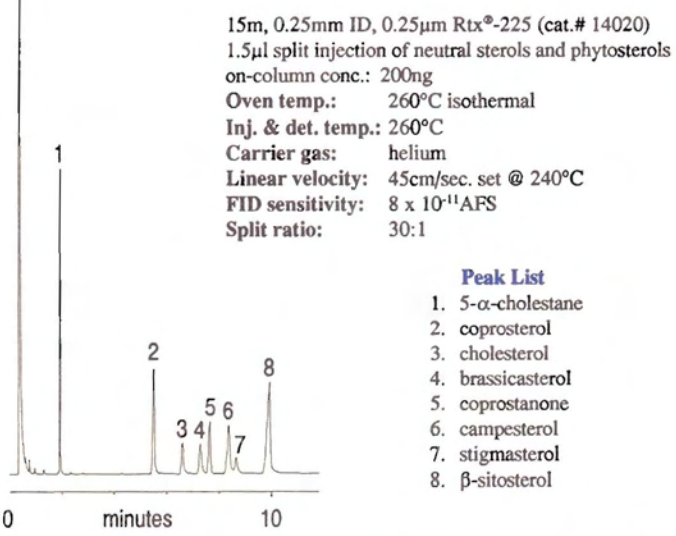
In an effort to determine how diet could slow down or even prevent the disease in humans, Dr. Ausman expanded her research by investigating the fecal sterol output in relation to the type of vegetable oil consumed in the diet. Several vegetable oils show a hypolipidemic response (lower lipid levels) when fed in place of the saturated fats in the "average American" diet. However, some vegetable oils work better than others. These vegetable oils contain plant sterols, termed phytosterols, which humans obtain also through the consumption of fruits, vegetables, grains and grain oils in their diet.

Capillary Gas Chromatography in Fecal Sterol Research

Capillary gas chromatography was used in this research to monitor fecal sterols. The method required separation of the neutral sterols: cholesterol, coprosterol and coprostanone, as well as plant sterols: β-sitosterol, campesterol, brassicasterol, and stigmasterol that were present in the specimens. Although non-polar stationary phases such as the Rtx®-1 (100% dimethyl polysiloxane) and Rtx®-5 (5% diphenyl-95% dimethyl polysiloxane) are suitable for analysis of cholesterol and other sterols, a more polar stationary phase was required for resolution of coprostanone and cholesterol. All analytes, including the cholesterol and coprostanone, are completely resolved on the Rtx®-225 (50% cyanopropylmethyl-50% phenylmethyl polysiloxane), an intermediately polar stationary phase.

Figure 1 illustrates the analysis of these neutral sterols and phytosterols, along with 5-α-cholestane as the internal standard. All analytes are well resolved in 10 minutes and show good peak shape on the 15m, 0.25mm ID, 0.25µm Rtx®-225.

Figure 1 - An Rtx®-225 provides excellent resolution and analysis times of neutral sterols.



There have been studies on humans that suggest those with colon cancer or those at high risk may not metabolize cholesterol to the extent of those who are at a lower risk of the disease. Dr. Ausman and her colleagues are studying consequences on cholesterol metabolism by replacing saturated fats with vegetable oils, which appear to have a hypolipidemic and/or anticholestermic effect. This is monitored by analyzing fecal sterol profiles including neutral sterols and phytosterols by capillary gas chromatography. The neutral sterols cholesterol and coprostanone coelute on non-polar stationary phases commonly used in sterol analysis. The Rtx®-225, however, is an excellent choice for the analysis of the neutral sterols: cholesterol, coprosterol, and coprostanone; and the phytosterols: β-sitosterol, campesterol, brassicasterol, and stigmasterol. All components are well resolved, illustrate good peak symmetry, and are quickly analyzed on this stationary phase.

Dr. Ausman's research also involves analysis of fecal bile acid profiles by capillary gas chromatography. This will be described in a future issue of *The Restek Advantage*.

Reference

1. Ausman, Lynne M., Julia A. Johnson, Catherine Guidry, and Padmanabhan P. Nair, Comp. Biochem Physiol. Vol. 105B, Nos. 3/4, pp. 655-663, 1993.

Product Listing

Rtx®-225
15m, 0.25mm ID, 0.25µm
cat.# 14020

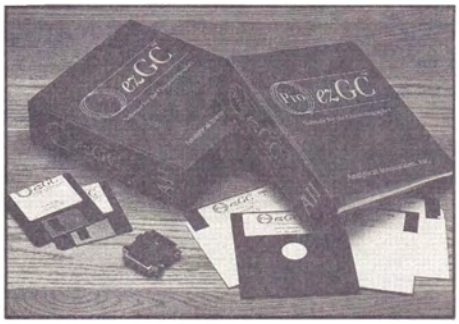
Pro ezGC™
Method Development Software Updates

Pro ezGC™ has become more powerful with new Version 1.5!

- Increase your labs' competitive advantage!
- Optimize temperature and pressure programming parameters to decrease analysis times and increase sample throughput.
- Improve resolution to meet or exceed method protocols.
- Optimize column length, diameter and film thickness ***before*** purchasing the column.
- Import data from ASCII or AIA(ANDI) formats to reduce data entry time.
- Calculate Kovats and Linear Temperature Program Indices, as well as Equivalent Chain Length (ECL) values, for qualitative analysis.

The addition of several new features allows simultaneous optimization of column length, internal diameter, and film thickness, as well as pressure programming. These features are added to the temperature program optimization features already in place. By using *Pro ezGC™*, you can improve the resolution of your analysis, shorten analysis times, increase sample throughput, and save money.

Pro ezGC™ uses thermodynamic retention indices (TRIs) to calculate retention times and elution characteristics for a set of components on a given stationary phase. By entering a column dead time and two temperature programmed runs of experimental data, the user can calculate TRIs. TRIs are then used to predict the performance of these components when any of the column parameters (length, ID, film thickness, carrier gas, or flow control) are changed. By using component libraries and TRIs generated by Restek, you can pick the best column and run conditions without ever installing a column. GC method development and analysis optimization couldn't be easier.



Version 1.5 now allows simultaneous optimization of temperature programs, column length, ID, film thickness and flow or pressure parameters. New component libraries include FAMEs, Pesticides, and PCBs. Call your local distributor to request a complete listing of all the component libraries.

***Pro ezGC™* Software ver. 1.5:**
cat# 21481, $1495

***Pro ezGC™* ver. 1.0 to *Pro ezGC™* ver. 1.5:**
cat.# 21485, $595

***ezGC™* ver. 1.0 or 1.5 to *Pro ezGC™* ver. 1.5:**
cat.# 21482, $1095

Three New Retention Index Libraries Available

Fatty Acid Methyl Ester (FAME) cat.# 21455

FAME thermodynamic retention index libraries are now available for 70 compounds on the Rtx®-2330 and Stabilwax® stationary phases. All straight chain saturates from methyl butanoate(C4:0) to methyl tetradocosanoate(C24:0) are included, along with unsaturates ranging from monounsaturate methyl undecenoate (C11:1) to the polyunsaturate methyl docosahexanoate(C22:6).

Environmental - Pesticides/Herbicides (Part 1) cat.# 21456

A collection of 62 chlorinated pesticides from EPA methods 505, 507, 508, 608.1, 608.2, 1618, and CLP Pesticides, as well as 19 derivatized phenoxy-acids found in EPA methods 515.1, 8150B, and 615 are included in this library. Thermodynamic retention indices are provided on the Rtx®-5, Rtx®-35, and Rtx®-1701 stationary phases.

Environmental - PCBs cat# 21454

A complete collection of retention indices for the 209 polychlorinated biphenyls (PCBs) on the Rtx®-5 stationary phase are included in this library.

Other Retention Index Libraries Available:

Food and Flavor Volatiles (cat.# 21451)
Drugs & Pharmaceuticals (cat.# 21453)
Environmental - Volatiles (cat.# 21452)
Solvents & Chemicals - Part 1 (cat.# 21450)

Please call your local distributor for additional information.

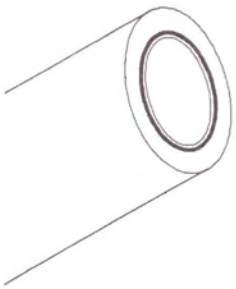
Stabilwax® and MXT®-WAX

The Longest Lasting, Most Inert, Bonded Carbowax® Capillary Columns Available

- Compatible with all solvents including water.
- Resists oxidative degradation.
- Available in polyimide coated fused silica (Stabilwax®) or fused silica lined stainless steel (MXT®-WAX).
- Polymer stable to 250°C.
- Available in a wide variety of IDs, lengths, and film thicknesses.

Stabilwax® (Fused Silica)		(Crossbond® Carbowax® - provides oxidation resistance) Polymer stable to 250°C **Applications:** FAMEs, flavors, acids, essential oils, amines, solvents, xylene isomers, BTEX, EPA Method 603.			
	df (µm)	**temp. range**	**15-Meter**	**30-Meter**	**60-Meter**
0.25mm ID	0.10	40 250°C	10605	10608	10611
	0.25	40 250°C	10620	10623	10626
	0.50	40 250°C	10635	10638	10641
0.32mm ID	0.10	40 250°C	10606	10609	10612
	0.25	40 250°C	10621	10624	10627
	0.50	40 250°C	10636	10639	10642
	1.00	40 240°C	10651	10654	10657
0.53mm ID	0.10	40 250°C	10607	10610	10613
	0.25	40 250°C	10622	10625	10628
	0.50	40 250°C	10637	10640	10643
	1.00	40 240°C	10652	10655	10658
	1.50	50 230°C	10666	10669	10672
	2.00	50 230°C	10667	10670	-----

MXT®-WAX (Silcosteel®)		(Crossbond® Carbowax® - provides oxidation resistance) Polymer stable to 250°C **Applications:** FAMEs, flavors, acids, essential oils, amines, solvents, xylene isomers, BTEX, EPA Method 603.			
	df (µm)	**temp. range**	**15-Meter**	**30-Meter**	**60-Meter**
0.28mm ID	0.25	40 250°C	70621	70624	70627
	0.50	40 250°C	70636	70639	70642
	1.00	40 240°C	70651	70654	70657
0.53mm ID	0.25	40 250°C	70622	70625	70628
	0.50	40 250°C	70637	70640	70643
	1.00	40 240°C	70652	70655	70658
	1.50	50 230°C	70666	70669	70672
	2.00	50 230°C	70667	70670	-----



NEW! Pre-Cleaned Copper Tubing

- Use for plumbing GC systems.
- Specially cleaned to be chromatographically free of background contamination.
- Adheres to ASTM B-280.

cat.#	OD	wall	ID	length
21590	1/8″	0.030″	0.065″	50′
21592	1/4″	0.030″	0.190″	50′

Chemical Standards - New USP 467 Calibration Mixture Available

In the November-December 1993 edition of Pharmocopeial Forum (Volume 19, Number 6) additional modifications have been proposed to USP Method 467. This most recent modification has been introduced to address requirements of the European Pharmacopeia Commission and the Japanese Pharmaceutical Manufacturers Association.

The proposal is to add three additional analytes to the method. These compounds are acetonitrile, pyridine, and 1,2-dichloroethane. Restek has been closely monitoring all proposed modifications, and has available from stock the required calibration mixture. This mixture is shipped complete with an MSDS and a certificate of analysis.

International USP 467 Calibration Mixture

benzene	100µg/ml
chloroform	50
1,4-dioxane	100
methylene chloride	500
trichloroethene	100
acetonitrile	50
pyridine	100
1,2-dichloroethane	100

Prepared in methanol, 1ml/ampul.
Cat.# 36003 each
36103 per pack of 10 ampuls

Other USP 467 Calibration Mixtures Available from Restek:

Revised USP 467 Mixture

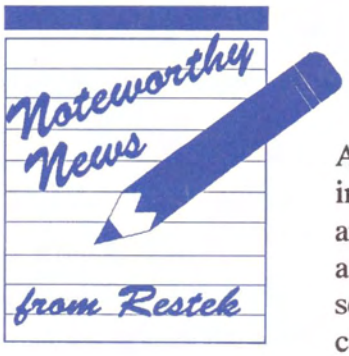
benzene	100µg/ml
chloroform	50
1,4-dioxane	100
methylene chloride	100
trichloroethene	100

Prepared in dimethyl sulfoxide, 1ml/ampul.
Cat.# 36001 each
36101 per pack of 10 ampuls

Proposed USP 467 Mixture

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

Prepared in methanol, 1ml/ampul.
Cat.# 36002 each
36102 per pack of 10 ampuls



Restek Achieves ISO 9001 Certification

We are proud to announce that Restek's quality system has been officially granted ISO 9001 registration by the AT&T Quality Registrar. ISO 9001 is the most encompassing ISO standard, which not only includes a quality assurance system for manufacturing, but also includes quality assurance systems for product design, development, and service. We are very proud of this accomplishment and will continue to improve our quality systems to bring you the best products and services.

The Restek Advantage is printed on recycled paper.

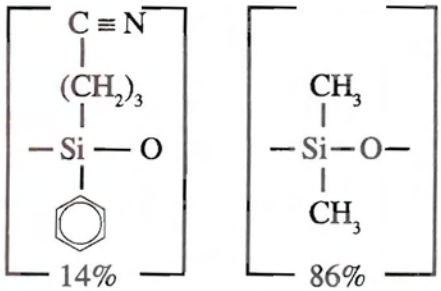
THE RESTEK ADVANTAGE

Rtx®-1701

The Highest Quality 1701 Columns Available

- Polymer synthesized and purified by Restek in our own laboratory.
- Ideal for pesticide, solvent, and drug analysis.
- Maximum operating temperature 280°C.
- Available in a wide variety of lengths, IDs, and film thicknesses.

Figure 1 - Rtx®-1701 phase composition.
14% cyanopropylphenyl
86% dimethyl polysiloxane



One of the most popular stationary phases used in capillary gas chromatography is the Rtx®-1701 (14% cyanopropyl phenyl/86% dimethyl polysiloxane). The unique polarity of this phase makes it ideal for a wide variety of applications including the analysis of chlorinated pesticides, solvents, and drugs. Rtx®-1701 columns are fully bonded, exhibit low bleed, and can be used with even the most sensitive detectors. Each Rtx®-1701 column is tested for inertness, efficiency, and bleed.

Thermal stability

The chemists at Restek have coupled innovative polymer synthesis with advanced deactivation techniques to produce an intermediate polar stationary phase with high thermal stability. The Rtx®-1701 polymer is thermally stable to 280°C. However, the usable operating temperature will decrease with increased film thickness. While a 1.5µm film of Rtx®-1701 is thermally stable to 280°C, the bleed would not be acceptable with most detectors. Therefore, the recommended operating temperature was reduced to 250°C as a practical usable operating temperature. Table I shows the film thicknesses and the recommended operating temperatures for Restek's Rtx®-1701 columns.

Table I: Rtx®-1701's Film Thicknesses and Recommended Operating Temperatures

Film Thickness	Recommended Operating Temperature
0.10 & 0.25µm	280°C
0.50µm	270°C
1.00µm	260°C
1.50µm	250°C

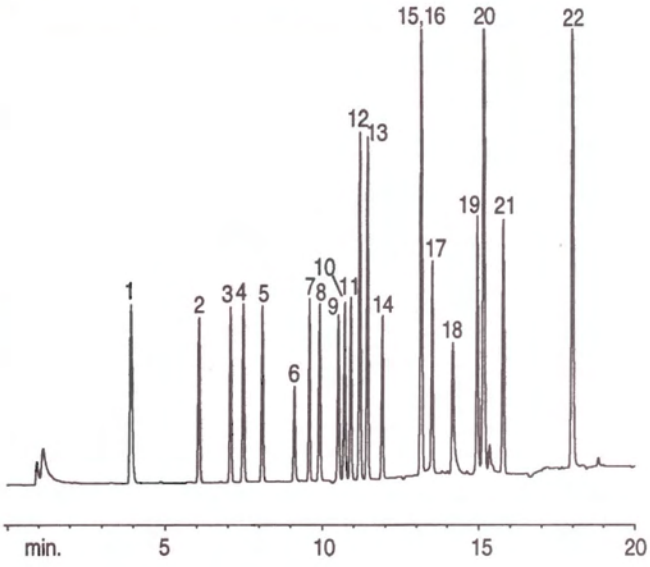
in this issue...

Applications

Rtx®-1701 is recommended for the analysis of CLP chlorinated pesticides

The analytical protocol for the EPA's Contract Lab Program (CLP) requires running samples on two different polarity columns for positive identification and improved quantitative reliability. One of the columns commonly used for CLP pesticide analysis is the Rtx®-1701. It is used in conjunction with either an Rtx®-5, Rtx®-35, or Rtx®-50 column. Most CLP labs use the 30 meter, 0.53mm ID, 0.50µm or the 30 meter, 0.53mm ID, 1.0µm Rtx®-1701 column. However, by using a thin film column, resolution can be improved, analysis time shortened, and bleed levels decreased. Figure 2 shows the analysis of CLP pesticides on a 30 meter, 0.53mm ID, 0.25µm Rtx®-1701 column. Analysis time is reduced to 18 minutes and resolution is improved compared with thicker film columns. Also, the thinner film produces slightly less bleed which is very important with sensitive Electron Capture Detectors (ECDs) used for this analysis.

Figure 2 - A thin film Rtx®-1701 column provides fast analyses and lower ECD bleed for CLP pesticides.



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

30m, 0.53mm ID, 0.25µm Rtx®-1701 (cat.# 12025)
1.0µl direct injection of pesticides
concentration: 80-800ppb (ng/ml)

Oven temp.:	150°C (hold 2 min.) to 275°C @ 7°C/min. (hold 1 min.)		
Inj./det. temp.:	200°C/275°C	**Detector:**	ECD
Carrier gas:	helium	**Linear velocity:**	40cm/sec. set @ 150°C
ECD sensitivity:	5.12 x 10^{-10} AFS	**Flow rate:**	5cc/min.

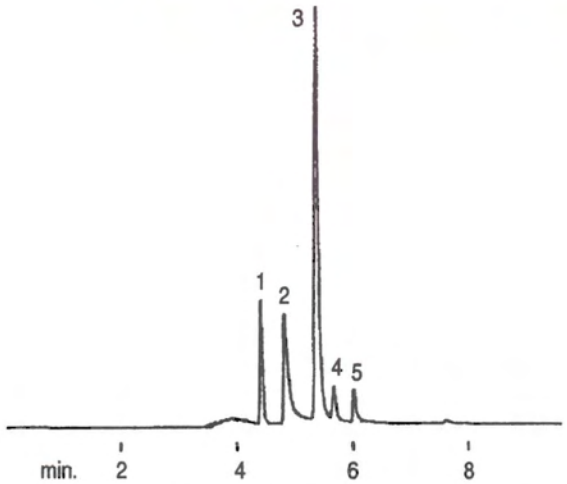
Separate formaldehyde, water, and methanol with an Rtx®-1701 column

Formaldehyde is the 24th highest volume chemical produced in the United States. It is used in the manufacture of phenolic resins and insulating foams. It is most commonly available as a 37-50% aqueous solution that can contain as much as 15% methanol. Therefore, the separation of formaldehyde from both water and methanol is critical for an accurate analysis of this common raw material. Figure 3 shows this important separation on a 60 meter, 0.25mm ID, 1.0µm Rtx®-1701 column. These three components, plus acetaldehyde, a common impurity in formaldehyde, are all separated in 6 minutes.

Analyze acidic/neutral drugs with an Rtx®-1701 column

Barbiturates, hypnotics, sedatives and anti-convulsants are acidic or neutral drugs that are considered to be polar in nature. Intermediate polarity columns produce better peak shapes for acidic and neutral drugs. Traditionally, the analysis

Figure 3 - The Rtx®-1701 resolves water and methanol from formaldehyde.

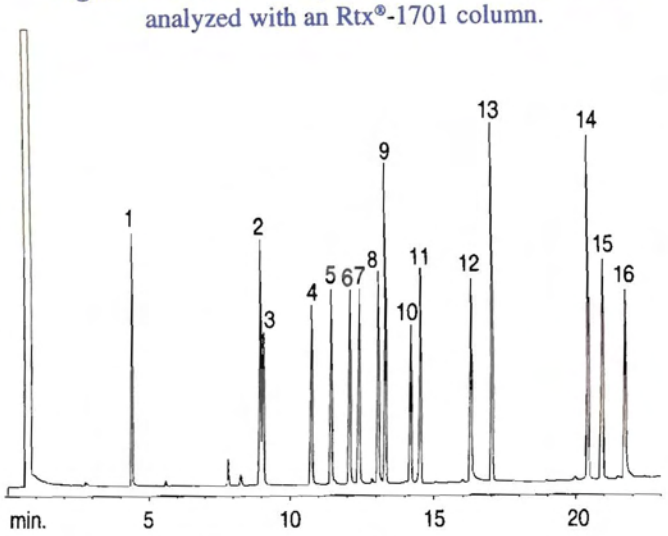


1. air
2. water
3. formaldehyde
4. acetaldehyde
5. methanol

60m, 0.25mm ID, 1.0µm Rtx®-1701 (cat.# 12056)
0.5µl split injection of a formaldehyde sample

Oven temp.:	40°C isothermal
Inj./det. temp.:	150°C/175°C
Detector:	TCD
Carrier gas:	helium
Linear velocity:	20cm/sec. (flow rate: 0.8cc/min.)
TCD sensitivity:	8 mV
Split vent:	30cc/min.

Figure 4 - A wide variety of acid/neutral drugs can be analyzed with an Rtx®-1701 column.



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 of acidic/neutral drugs
concentration: 50µg/ml

Oven temp.:	100°C to 280°C @ 7°C/min.		
Inj./det. temp.:	250°C/175°C	**Detector:**	TCD
Carrier gas:	helium	**Linear velocity:**	40cm/sec. set @ 100°C
FID sensitivity:	5.12 x 10^{-10} AFS	**Splitless hold time:**	0.5 min.

of acidic and neutral drugs is performed on phenyl containing stationary phases like the Rtx®-20 or the Rtx®-35. The unique polarity of the Rtx®-1701 makes it ideal for use as either a primary column or as a confirmational column for these compounds. Figure 4 shows the analysis of a group of acidic and neutral drugs on a 15 meter, 0.53mm ID, 0.50µm Rtx®-1701 column. Good peak shapes and resolution can be maintained while providing elution order switching when compared to phenyl phases.

The low bleed and inertness of the Rtx®-1701 make it ideal for many types of analyses. It is available in a wide variety of lengths, IDs, and film thicknesses for a broad range of applications. Whether you are analyzing pesticides, solvents, or drugs the Rtx®-1701 will yield consistent and accurate results.

Rtx-1701 polymer is synthesized & purified in our own laboratory.

Product Listing

Rtx®-1701 (Fused Silica)		(Crossbond® 14% cyanopropylphenyl-86% methyl polysiloxane) Polymer stable to 280°C **Applications:** pesticides, PCBs, oxygenates, pharmaceuticals, solvents				
	df (µm)	**temp. range**	**15-meter**	**30-meter**	**60-meter**	**105-meter**
0.25mm ID	0.10	-20 280°C	12005	12008	12011	12014
	0.25	-20 280°C	12020	12023	12026	12029
	0.50	-20 270°C	12035	12038	12041	12044
	1.00	-20 260°C	12050	12053	12056	12059
0.32mm ID	0.10	-20 280°C	12006	12009	12012	12015
	0.25	-20 280°C	12021	12024	12027	12030
	0.50	-20 270°C	12036	12039	12042	12045
	1.00	-20 260°C	12051	12054	12057	12060
	1.50	-20 250°C	12066	12069	12072	12075
0.53mm ID	0.10	-20 280°C	12007	12010	12013	Custom
	0.25	-20 280°C	12022	12025	12028	
	0.50	-20 270°C	12037	12040	12043	
	1.00	-20 260°C	12052	12055	12058	
	1.50	-20 250°C	12067	12070	12073	
0.18mm ID	**df (µm)**	**temp. range**	**10-meter**	**20-meter**	**40-meter**	
	0.20	-20 280°C	42001	42002	42003	
	0.40	-20 270°C	42010	42011	42012	

MXT®-1701 metal columns are also available. Please see our 1994-95 Chromatography Products Catalog or call your local distributor for more information.

Analyze Fixed Gases Using the New Rt-Msieve™ 13X PLOT Columns

- Unique selectivity of Rt-Msieve 13X improves overall analysis.
- Immobilized to eliminate particle generation.
- Columns can be reactivated after water contamination.
- Guaranteed column-to-column reproducibility.
- Available in 0.53mm and 0.32mm IDs.

Until recently, the only way to achieve rapid separations of fixed gases was the use of molecular sieve packed and micropacked columns. Traditional Molecular Sieve5Å Porous Layer Open Tubular (PLOT) columns have been useful, but the extended retention and broadened peak width of carbon monoxide has been unavoidable. The Restek wizards have developed the Rt-Msieve™ 13X PLOT column to improve the analysis of fixed gases.

After ten minutes of aggressive sonication, the molecular sieve particles remain intact.

Fast and efficient analysis of fixed gases

The Rt-Msieve™ 13X combines the efficiency of traditional molecular sieve PLOT columns with the unique selectivity of 13X molecular sieve. Figure 1 shows the rapid and efficient analysis of the permanent gases on the 30m, 0.32mm ID Rt-Msieve™ 13X. Baseline separation of all compounds is achieved in just over 2 minutes. Figure 2 shows the same analysis using the 15m, 0.32mm ID Rt-Msieve™ 13X PLOT column with complete resolution in 1.5 minutes.

Unique selectivity of Molecular Sieve 13X material

Until now, only Molecular Sieve 5Å PLOT columns have been available. With the Rt-Msieve 13X PLOT columns, the separation of nitrogen and methane is increased while overall analysis time is decreased by reducing the retention of carbon monoxide.[1] The 13X molecular sieve also produces a narrower peak shape for carbon monoxide allowing for lower levels of detection. Figure 3 shows the analysis of the permanent gases on a Molecular Sieve 5Å PLOT column. While the 5Å PLOT column provides good resolution, the peak shapes are broadened, thus decreasing the minimum detection limit approximately 10-fold.

Resists particle generation

PLOT columns are prepared by coating a thick film of very small particles on the inside column wall. A major drawback of PLOT columns is particle generation caused by vibration or pressure surges. The material in the Rt-Msieve™ 13X has been immobilized by a process unique to Restek to minimize any particle generation. This immobilization is stable for applications where column flow rates are disrupted during valve switching or backflushing operations. Non-immobilized PLOT columns will damage or clog valves, causing expensive repairs and down time.

Available in 0.53mm ID and 0.32mm ID

The Rt-Msieve™ 13X is available in two configurations to satisfy a wide variety of applications. Use the 30m, 0.53mm ID Rt-Msieve™ 13X for most applications and when using on-line analyzers. The 0.53mm ID Rt-Msieve™ 13X PLOT columns offer the flexibility and increased capacity many analysts require. For increased efficiency and low flow applications, Restek offers the 30m, 0.32mm ID Rt-Msieve™ 13X. The 0.32mm ID Rt-Msieve™ 13X PLOT is ideal for portable analyzers having limited gas supplies where low carrier gas flow is essential. For decreased analysis times, 15-meter versions are available for both IDs.

Columns can be reactivated

Molecular sieves are very hydrophyllic and will adsorb any water present in the sample. Water contamination will have detrimental affects on separations causing, 1) the carbon monoxide peak shape to deteriorate and, 2) a reduction in overall resolution. Rt-Msieve™ 13X PLOT columns can be reactivated after water contamination by conditioning at 300°C under dry carrier gas flow, thus extending column lifetime.

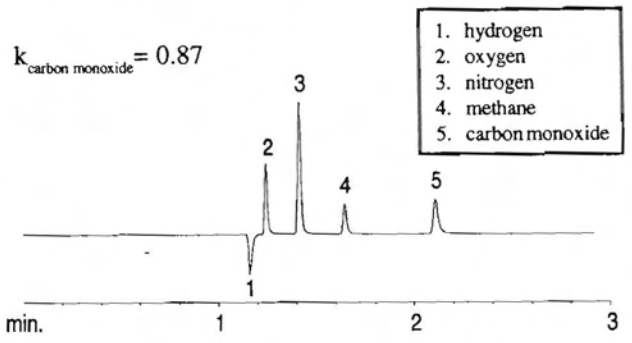
Column-to-column reproducibility guaranteed

All Rt-Msieve™ 13X PLOT columns are tested with a mixture of permanent gases. Columns must pass rigorous specifications for efficiency and strict retention time criteria. This stringent testing insures analysts of column-to-column and run-to-run reproducibility.

The resolution of permanent gases can be improved and the overall analysis time can be reduced using the new Rt-Msieve™ 13X PLOT columns. The immobilized particles minimize potential damage to valves and reduce detector noise. These columns are available in 0.53mm ID for increased capacity or in 0.32mm ID for reduced carrier gas consumption. Rigorous testing guarantees the performance of all Rt-Msieve™ 13X columns.

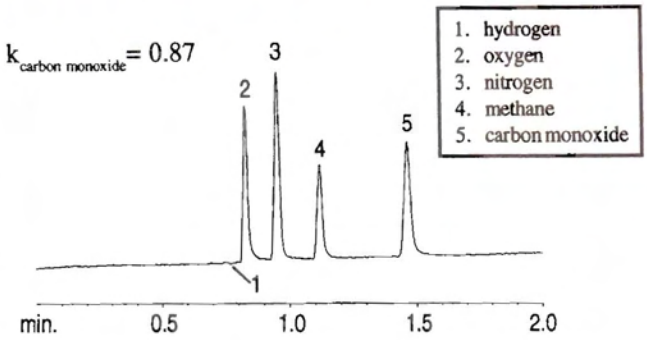
1. Cowper, C.J., DeRose, A.J., *The Analysis of Gases by Chromatography,* Pergamon Press, 1983.

Figure 1 - Resolve permanent gases in just over 2 minutes with a 30-meter Rt-Msieve™ 13X PLOT column.



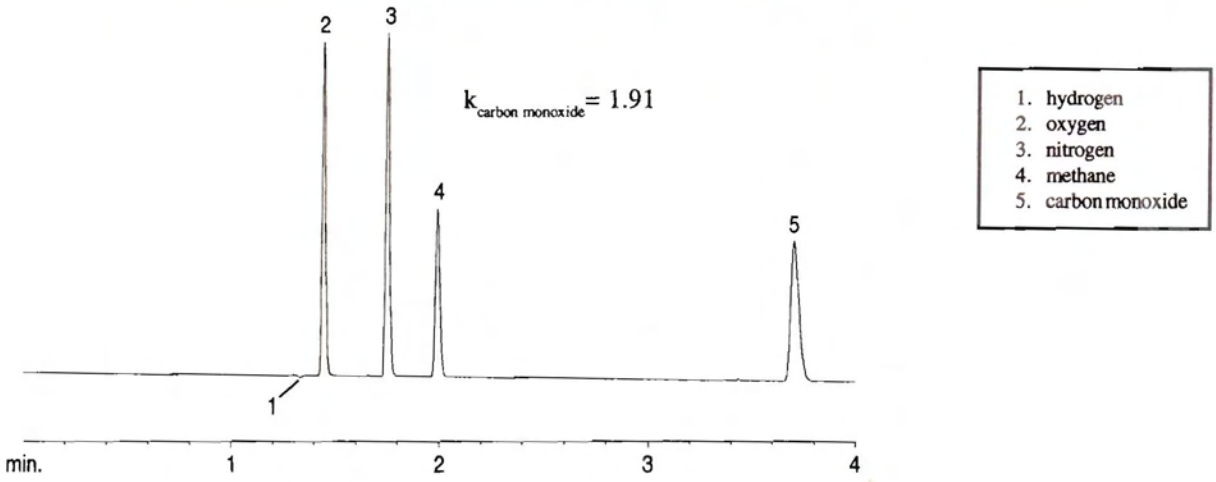
30m, 0.32mm ID Rt-Msieve™ 13X PLOT column (cat.# 19705)
15µl split injection of permanent gases (hydrogen spiked)
Oven temp.: 40°C isothermal
Inj./det. temp.: 200°C/200°C **Detector:** microcell TCD
Carrier gas: helium
Linear velocity: 44cm/sec. set @ 40°C (2cc/min.)
Detector sensitivity: 50mV full scale **Split ratio:** 15:1

Figure 2 - Resolve permanent gases in 1½ minutes with a 15-meter Rt-Msieve™ 13X PLOT column.



15m, 0.32mm ID Rt-Msieve™ 13X PLOT column (cat.# 19707)
20µl split injection of permanent gases
Oven temp.: 40°C isothermal
Inj./det. temp.: 200°C/200°C **Detector:** microcell TCD
Carrier gas: helium
Linear velocity: 32cm/sec. set @ 40°C (1.5cc/min.)
Detector sensitivity: 50mV full scale **Split ratio:** 15:1

Figure 3 - The Molecular Sieve 5Å column produces broader peak shapes and longer retention times for carbon monoxide than the Rt-Msieve™ 13X.



30m, 32mm ID Molecular Sieve 5A PLOT column
20µl split injection of permanent gases
Oven temp.: 40°C isothermal
Inj./det. temp.: 200°C/200°C **Detector:** microcell TCD
Carrier gas: helium **Linear velocity:** 39cm/sec. set @ 40°C (1.85cc/min.)
Detector sensitivity: 50mV full scale **Split ratio:** 15:1

Immobilized to eliminate particle generation.

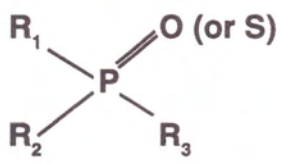
Rt-Msieve™ 13X Columns

15m, 0.32mm ID	cat. # 19707
15m, 0.53mm ID	cat. # 19708
30m, 0.32mm ID	cat. # 19705
30m, 0.53mm ID	cat. # 19706

Analyzing Organophosphorus Pesticides

Pesticides are comprised of many different classes of compounds such as phenoxy-acids and carbamates, as well as molecules containing chlorinated, nitrogen, and organophosphorus functionalities. Organophosphorus pesticides commonly exist as neutral phosphoryl or thiophosphoryl compounds (Figure 1). Their pentavalent character gives them literally hundreds of phosphorous moieties that result in a variety of chemical, physical, and biological properties. They are applied as fungicides, herbicides, insect chemosterilants, and contact or systemic insecticides. Organophosphorus pesticides have a short residence time in the environment due to their instability. Most readily hydrolyze, some photodegrade, and others decompose in alkaline conditions. These characteristics greatly contrast to chlorinated pesticides which show long-term persistence in the environment. The diversity of organophosphorus compounds and their limited life expectancy in the environment makes their use increasingly popular in common pesticide applications.

Figure 1



Most organophosphorus pesticides exist as phosphoryl or thiophosphoryl compounds.

The Environmental Protection Agency has developed specific methods to determine the presence and concentration of these compounds. EPA Method 8141A is a capillary gas chromatographic method that determines the level of organophosphorus compounds in water and soil matrices. This method recommends several different capillary columns for resolving the compounds of interest. In addition, the method has recommendations on detection systems and calibration procedures.

Restek's deactivation produces an inert column that reduces degradation of organophosphorus compounds.

When selecting a column for organophosphorus pesticide analysis, a chromatographer must consider three important factors: column inertness, efficiency, and retention time reproducibility. Because these pesticides are prone to breakdown, the column must be inert to prevent decomposition or reactivity. When analyzing complex mixtures, the column must exhibit high efficiency for maximum resolution. Some methods suggest the use of retention time windows. Retention time windowing requires that the column must demonstrate reproducible results to insure proper identification. Restek offers capillary columns that meet the requirements for analyzing organophosphorus pesticides. Two calibration mixtures containing 28 EPA Method 8141A compounds were completely resolved using an Rtx®-35, 30m, 0.53mm, 0.50µm column (Figures 2 and 3). The active organophosphorus pesticides exhibit minimal breakdown and were well resolved on the Rtx®-35 stationary phase. Relative standard deviations for retention times were less than 1%.

Figure 2 - Rtx®-35 is an excellent column for resolving organophosphorus pesticides.

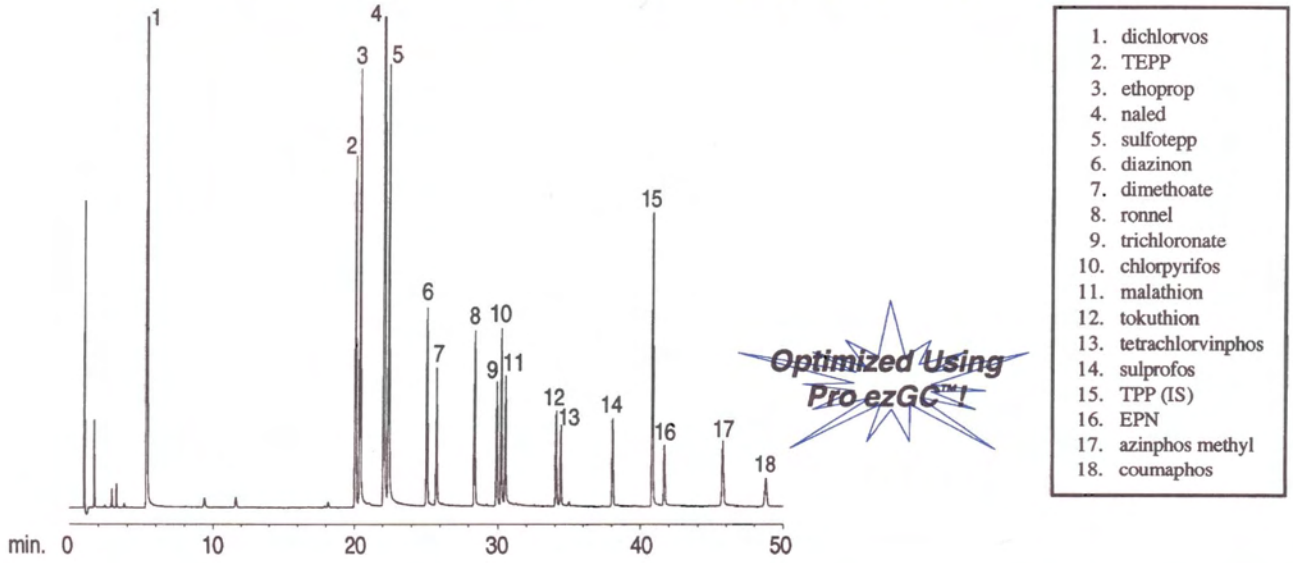
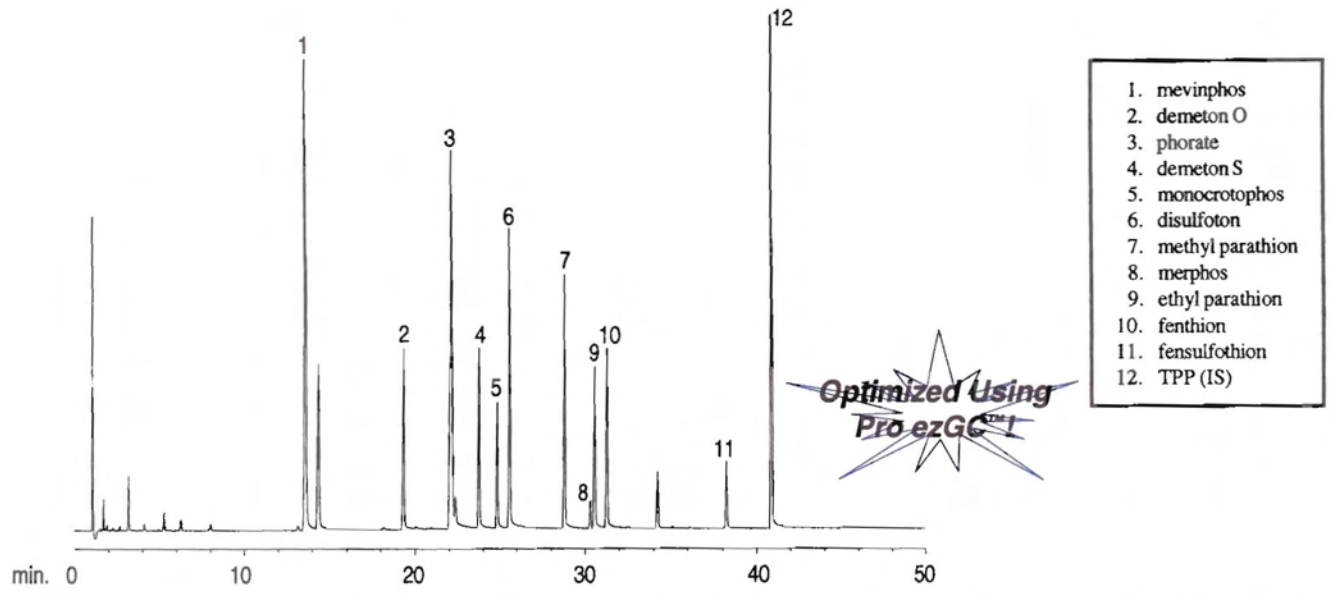


Figure 3 - The Rtx®-35 baseline resolves all organophosphorus pesticides.



In addition to column considerations, frequent maintenance must be performed on the inlet system. Over numerous analyses, the rate of adsorption may increase due to the non-volatile contamination that can build up in the inlet sleeve. Sleeve cleaning or replacement will typically restore chromatographic performance.

Analyzing organophosphorus pesticides can be demanding due to their unstable nature. Also, the wide variety of these pesticides can create difficult separation problems. Highly inert capillary columns, available from Restek, can alleviate the problem of analyte adsorption. These efficient and reproducible columns prevent coelutions and minimize percent errors in relative retention times.

References:
1. Eto, Morifusa. *Organophosphorus Pesticides: Organic and Biological Chemistry.* CRC Press, Inc., Ohio, 1974.
2. EPA SW-846 Module Method 8141A: *Organophosphorus Compounds By Gas Chromatography: Capillary Column Technique.* Non-promulgated, 1990.

Run Conditions for Figures 2 & 3

30m, 0.53mm ID, 0.50µm Rtx®-35 (cat.# 10440)
0.5µl direct injection of Organophosphorus Pesticide
Mix A (Fig. 2) & Mix B (Fig. 3)*
on-column concentration: 25-100ng
Oven temp.: 125°C (hold 10 min.) to 250°C @ 4°C/min. (hold 15 min.)
Inj./det. temp.: 200°C/250°C
Detector: FPD
Carrier gas: helium
Linear velocity: 35cm/sec. set @ 125°C
FPD sensivity: 2 x 10^{-9}AFS

Product Listing

Rtx®-35 column
30m, 0.53mm ID, 0.50µm
cat.# 10440

Vu-Tight® Direct Injection Inlet Sleeves

- Visually observe the press-tight connection between the column end and sleeve.
- Fits 0.32 and 0.53mm ID capillary columns.
- Slotted top prevents obstruction of carrier gas flow.
- Designed for 1/4" injection ports.

Vu-Tight® Direct Injection Sleeve (1/4" OD)

Can be easily packed with wool for dirty samples.

cat.# 20342 each
cat.# 20343, 5-pk.
cat.# 20344, 25-pk.

Cyclo Vu-Tight® Direct Injection Sleeve (1/4" OD)

Is ideal for dirty samples and prevents non-volatile residue from contaminating the column.

cat.# 20787 each
cat.# 20788, 5-pk.

Vu-Tight® Installation Fittings: Includes a 1/4" SS nut & graphite ferrule for attaching the sleeve to the GC inlet, and a 1/4" to 1/16" SS reducer & 1/4" by 0.5mm ID graphite ferrule for attaching the column to the sleeve. **cat.# 20504, kit**

Restek's Rtx®-65

Highest Percent Phenyl Stationary Phase Available

- Selective for aromatic compounds.
- Maximum operating temperature 300°C.
- Available in a variety of lengths, IDs, and film thicknesses.

Many of the most popular stationary phases are based on phenyl methyl polysiloxanes including Rtx®-5, Rtx®-20, Rtx®-35, and Rtx®-50. The popularity of these stationary phases is due to their high thermal stability and their selectivity for aromatic compounds. With increased phenyl substitution, better separation of similar aromatic compounds can be achieved through increased interaction of these compounds with the phase. The Rtx®-65TG, developed specifically for the analysis of triglycerides, resolves triglycerides by degree of unsaturation as well as by carbon number. This same stationary phase, Rtx®-65 (65%diphenyl/35%dimethyl polysiloxane), is now available for a wide variety of applications.

Retention indices

As the concentration of phenyl groups increase, stationary phase polarity also increases. For compounds in a homologous series, as stationary phase polarity increases, absolute retention of the compounds also increases. When analyzing different classes of compounds, higher polarity phenyl/methyl stationary phases will preferentially retain polar, aromatic compounds. Table I lists Kovat's retention indices of the Rtx®-65 compared to the Rtx®-50 and Stabilwax® phases. As the retention indices indicate, the Rtx®-65 shows greater retention than the Rtx®-50, but lower retention than "polar" phases such as the polyethylene glycol (Stabilwax®). The selective characteristics of the Rtx®-65 offer analysts a column at the upper end of the intermediate polarity range and separations previously unobtainable on lower or higher polarity phases.

Table I - Rtx®-65 Kovat's Retention Indices

phase	benzene	butanol	2-pentanone	nitropropane	average
Rtx®-50	777	764	806	911	815
Rtx®-65	794	779	825	937	834
Stabilwax®	956	1142	987	1217	1076

Thermal stability

The chemists at Restek have coupled innovative polymer synthesis with advanced deactivation techniques to produce an intermediate polarity stationary phase with high thermal stability. The maximum operating temperature of the Rtx®-65 is 300°C. However, due to the increased aromatic content, the minimum operating temperature of the phase is restricted to 50°C. Operating Rtx®-65 columns below this temperature will result in broad peak shapes of early eluting components. The film thicknesses and recommended minimum and maximum operating temperatures of the Rtx®-65 columns offered by Restek are shown in the product listing on page 9.

Applications

Rtx®-65 as a confirmational column for EPA Method 604 Phenols

Confirmatory analyses are often required in many EPA methods. By running samples on a second column of different polarity, a more positive confirmation of component identity is achieved. The analysis of priority pollutant phenols is routinely performed on an Rtx®-5 (5%diphenyl/ 95% dimethyl polysiloxane) column. The Rtx®-65 is an excellent confirmational column to the Rtx®-5 for the analysis of EPA Method 604 as shown in Figure 2. The Rtx®-65 produces a different elution order for 4 of the eleven phenols and the analysis time is less than 23 minutes. The inertness of both columns is demonstrated by the excellent response of the compounds such as 2,4-dinitrophenol and pentachlorophenol, even at low concentrations.

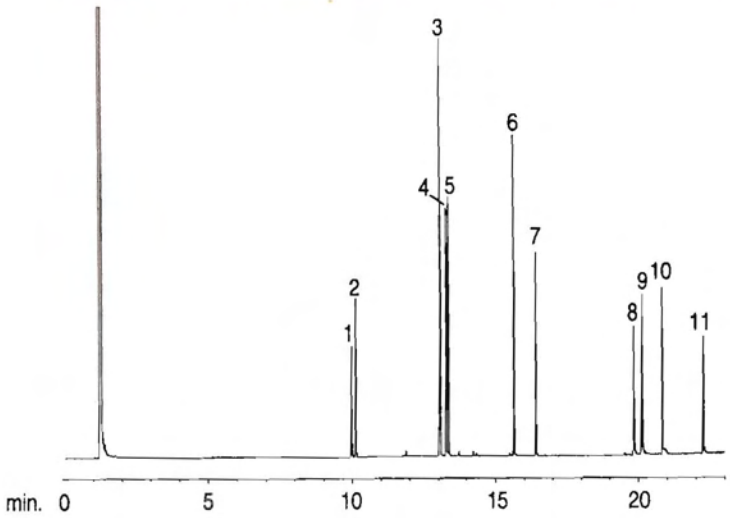
FAMEs

Because the consumption of large amounts of saturated fats has been linked to heart disease and cancer, accurate fatty acid analysis of food products is extremely important. Fatty acids are frequently analyzed in their methylated form to increase sample volatility, improve peak shape, and provide more accurate chromatographic data. Fatty Acid Methyl Esters (FAMEs) are commonly analyzed on polar stationary phases such as the Stabilwax® (polyethylene glycol or PEG) or the Rtx®-2330 (90%biscyanopropyl/ 10% cyanopropylphenyl polysiloxane). However, both of these stationary phases suffer from limited thermal stability and short column lifetimes. The Rtx®-65 is an excellent column choice for FAME analysis as shown in Figure 3. The Rtx®-65 provides resolution of the C14 to C24 fatty acids in canola oil in under 12 minutes and elutes FAMEs according to equivalent chain length similarly to the Stabilwax® column.

Triglycerides

The Rtx®-65TG is the same polymer as the Rtx®-65 but is designed specifically for triglyceride analysis. The Rtx®-65TG is selective in resolving triglycerides according to degree of unsaturation, as well as carbon number and has a maximum operating temperature of 370°C. The Rtx®-65TG is available in both 15 and 30 meter lengths in 0.25, 0.32, and 0.53mm IDs with a 0.10µm film thickness. Rtx®-65TGs are specially tested with a temperature programmed triglyceride test mixture and are guaranteed for low column bleed and high efficiency. Separation by degree of unsaturation as well as carbon number can be achieved in 32 minutes with minimal column bleed at 365°C.

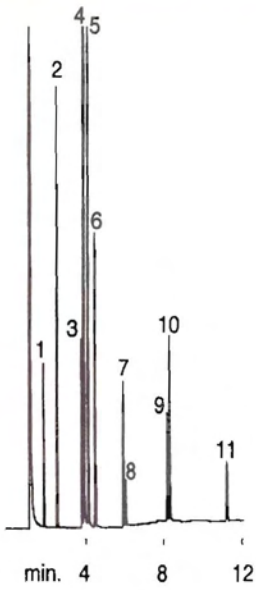
Figure 2 - The Rtx®-65 provides a different elution order than the Rtx®-5 or Rtx®-200 for EPA Method 604 Phenols.



1. 2-chlorophenol
2. phenol
3. 2,4-dimethylphenol
4. 2-nitrophenol
5. 2,4-dichlorophenol
6. 4-chloro-3-methylphenol
7. 2,4,6-trichlorophenol
8. 2,4-dinitrophenol
9. 4-nitrophenol
10. 2-methyl-4,6-dinitrophenol
11. pentachlorophenol

30m, 0.25mm ID, 0.25µm Rtx®-65 (cat.# 17023)
1µl split injection of 604 phenols
on-column concentration=50ng/µl
Oven temp.: 40°C (hold 4 min.) to 250°C @ 10°C/min.
Inj./det. temp.: 300°C/300°C **Detector:** FID
Carrier gas: hydrogen **Linear velocity:** 40cm/sec. set @ 40°C
FID sensitivity: 1.28 x 10^{-10} AFS **Split ratio:** 40:1

Figure 3 - Resolve saturated fatty acid methyl esters according to equivalent chain length on the Rtx®-65.



1. C14:0
2. C16:0
3. C18:0
4. C18:1
5. C18:2
6. C18:3
7. C20:0
8. C20:1
9. C22:0
10. C22:1
11. C24:0

30m, 0.25mm ID, 0.25µm Rtx®-65 (cat.# 17023)
0.5µl split injection of FAMEs (Canola Oil)
Oven temp.: 225°C (hold 5 min.) to 250°C @ 10°C/min. (hold 10 min.)
Inj. & det. temp.: 250°C **Detector:** FID
Carrier gas: hydrogen **Linear velocity:** 40cm/sec. set @ 225°C
FID sensitivity: 4 x 10^{-11} AFS

The Rtx®-65 column line is made with an intermediate polarity phenyl methyl stationary phase that is ideal for many types of analyses. Because of the high thermal stability, Rtx®-65 columns offer an excellent alternative to the more polar stationary phases such as the polyethylene glycols and bis-cyanopropyl phenyls. The Rtx®-65TG is ideal for triglyceride analysis providing separation of fatty acids by degree of unsaturation as well as carbon number. Low column bleed and excellent column efficiency are also traits of this new stationary phase.

Product Listing

Use Rtx®-65 columns for general purpose separations like phenols and fatty acids.

Rtx®-65 (Fused Silica)		(Crossbond® 65% diphenyl-35% dimethyl polysiloxane)	
	df (µm)	15-meter	30-meter
0.25mm ID	0.25	17020	17023
	0.50	17035	17038
	1.00	17050	17053
0.32mm ID	0.25	17021	17024
	0.50	17036	17039
	1.00	17051	17054
0.53mm ID	0.25	17022	17025
	0.50	17037	17040
	1.00	17052	17055

Use Rtx®-65TG columns for separating triglycerides requiring high column temperatures for elution.

Rtx®-65TG (Fused Silica)		(Crossbond® 65% diphenyl-35% dimethyl polysiloxane)	
ID	df (µm)	15-meter	30-meter
0.25mm	0.10	17005	17008
0.32mm	0.10	17006	17009
0.53mm	0.10	17007	17010

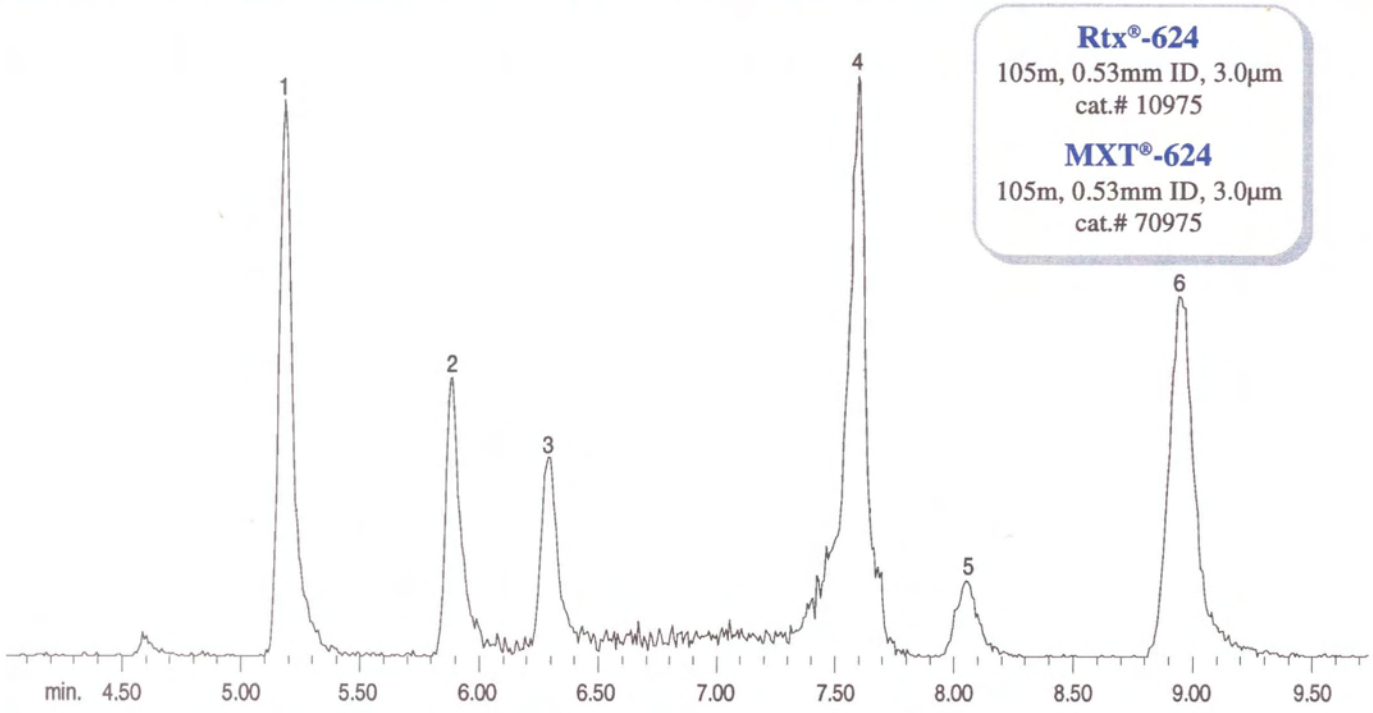
Rtx®-624 Capillary Column Meets New EPA CLP Resolution Requirements for VOA Gases

The February 1994 revision of the Contract Lab Program (CLP) Statement of Work for Volatile Organic Analysis includes a change in requirements for the separation of volatile gases. The new criteria reads as follows: *"For capillary columns, if the gaseous compounds chloromethane, bromomethane, vinyl chloride, and chloroethane fail to exhibit narrow, symmetrical peak shapes, are not separated from the solvent front, or are not resolved greater than 90% from each other, as evidenced by the RIC, then a sub-ambient oven controller must be used, and the initial temperature must be less than or equal to 10°C."*

Restek's 105m, 0.53mm ID, 3.0µm Rtx®-624 fused silica and MXT®-624 metal capillary columns meet this resolution requirement without the need for sub-ambient cooling. The Rtx®-624 stationary phase was specially designed for the separation of volatile organic compounds. Figure 1 shows the typical separation of the first six gases obtained with the Rtx®-624 column. All six compounds are baseline resolved using an initial starting temperature of 35°C.

The resolution of these volatile gases can be affected by the desorb flow rate, the trap type, and the dead volume between the purge & trap system and the column. A desorb flow of 8 to 10ml/min. produces a narrow sample band which results in improved separation. The VOCARB™ 3000 trap produced the best resolution when compared to other commonly used traps. The dead volume between the purge & trap system must be minimized to ensure good peak symmetry. The use of a low volume injector or a narrow bore inlet sleeve installed into a standard injector will result in better resolution of the volatile gases. If these three recommendations are followed, the new CLP resolution criteria can be easily met using the Rtx®-624 column.

Figure 1 - The 105-meter Rtx®-624 column completely resolves the VOA gases to meet the latest revision of the CLP SOW 2/94.



105m, 0.53mm ID, 3.0µm Rtx®-624 (cat.# 10975)
Oven temp.: 35°C (hold 8 min.) to 220°C @ 8°C/min.
Inj./Det. temp.: 100°C/280°C
Detector: HP 5971MSD
Scan range: 35-260 AMU
Purge & Trap: Tekmar 3000 interfaced to the GC system using a Low Volume Injector

Trap: VOCARB™ 3000
Purge: 11 min.
Trap pressure control: 4psi
Desorb preheat: 245°C
Desorb temp.: 250°C
Desorb time: 2 min.
Desorb flow rate: 10cc/min.

Peak Identifications
1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane

Pro ezGC™ Retention Index Libraries

Three New Libraries are Available

Restek's retention index libraries and *Pro ezGC*™ software can be used to select the most appropriate column and optimize chromatographic methods without making a single injection. Restek has added three new libraries and updated two existing libraries to increase the versatility of this method development tool. Analysts can choose from a wide range of environmental, pharmaceutical, clinical/forensic, solvent & chemical, and food & flavor components on common stationary phases. The libraries have been generated using Restek stationary phases, but for modeling purposes, the data closely simulates the performance of other commercially available phases (DB™-1, SPB-1, DB™-5, SPB-5, etc.). Call your local distributor for a list of components or more information on using *Pro ezGC*™ and retention index libraries to optimize your chromatographic analyses.

Environmental - Base, Neutral and Acid Extractables: The analysis of BNAs is one of the most common GC/MS applications performed by environmental laboratories. This library contains 96 semi-volatile retention indices for EPA methods 525, 625, and 8270. These indices were determined using the Restek Rtx®-5/XTI®-5 stationary phase.
cat.# 21457

Environmental - Pesticides/Herbicides Part 2: This library contains a collection of 30 organophosphorous and 53 nitrogen containing pesticides from EPA Methods 507, 614, 619, 1618, and 8141A. Thermodynamic retention indices are provided using the Rtx®-5, -35, and -1701 phases. Part 2 is designed to compliment the chlorinated pesticides and phenoxy acid herbicides offered in Restek's Pesticides/Herbicides Part 1.
cat.# 21458

Solvents and Chemicals Part 2: This new library contains over 120 aromatics, esters, and ethers and is designed to compliment Solvents and Chemicals, Part 1 containing alcohols, aldehydes, and ketones. Retention indices are provided for the Rtx®-1, Rtx®-624, and Stabilwax® stationary phases.
cat.# 21459

Food and Flavor Volatiles Rev. 2.0: This updated library contains 150 new indices for alcohols, esters and ketones in addition to terpenes and other compounds originally offered in Rev. 1.0. In total there are now 300 retention indices for compounds found in food and flavor analyses, calculated on the Rtx®-1 and Stabilwax® stationary phases.
cat.# 21451

Drugs and Pharmaceuticals Rev. 2.0: This new update adds Rtx®-1 and Rtx®-35 stationary phases to the existing Rtx®-5, -50, and -200 stationary phases. The expanded library now contains retention indices for over 100 drugs and pharmaceuticals frequently analyzed by forensic, clinical, and drug testing laboratories.
cat.# 21453

Other Retention Index Libraries Available:

Fatty Acid Methyl Ester (FAME): cat.# 21455

Environmental - Pesticides/Herbicides (Part 1): cat.# 21456

Environmental - PCBs: cat.# 21454

Environmental - Volatiles: cat.# 21452

Solvents and Chemicals (Part 1): cat.# 21450

Software:

***Pro ezGC*™ Software ver. 1.5: cat.# 21481**

***Pro ezGC*™ ver. 1.0 to *Pro ezGC*™ ver. 1.5: cat.# 21485**

***ezGC*™ ver. 1.0 or 1.5 upgrade to *Pro ezGC*™ ver. 1.5: cat.# 21482**

***ezGC*™ software ver. 1.5: cat.# 21480**

***ezGC*™ ver. 1.0 to *ezGC*™ ver. 1.5: cat.# 21483**

ezGC™ *and Pro ezGC*™ *Method Development Software*

Hints for the Capillary Chromatographer

Using Electrolytic Conductivity Detectors

The electrolytic conductivity detector (ELCD) was developed in the early 1960's for the detection of organics in aqueous solutions. The ELCD is a highly selective, destructive detector for organic molecules containing fluorine, chlorine, bromine, nitrogen, and sulfur. It can be operated in three different modes: halogen, nitrogen, or sulfur. The ELCD is extensively used in the environmental field for the analysis of halogenated organics compounds such as those monitored in EPA Methods 502.1, 502.2, 601, 602, 8010, and 8021. It can also be used for various other environmental applications where compounds containing nitrogen, sulfur, or chlorine are of interest, such as organochlorine pesticides, PCBs, organophosphorus pesticides, and nitrosamines. It is also commonly used for pharmaceutical samples.

Detector Design and Operation

The ELCD operates as an electrical conductivity measuring device. However, it is the chemical aspect of the ELCD that provides the basis for its selectivity. The ELCD system consists of four main areas: reactor, conductivity cell, solvent, and electronic detection system.

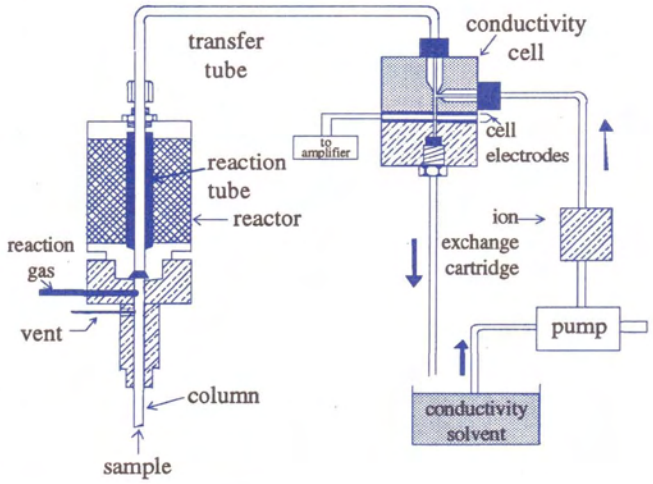
Reactor tube

The reactor is the bridge between the capillary column and the conductivity cell. It consists of the detector base, reaction tube, and heating element. Organic compounds eluting from the capillary column, enter the detector base, combine with reaction gas, and proceed through a high temperature reaction tube usually made of nickel or fused silica. In the reaction tube, most of the compounds are pyrolyzed and, with the presence of an active gas such as hydrogen or oxygen, chemical reactions will occur. In the sulfur mode, the sample components are oxidized using O_2 reaction gas to form SO_2. In the halogen and nitrogen modes, the sample components are reduced using hydrogen as a reaction gas to form HCl, HBr, HF, or NH_3. The reaction tube acts as a catalyst to help speed up the reaction.

Conductivity cell

The reacted sample is then swept through a Teflon® transfer line into the conductivity cell. In the conductivity cell, species formed in the reaction tube are dissolved and ionized in a deionized conductivity solvent flowing through the conductivity cell. Different solvents are used depending on the reaction mode. The change in the conductivity, caused by the reacted sample, is measured in the conductivity cell. Any species which is ionized during dissolution gives increased conductivity to the cell.

Figure 1 - Understanding the basic parts and operation of an ELCD enhances an analyst's ability to properly use the detector.



Solvent/ Resin Beds

In order to obtain a good response from the conductivity cell, the solvent flow and pH must be optimized. The sensitivity of the detector is inversely proportional to flow rate. Therefore, higher flow rates can be used where sensitivity is of little concern. The pH of the solvent is controlled by passing it through an ion exchange resin located in the solvent reservoir bottle. The proper resin mixture will provide the correct pH for the solvent. The halogen and sulfur modes are acidic and require an acidic solvent, whereas the nitrogen mode is basic and requires a basic solvent.

Detection Modes

Halogen compounds

The most common use of the ELCD is for the detection of volatile halocarbons in water. The ELCD reduces halogenated compounds in a nickel reaction tube (850-1000°C) to haloacids by mixing them with hydrogen reaction gas. Non-halogenated hydrocarbons are reduced to methane which is non-ionic. The haloacids are dissolved in n-propanol and the change in solvent conductivity is measured at the cell.

Sulfur compounds

In the sulfur mode, air is used as a reaction gas. Sulfur compounds are oxidized in a nickel reaction tube (800°C) producing SO_2 and/or SO_3. Hydrocarbons in the sample are

oxidized to form CO or CO_2. Pure methanol or a methanol/water mixture is the preferred solvent for this mode because it allows ionization of the sulfur compounds but minimizes the formation of CO_2. To eliminate the interference of haloacids from halogenated compounds in the sample, a scrubber is positioned after the reactor, but prior to the conductivity cell. The scrubber is made of stainless steel or copper tubing with several strands of silver wire positioned inside the tubing. The silver complexes with the haloacids and removes them from the sample stream.

Nitrogen compounds

The nitrogen mode reduces nitrogen containing compounds using hydrogen reaction gas in a nickel reaction tube (800°C) to form NH_3. The NH_3 will not completely ionize unless it is dissolved in an aqueous solution. Therefore, water containing a small amount of an organic solvent is recommended for greater sensitivity and better performance. A scrubber containing quartz thread is used for the nitrogen mode to remove other components from the sample.

Operating Hints

ELCD performance depends on the reactor, conductivity cell, and the solvent system. Because the reaction tube is the major part of the reactor, the tube should be replaced routinely. Hydrocarbons and column bleed can coat the inside of the reaction tube and decrease its catalytic activity. A drop in sensitivity, especially of brominated compounds, is a good indication the reaction tube needs to be replaced. Other factors, like baseline instability and ghost peaks, are also an indication the reaction tube may be fouled and requires replacement. A solvent vent, controlled by a solenoid valve, is located between the GC column and the reaction tube. This valve allows venting of the solvent to prevent premature fouling of the reaction tube. It may be necessary to condition a new reaction tube after replacement. Conditioning the tube with the reaction gas (H_2) flowing for 24 hours is recommended. Cooling the reactor before removing the capillary column from the detector is critical. If room air is allowed into the detector while the reactor is still hot, the nickel reaction tube will oxidize. An oxidized nickel reaction tube can result in sensitivity loss and tailing peaks.

The internal volume of the electrolytic conductivity cell is also important in determining ELCD performance. Older ELCDs have large cell volumes that were developed for use with packed columns operated at high flow rates. If these older detectors are used with capillary columns at low flow rates, excessive peak tailing will occur. Newer ELCDs have much smaller conductivity cells which significantly reduce peak tailing, even when operated at lower flow rates.

The Teflon transfer line, located between the reactor and the conductivity cell, requires cleaning and replacement depending on its usage. Clean the transfer line by rinsing with methanol or, for a more thorough cleaning, rinse with a 10% solution of HCl, followed by a methanol rinse. Dry the tubing before reinstalling it. With the time and effort required to clean the transfer line, it may be more cost effective to simply replace it.

Table I - Troubleshooting Hints for ELCDs

Symptom	Remedy
Noisy baseline	Clean transfer line from reactor to cell. Replace reaction tube. Use pure carrier gas and filters. Replace quartz insert.
Peak tailing	Clean conductivity cell/backflush. Increase reactor temperature. Replace reaction tube. Clean transfer line. Replace or cut 10cm off the detector end of the column.
Low response	Replace reaction tube. Clean or replace transfer line. Use correct solvent and replace if necessary. Optimize detector parameters.
High background	Incompatible column phase (F,N) Contaminated gases. Incorrect column installation. Condition column. Condition reaction tube.

Always use high purity solvents (HPLC grade for halogen mode) in the ELCD. The pH and background conductivity of the solvent are maintained by circulation through an ion-exchange resin. It is recommended to change the resin every six months. Problems often associated with the solvent reservoir may be sudden low or negative response, or baseline instability. It is important to maintain the proper solvent pH in the nitrogen mode to avoid the presence of negative peaks. The solvent may be slightly acidic due to trace amounts of CO_2. This can cause neutralization of low levels of NH_3, resulting in negative peaks. One way to avoid this problem is to totally exclude CO_2 from the solvent system by using nonpermeable tubing.

Table I lists some of the common problems experienced with ELCDs and troubleshooting hints.

Electrolytic conductivity detectors are excellent for environmental and pharmaceutical analyses due to their highly selective nature. Because of the selectivity of the ELCD, sample cleanup procedures do not have to be as stringent as with other detectors where interference can be a problem. However, because the ELCD is a more complex detector, frequent maintenance and optimization of detector flow rates is required. Attention to the basic operating hints, as outlined above, will result in a highly sensitive, reliable detection system.

References:

1. Hill, Herbert and Dennis McMinn, ed., *Detectors for Capillary Chromatography*, John Wiley & Sons, New York, 1992.
2. Buffington, Rosemary and Michael K. Wilson, *Detectors for Gas Chromatography - A Practical Primer*, Hewlett-Packard Co., Avondale, PA, 1991.

Peak Performers

The affordable solution for GC leak detection...

Restek's Leak Detective™

- Detects minute leaks not possible with liquid leak detectors.
- Compact, lightweight, hand-held design.
- Lowest cost thermal conductivity leak detector available.
- Contamination-free leak detection.
- Battery or line operated.
- Detects leaks of helium or hydrogen at ≥20µl/min. or ≥200ppm

Restek's new Leak Detective™ is the affordable solution for GC leak detection. Leaks can increase detector noise, cause baseline instability, waste carrier gas, and shorten column lifetimes. The Leak Detective™ allows detection of minute gas leaks which may go undetected by liquid leak detectors.

The compact design of the Leak Detective™ ensures comfortable hand-held operation. Trace leaks of both helium and hydrogen* can be detected. Sensitivity is similar to other models on the market with detectability of helium or hydrogen and leak rates of 20µl/min. or an absolute concentration less than 200ppm Leaks are indicated by an audible alarm, as well as an LED readout. Two 9-volt batteries (included) provide 10-12 hours of continuous operation, or the unit can be used with an AC adaptor (included).

Restek Leak Detective™
cat.# 21607 each

**not designed for use in explosive atmospheres*

Channeltron® 5778 Electron Multiplier

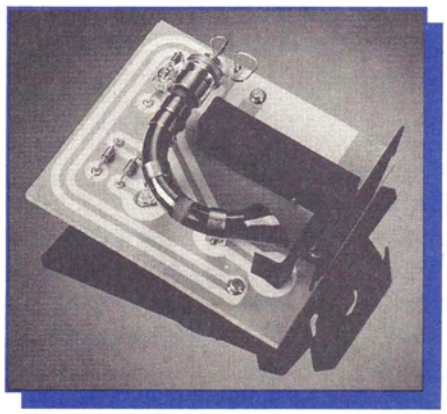
for the HP 5971A and 5972A MSD

The new 5778 provides these benefits over the multipliers originally supplied with HP 5871A and 5972A MSDs:

- 25-50% increase in sensitivity
- 2-5X increase in dynamic range
- Double the lifetime
- Easy, self-aligning installation

The new Channeltron® 5778 Electron Multiplier from Galileo offers increased performance for HP 5971A and 5972A MSDs and the new HP GCD. If your applications demand high sensitivity, extended linear dynamic range, and longer multiplier lifetime, the 5778 Electron Multiplier will meet these requirements.

The 5778 design increases sensitivity through superior signal collection and a reduction of unwanted noise. This sensitivity allows detection of sub picogram and femtogram levels from complex mixtures. The separate input and booster stages of the 5778 provide a linear response and dynamic range which



exceeds the limits of the instrument and of other available multipliers. The patented plug-in, ceramic board-mounted design allows easy installation and assures alignment of the critical ion optics.

Channeltron® Electron Multipliers are available for other mass selective detectors upon request. Please call your local distributor for more information.

Channeltron® 5778 Electron Multiplier: cat.# 21608 each

Inlet Seals with Both Large and Small Holes for HP 5890 GCs

Now Available in Two Different Sized Openings

The inlet seal at the base of the HP 5890 GC injection port comes into contact with the sample and must be changed frequently to prevent adsorption and/or breakdown of active compounds. Restek now offers inlet seals with two different sized openings: 1.2mm and 0.8mm. The 1.2mm inlet seal is recommended for use with Vespel®/Graphite ferrules or when installing two columns using a 2-hole ferrule. The 0.8mm inlet seal is recommended for use with graphite ferrules and single capillary column installations.

Inlet seals with the 0.8mm or 1.2mm opening are available in three styles: stainless steel, Silcosteel®, or gold plated. The gold surface offers better inertness and easier sealing than standard stainless steel. Restek's unique Silcosteel® process places a micron thin layer of fused silica and a deactivation layer over the stainless steel to provide inertness similar to a fused silica capillary column. Both the Silcosteel® and gold plated inlet seals reduce breakdown and adsorption of active compounds.

1.2mm Replacement Inlet Seals
cat# 20390, 2-pk.
cat.#20391, 10-pk.

1.2mm Gold Plated Inlet Seals
cat.#21305, 2-pk.
cat.#21306, 10-pk.

1.2mm Silcosteel® Treated Inlet Seals
cat.#21307, 2-pk.
cat.#21308, 10-pk.

0.8mm Replacement Inlet Seals
cat# 21315, 2-pk.
cat.# 21316, 10-pk.

0.8mm Gold Plated Inlet Seals
cat.# 21317, 2-pk.
cat.# 21318, 10-pk.

0.8mm Silcosteel® Treated Inlet Seals
cat.# 21319, 2-pk.
cat.# 21320, 10-pk.

PRICES SLASHED on XTI®-5 Columns

- Low bleed for GC/MS.
- Thermal stability to 360°C.
- Displays high response factors for active compounds.
- Guaranteed low bleed at maximum temperature.

The technology used to produce XTI®-5 columns increases the polymer stability by 20°C over our standard Rtx®-5 column, giving them the highest operating temperature of any 5% diphenyl/95% dimethyl polysiloxane capillary column available.

The XTI®-5 is inert to the most reactive environmental compounds, and it has the efficiency to resolve closely eluting isomers. Every XTI®-5 column is tested with a specially designed, temperature programmed environmental test mix to ensure that it meets strict performance requirements. In addition, each column is programmed to its maximum temperature and monitored for bleed.

100% Satisfaction Guarantee
We are confident that the XTI®-5 out-performs any competitor's column for environmental analyses or columns specially marketed for use with mass spectrometers. If the XTI®-5 does not consistently provide the highest response factors, lowest bleed, and best thermal stability for priority pollutant analyses, just contact our technical service department. We'll replace the column or give you a complete refund.

Call your local distributor for new price information.

	df(µm)	temp. range	30-meter
0.25mm ID	0.25	-60 360°C	cat.# 12223
	0.50	-60 330°C	cat.# 12238
	1.00	-60 325°C	cat.# 12253
0.32mm ID	0.25	-60 360°C	cat.# 12224
	0.50	-60 330°C	cat.# 12239
	1.00	-60 325°C	cat.# 12254
0.53mm ID	0.50	-60 330°C	cat.# 12240
	1.00	-60 325°C	cat.# 12255
	1.50	-60 320°C	cat.# 12270
	df(µm)	**temp. range**	**15-meter**
0.25mm ID	0.25	-60 360°C	cat.# 12220
	0.50	-60 330°C	cat.# 12235

The Restek Advantage is printed on recycled paper.

THE RESTEK ADVANTAGE

Analyzing Free Fatty Acids Without Derivatization[1]

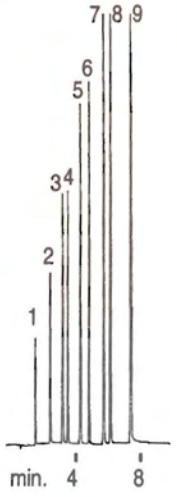
Dietary intake of fats has become an important concern for the health conscious public. Therefore, laboratories involved in analyzing food products are often faced with the difficult task of analyzing fatty acids. The lack of volatility coupled with their adsorptive nature has complicated gas chromatographic analysis of free fatty acids. As a result, many analysts derivatize fatty acids to their methyl ester state. Derivatization increases the volatility of free acids and reduces their activity making them more amenable to GC analysis. However, derivatization adds additional sample prep costs and some uncertainty to the analysis due to the possibility for sample loss or incomplete methylation. Therefore, many analysts would prefer to analyze fatty acids in their free state.

Volatile Fatty Acids

Several capillary column techniques have been developed for the analysis of volatile fatty acids. One approach is to use very thick film non-polar stationary phases for analyzing volatile free acids. Thick film columns are necessary to increase the sample capacity of non-polar phases since they have little affinity for highly polar acids. Figure 1 shows the analysis of C_2 to C_7 organic acids on a 30 meter, 0.53mm ID, 5.0µm Rtx®-1 column. Excellent separation of these volatile acids can be achieved in less than 8 minutes with minimal peak tailing. Another approach for analyzing volatile fatty acids is to use

Figure 1 - A thick film Rtx®-1 column resolves lower molecular weight free fatty acids.

1	acetic acid
2	propionic acid
3	isobutyric acid
4	n-butyric acid
5	isovaleric acid
6	n-valeric acid
7	isocaproic acid
8	caproic acid
9	heptanoic acid



30m, 0.53mm ID, 5.0µm Rtx®-1 (cat.# 10179)
0.2µl injection of a 10-20ng/µl free fatty acid standard in water
Direct injection using a Uniliner®.
Oven temp.: 60°C to 180°C @ 15°C/min.
Inj. & det. temp.: 250°C **Carrier Gas:** hydrogen
Linear velocity: 50cm/sec. (flow rate: 6cc/min.)
FID sensitivity: 4×10^{-11} AFS

(article is continued on page 2)

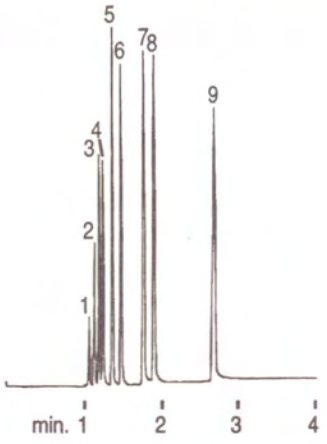
in this issue...

Another approach for analyzing volatile fatty acids is to use highly polar bonded Carbowax® capillary columns that have been deactivated for acidic compounds. The strong affinity of the acids on this polar column results in excellent separation of peaks, even with a 15 meter column. The acidic deactivation of the Carbowax® column produces excellent peak shape for these free fatty acids.

Moderately polar stationary phases can also be used for the analysis of volatile free fatty acids. Figure 2 shows the analysis of C_2 to C_7 organic acids on a 30 meter, 0.25mm ID, 0.25µm Rtx®-200 column. This trifluoropropylmethyl stationary phase shows good separation and peak shape of these volatile acids and the analysis time is less than 3 minutes, making the Rtx®-200 an excellent screening column.

Figure 2 - Fast analysis of low molecular weight free fatty acids can be achieved with an Rtx®-200 column.

(same peak list as Figure 1)



30m, 0.25mm ID, 0.25µm Rtx®-200 (cat.# 15023)
0.8µl split injection of a free fatty acid standard
Concentration approximately 10 to 20ng/µl
Oven temp.: 90°C isothermal
Inj. & det. temp.: 250°C **Carrier Gas:** hydrogen
Linear velocity: 40cm/sec. (flow rate: 1.4cc/min.)
FID sensitivity: 4 x 10^{-11} AFS **Split vent:** 40cc/min.

Saturated & Unsaturated Fatty Acids

Polar stationary phases are also capable of resolving saturated and unsaturated fatty acids. Figure 3 shows the analysis of saturated and unsaturated fatty acids on a 30 meter, 0.53mm ID, 0.25µm Stabilwax®-DA column. Palmitic acid (C16:0) can easily be resolved from Palmitoleic acid (C16:1), and Stearic acid (C18:0) can be resolved from Oleic (C18:1), Linoleic (C18:2), and Linolenic acid (C18:3) on this column.

Methylation of fatty acids for GC analysis may not always be necessary. Several options are available for analyzing fatty acids in their free form using capillary columns. Column selection will depend on the molecular weight range of fatty acids and the resolution required to separate saturated from unsaturated forms.

Product Listing

Rtx®-1
30m, 0.53mmID, 5.0µm
cat.#10179

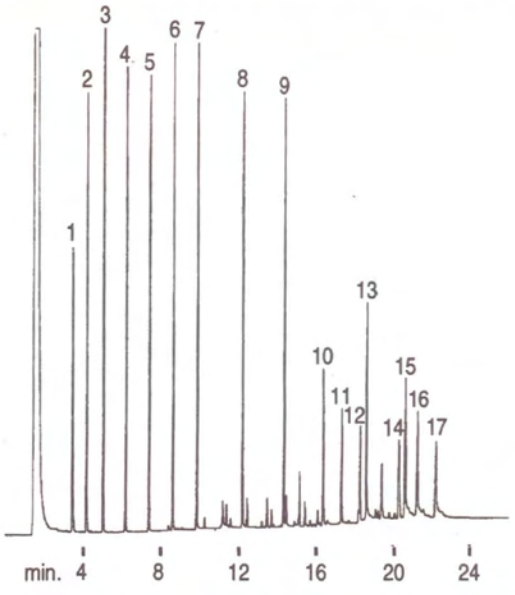
Rtx®-200
30m, 0.25mmID, 0.25µm
cat.#15023

Stabilwax®-DA
15m, 0.53mmID, 1.0µm
cat.#11052

30m, 0.53mmID, 0.25µm
cat.#11025

[1]This article is a partial reprint from *The Restek Advantage*, Vol. 3 No. 5, September 1992, p. 8-9.

Figure 3 - Saturated and unsaturated free fatty acids can be separated on a 30-meter Stabilwax®-DA column.



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

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

Cover Your Bases with Rtx®-5 Amine Columns

The Rtx®-5 Amine column has been developed with a unique deactivation technology that reduces adsorption and improves the response of basic compounds. Analyses that previously required derivatization or another analytical technique such as HPLC can now be performed on the Rtx®-5 Amine column. Clinical chemists can now save time and money when analyzing pharmaceutical compounds with the Rtx®-5 Amine. (Related article on page 5.) Because basic samples can be analyzed in their free base or salt form with the Rtx®-5 Amine, derivatization is not necessarily required. This column can also be used to analyze neutral and mildly acidic compounds with the same efficiency as our standard Rtx®-5 columns.

The Rtx®-5 Amine can be used to analyze a wide variety of basic compounds such as alkylamines, diamines, triamines, and nitro-containing heterocycles. Additionally, the Rtx®-5 Amine permits the analysis of neutral and weakly acidic analytes. Figure 1 shows the simultaneous analysis of several basic compounds and phenols in water. Excellent peak symmetry of both the basic and acidic compounds is obtained with the Rtx®-5 Amine.

Chemists using guard columns for the analysis of basic compounds frequently experience peak tailing and low recovery. This problem occurs because the deactivated surface of the guard column can be adsorptive to basic compounds. Restek now offers base deactivated guard columns for a completely inert, basic pathway for basic compound analysis. These base deactivated guard columns are made with the same technology as the Rtx®-5 Amine columns to guarantee excellent response of basic compounds.

If your lab is analyzing amines or other strongly basic compounds, the Rtx®-5 Amine is guaranteed to give you consistent, reproducible results from run-to-run. The Rtx®-5 Amine can also be used for samples where both basic and weakly acidic compounds must be analyzed. Choose Rtx®-5 Amine columns and base deactivated guard columns for your next basic compound analysis. These products were designed to meet your chromatographic challenges!

Product Listing

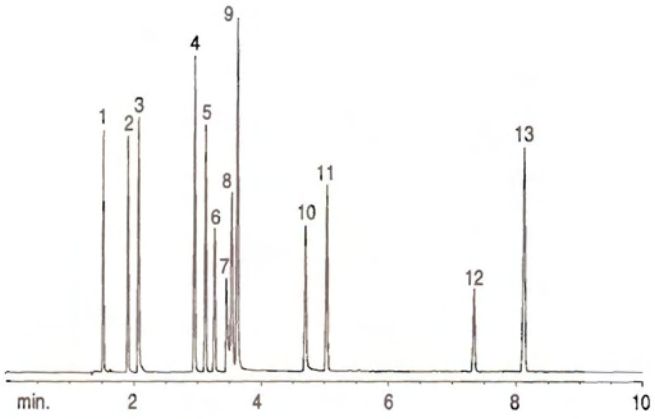
Rtx®-5 Amine Columns

	df(µm)	15-meter	30-meter
0.25mm ID	0.50	12335	12338
	1.00	12350	12353
0.32mm ID	1.00	12351	12354
	1.50	12366	12369
0.53mm ID	1.00	12352	12355
	3.00	12382	12385

New! Base Deactivated Guard Columns

5m, 0.25mm ID	cat.# 10000, each
5m, 0.32mm ID	cat.# 10001, each
5m, 0.53mm ID	cat.# 10002, each

Figure 1 - The versatility of the Rtx®-5 Amine column permits the analysis of both basic and weakly acidic compounds - simultaneously!



1. diethylamine
2. pyridine
3. morpholine
4. phenol
5. aniline
6. 2-chlorophenol
7. diethylenetriamine
8. octylamine
9. 1-methyl-2-pyrrolidinone
10. 2-nitrophenol
11. 2,6-dimethylaniline
12. nicotine
13. 2-nitroaniline

30m, 0.32mm ID, 1.0µm Rtx®-5 Amine column (cat.# 12354)
1.0µl split injection of miscellaneous amines and phenols in water, on-column concentration: 22ng

Oven temp.: 120°C to 220°C @ 10°C/min.
Inj. & det. temp.: 305°C **Detector:** FID
Carrier gas: hydrogen **Linear velocity:** 38cm/sec. set @ 120°C
Detector sensitivity: 6.4 x 10^{-11} AFS **Split ratio:** 25:1

Clinical Corner

Analysis of Antiepileptic Drugs by Capillary GC

Epilepsy is a central nervous system disorder characterized by seizures resulting from excessive electrical discharges in the brain. Epileptic syndromes can be categorized into two main divisions. Partial seizures are limited to one part of the brain and are manifested by convulsions of single muscles or muscle groups. Generalized seizures affect the entire brain and can result in varying degrees of impaired consciousness and major convulsions that involve all body muscle groups. Status epilepticus is a prolonged period of seizure activity, either partial or generalized, during which normal consciousness is not regained, usually greater than 30 minutes[1]. Untreated status epilepticus is associated with higher morbidity rates and can result in permanent brain damage.

Measuring concentrations of antiepileptic drugs in serum or plasma is especially important in establishing control of seizure activity, monitoring patient compliance and minimizing side effects.

Drugs used to control epileptic disorders focus on inhibiting the initial discharge of electrical stimuli from seizure foci or preventing the spread of uncontrolled electrical activity to other neurons[2]. Choice of drug therapy to control convulsions is dependent upon the type of seizure experienced by the patient as well as other associated clinical factors. Hydantoins, succinimides, barbiturates and a few specific, non-related compounds make up the bulk of drugs used in the long term treatment of epileptic disorders.

All of the antiepileptic drugs have polar characteristics through the inclusion of keto, azo and carboxylic acid functional groups. Absence seizures are treated most frequently with ethosuximide or valproic acid. However, concerns over hepatotoxicity limit the use of valproic acid in children. Carbamazepine and diphenylhydantoin are the agents of choice in the treatment of generalized tonic-clonic seizures.

Measuring concentrations of antiepileptic drugs in serum or plasma is especially important in establishing control of seizure activity, monitoring patient compliance and minimizing side effects. Therapeutic concentrations of most antiepileptic drugs are in the µg/ml range and extraction of antiepileptic drugs from biological matrices is straightforward. Low limits of detection are easily achieved with commonly used detectors such as FID.

Despite being characterized as polar compounds, antiepileptic drugs can be analyzed without being derivatized. Intermediate polarity columns are recommeded over non-polar columns to obtain symmetrical peak shapes and maximum resolution. Figure 1 (on page 5) shows the analysis of a set of antiepileptic drugs on an Rtx®-20 column. All compounds exhibit good peak shape and resolution except for carbamazepine and diphenylhydantoin, which are only partially resolved. By using another intermediate polarity column with a different selective mode of retention, this coelution can be resolved without compromising the resolution of any of the other compounds in this group. Figure 2 (on page 5) shows the analysis of the same group of antiepileptic drugs using the same chromatographic conditions on an Rtx®-1701 column. Changes in retention time and relative elution order allow both of these columns to be used for screening and confirming the presence of antiepileptic drugs in a dual column configuration.

Monitoring the concentrations of antiepileptic drugs is critical in providing optimal drug therapy for epileptic patients. Capillary gas chromatography provides a quick and versatile method to screen and quantitate a wide variety of antiepileptic drugs. Intermediate polarity columns like the Rtx®-20 and the Rtx®-1701 provide the best overall peak shape and resolution for these compounds.

References

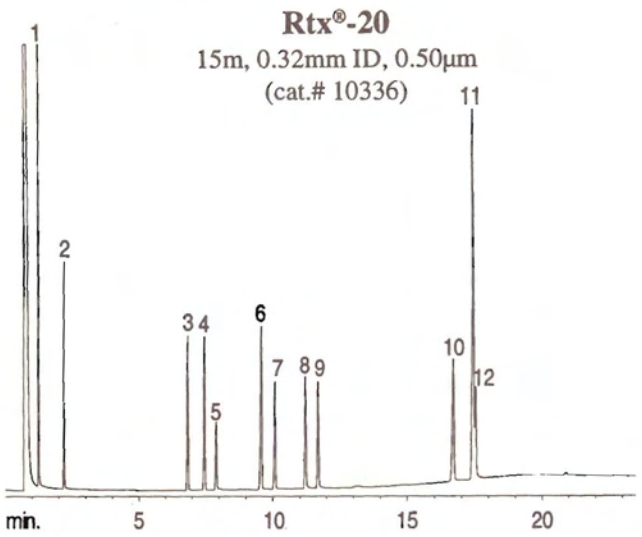
1. Martindale, *The Extra Pharmacopeia*, 30th Edition, 1993, p. 292.
2. Goodman and Gillman's, *The Pharmacological Basis of Therapeutics*, 8th Edition, p. 438-439.

Product Listing

Rtx®-20
15m, 0.32mm ID, 0.50µm
cat.# 10336, each

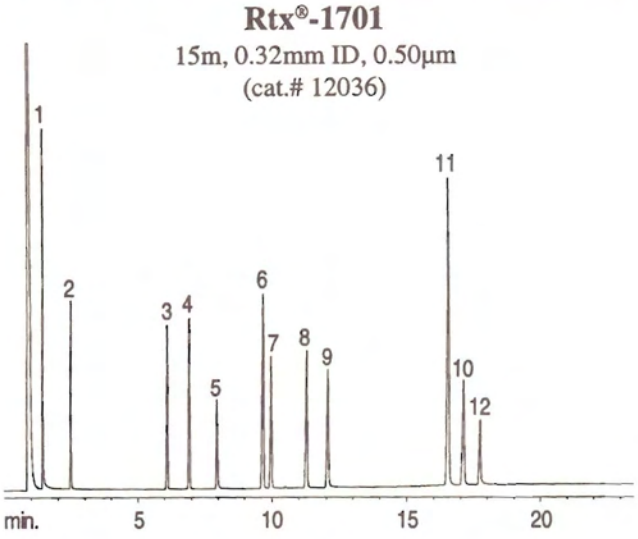
Rtx®-1701
15m, 0.32mm ID, 0.25µm
cat.# 12036, each

Figure 1 - The Rtx®-20 shows good peak shape and resolution for all compounds in the antiepileptic drug sample except for a coelution of carbamazepine and diphenylhydantoin.



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

Figure 2 - Analysis of the same compounds on an Rtx®-1701 resolves the coelution of carbamazepine and diphenylhydantoin without compromising resolution of the other compounds.

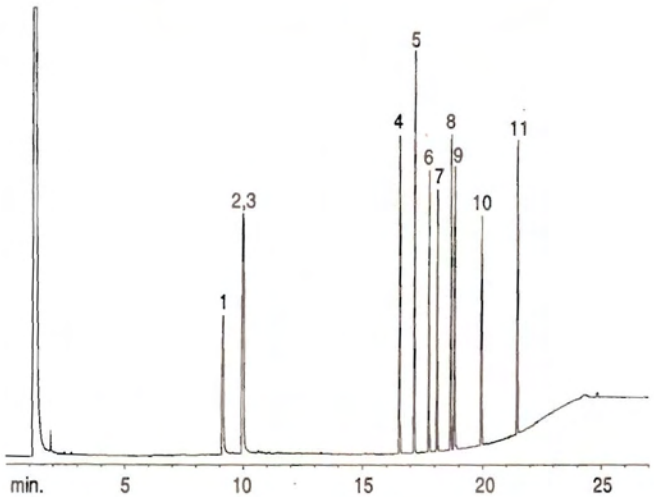


1.0µl split injection of Antiepileptic Drugs
on-column concentration: 30ng
Oven temp.: 150°C to 280°C @ 7°C/min. (hold 5 min.)
Inj./det. temp.: 250°C/270°C
Detector: FID **Carrier gas:** helium
Linear velocity: 30cm/sec. set @ 150°C
Detector sensitivity: 1.28 x 10^{-10} AFS
Split ratio: 30:1

Analysis of Antihistamines on an Rtx®-5 Amine Column

Antihistamines are common ingredients in over-the-counter cold medicines. Antihistamines typically have amine functional groups in their structure and are classified as basic compounds. Because of their basic nature, antihistamines often exhibit adsorption or tailing peaks on poorly deactivated columns. With the Rtx®-5 Amine, excellent peak symmetry and enhanced response is obtained for eleven antihistamines in their salt form, as illustrated in Figure 1.

Figure 1 - Analyze antihistamines in their salt form on the Rtx®-5 Amine.



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

30m, 0.32mm ID, 1.0µm Rtx®-5 Amine column (cat.# 12354)
1.0µl split injection of antihistamines in methanol,
on-column concentration: 22ppm
Oven temp.: 130°C (hold 5 min.) to 305°C @ 10°C/min. (hold 5 min.)
Inj. & det. temp.: 305°C
Detector: FID
Carrier gas: hydrogen
Linear velocity: 43.3cm/sec. set @ 130°C
Detector sensitivity: 6.4 x 10^{-11} AFS
Split ratio: 27:1

See page 3 for more information on the Rtx®-5 Amine column.

Rtx®-1 - A New Bonded Packed Column for Simulated Distillation

- Rtx®-1 bonded packed column requires minimal conditioning.
- Meets or exceeds all specifications of ASTM D2887-93.
- Stable baseline to 350°C and repeatable RT's "right from the box".
- Deactivated Silcosteel® tubing and Silcoport™ packing for high inertness.
- Column lifetime superior to existing Sim Dist columns.

Simulated Distillation (Sim Dist), according to The American Society for Testing Materials (ASTM) test method D2887-93, can be performed using either packed or capillary columns. Some of the advantages of capillary columns are the columns are preconditioned so hey can be used after only minimal conditioning, and the bonded stationary phases exhibit stable baselines and retention times. There are many laboratories, currently using packed columns, which would like to take advantage of bonded phases but do not have GC equipment which can be easily converted for use with capillary columns. Restek's Rtx®-1 Sim Dist column is the first in a new generation of bonded packed columns having superior inertness and stability compared to conventional packed columns. Improvements are obtained by preparing the columns with Silcosteel® tubing and bonding the Rtx®-1 stationary phase to a highly deactivated Silcoport™ support. The column dimensions and packing (1/8" Silcosteel® with 10% Rtx®-1 on Silcoport™) are designed to exceed all requirements specified in ASTM Test Methods D2887-93 and D3710-93.

Table I - Retention Time Repeatability for Calibration after only 30 minutes conditioning.

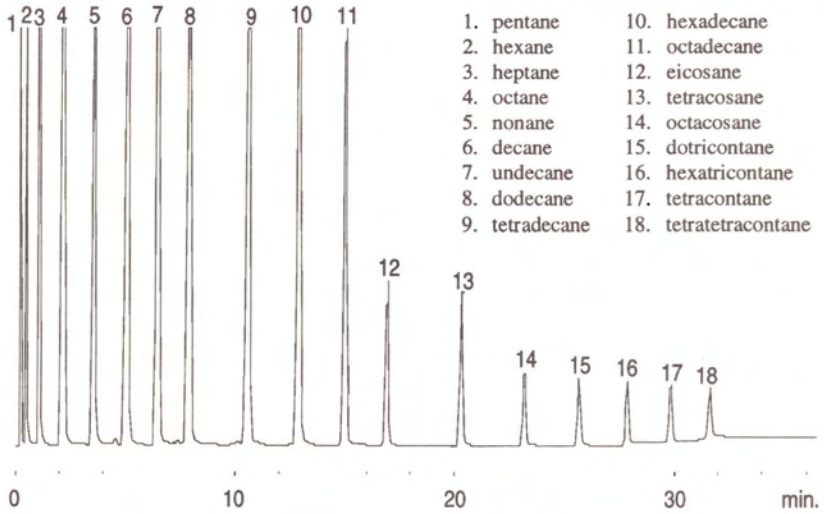
Hydrocarbon	Min Rt	Max Rt	Avg. RT	Stand. Dev.
C_5	0.241	0.243	0.242	0.001
C_6	0.493	0.497	0.495	0.002
C_{10}	5.746	5.765	5.752	0.005
C_{20}	18.482	18.491	18.486	0.004
C_{28}	25.093	25.103	25.098	0.004
C_{40}	32.160	32.171	32.166	0.004
C_{44}	34.316	34.328	34.326	0.007

n=9

Bonded stationary phases require minimal conditioning and give stable baselines and retention times "right from the box"

Simulated distillation is a gas chromatographic procedure which differs from typical GC analyses requiring peak resolution and integration. The sample is analyzed using a linear temperature program so that the retention time of the hydrocarbons are proportional to their boiling points. The sample boiling range distribution is calculated by comparing the sample area and its retention time with that of an alkane calibration standard. In order for the calibration to be valid for sample analysis, it is crucial retention times be repeatable until the next calibration is performed. Figure 1 is an example of the analysis of the Restek D2887 Calibration Mix (cat.# 31222) illustrating the typical pattern obtained for the alkanes under temperature programmed conditions. To demonstrate the stability of the Rtx®-1 column, a series of calibration standards were analyzed after only 30 minutes of conditioning at 350°C. Table I shows the excellent retention time repeatability obtained with the column, indicating the column is suitable for sample analysis after minimal conditioning.

Figure 1 - C5 to C44 calibration analysis after only 30 minutes conditioning.

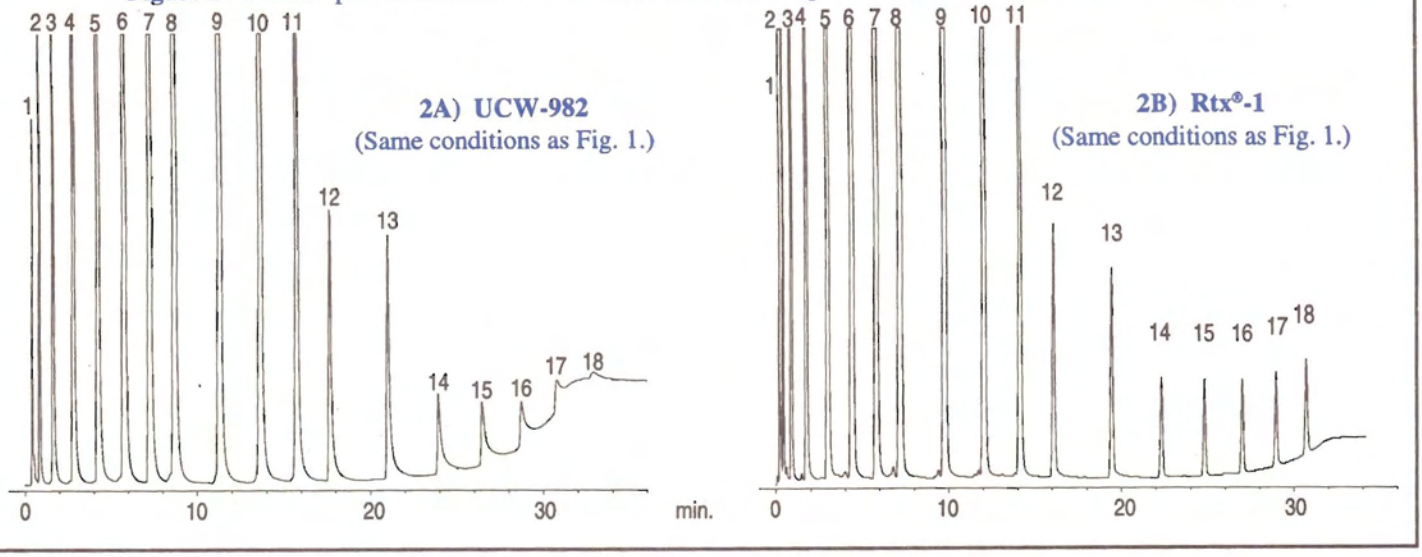


25" x 1/8" Rtx®-1 Sim Dist 2887 Silcosteel® column
1.0µl direct injection of D2887 Calibration Mix (cat.# 31222)

Oven temp.:	35°C to 350°C @ 10°C/min. (hold 5 min.)
Inj. & det. temp.:	350°C
Carrier gas:	helium @ 25ml/min.
FID sensitivity:	256 x 10^{-11} AFS

Column bleed is another important consideration for selecting a Sim Dist column. The baseline must be stable and free of any artifacts during the temperature program up to 350°C. Although baseline subtraction is permitted in the method, this compensation will produce errors if the baseline rise is not consistent. Conventional packed columns require up to 14 hours of conditioning and frequent updating of the baseline compensation run because the stationary phase is not bonded. Rtx®-1 columns, however, exhibit stable and reproducible baselines with just 30

Figure 2 - Bonded packed columns exhibit lower bleed and longer lifetimes after 170 temperature cycles.



minutes of conditioning. This results in fewer baseline blanks and less frequent calibration increasing laboratory productivity.

Bonded stationary phases extend column lifetime

The Rtx®-1 stationary phase is bonded to the diatomite particles resulting in an immobilized coating which is resistant to solvents and lower in bleed than conventional packings. Since the packing is preconditioned, there is no need for extended conditioning in the GC oven. This conditioning must be carried out in an oxygen-free system, otherwise high bleed will result. Since GC systems often have leaks or carrier gas which contain oxygen, it is more likely that conventional columns will be damaged during the conditioning process. Figure 2A shows a conventional UCW-982 column after only 170 temperature cycles, demonstrating higher bleed and more tailing than the Rtx®-1 Sim Dist column (Figure 2B). Although actual column lifetimes depend upon the system and types of samples analyzed, the bonded stationary phase should result in longer lifetime than its non-bonded equivalent.

Rtx®-1 Sim Dist columns have equivalent polarity to OV-101 and UCW-982

In order for a stationary phase to be acceptable for ASTM methods, the column must not exhibit selective retention for aromatic hydrocarbons compared to aliphatic hydrocarbons. This is an important test because if the polarity of a column is different, the boiling point results will demonstrate a bias, especially for highly aromatic samples. The "polarity" of the bonded Rtx®-1 column was compared with OV-101 and UCW-982, two of the most common stationary phases currently used for simulated distillation. The results of the calculated boiling points for aromatics compared to the published boiling points appear in Table II. All three silicone columns tested are essentially identical in they elute aromatics at a slightly lower temperature than the alkanes. This confirms the polarity of the Rtx®-1 column is equivalent, and the boiling range values obtained will agree with OV-101 and UCW-982 columns.

Table II - Comparison of bonded and conventional packed columns indicates no polarity differences.

Aromatic Hydrocarbon	Published BP[1] (°C)	Calculated BP		
		Rtx®-1	UCW-982	OV-101
benzene	80	81.3	82	80.3
p-xylene	139	138.6	140.2	137.7
naphthalene	218	204.6	206.9	204.3
acenaphthylene	280	252.7	255.6	252.2
anthracene	342	304.1	307.2	303.4
chrysene	447	385.6	389.2	384.9
dibenzo(a,h)anthracene	524	452.3	455.7	450.4

Rtx®-1 is an excellent choice for Sim Dist using packed columns

Simulated Distillation is one of the most common GC analyses performed in the petroleum laboratory. ASTM test methods D2887 and D3710 can be performed with either packed or capillary columns, but until now the benefits of bonded phases were available only to capillary users. The Rtx®-1 packed column uses a bonded stationary phase which is immobilized on Silcoport™, a specially deactivated support. The columns are prepared using Silcosteel® tubing for inertness unavailable with conventional metal tubing. Rtx®-1 bonded packed columns require minimal conditioning and give stable baselines and retention even after only 30 minutes of operation at 350°C. If your laboratory has been looking for a better solution to Sim Dist analysis, Restek's Rtx®-1 packed columns are the answer.

Product Listing

Rtx®-1 Sim Dist 25" x 1/8"
Silcosteel® Packed Column
cat.# 80000, each

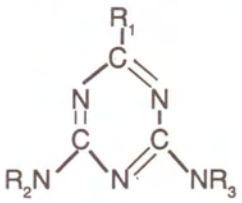
D2887 Calibration Mix
cat.# 31222, each cat.# 31322, 10-pk.

D3710 Calibration Mix
cat.# 31223, each cat.# 31323, 10-pk.

Analysis of Triazine Herbicides

Triazine herbicides are a class of herbicides that have risen in popularity and usage over the last decade. Because of their low toxicity towards mammals, they are not considered to be a threat to the environment. However, triazine herbicides are relatively stable and can be detected in the environment for long periods of time following their application. Due to their long residence time, monitoring for the presence of triazine herbicides has become a concern. The Environmental Protection Agency (EPA) has addressed these issues by drafting a method for the determination of triazine herbicides in industrial and municipal wastewater.

Figure 1 - Structure of diamino-s-triazine.

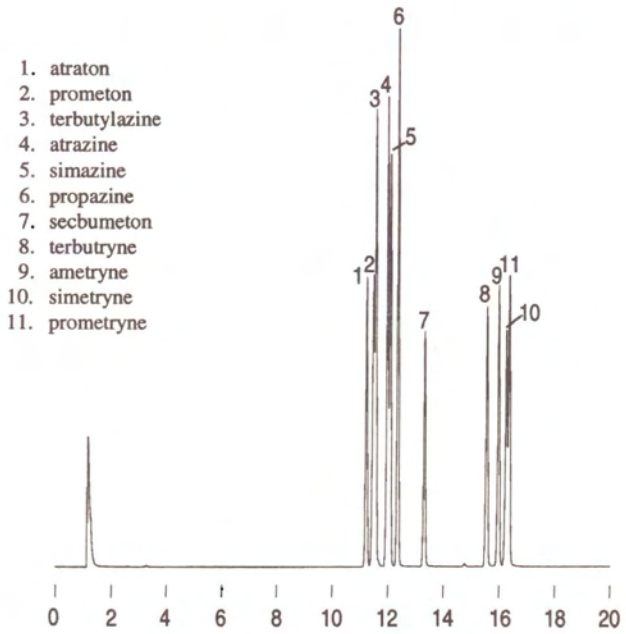


EPA Method 619 is a gas chromatographic method designed for the analysis of triazine herbicides using packed columns. This method can be modified for use with capillary columns. Resolving the eleven analytes for this method, even with highly efficient capillary columns, can be very difficult. The structures for all of the triazine herbicides are very similar and most common stationary phases do not offer the polarity or selectivity needed to obtain efficient resolution of these compounds. By matching the chemical characteristics of these compounds to stationary phases with selective retention mechanisms, better peak shape and resolution can be achieved.

Triazine herbicides are referred to as s-triazines, meaning they are symmetrical in structure. Triazines that have greater bioactivity generally contain halogenated (R1) and diamino (R2 and R3) functionalities (Figure 1). These herbicides are based on a six membered ring containing nitrogen constituents at the 1,3 and 5 positions. The electron density resulting from the inclusion of the nitrogen in the ring and the diamino substituted groups imparts significant polarity to these compounds. The degree of polarity will change depending on the functional groups present at either the R1, R2 or R3 substitution sites. Columns containing stationary phases with intermediate polarity are better suited for these compounds.

Figure 2 shows the analysis on a Rtx®-50 column (50% methyl-50% phenyl polysiloxane). The Rtx®-50 is a common intermediate polarity stationary phase used for the analysis of pesticides and herbicides. It completely resolves five compounds and partially resolves the remaining six; thus, the Rtx®-50 can effectively be used for the primary analytical column. Figure 3 shows the same compounds analyzed on a Rtx®-200 column. The Rtx®-200 is a trifluoropropylmethyl polysiloxane that has unique selectivity for compounds containing lone pair electrons. The Rtx®-200 completely resolves four compounds, partially resolves five, with two remaining unresolved. The elution pattern for the Rtx®-200 differs greatly from the Rtx®-50 making it an excellent choice as a confirmational column.

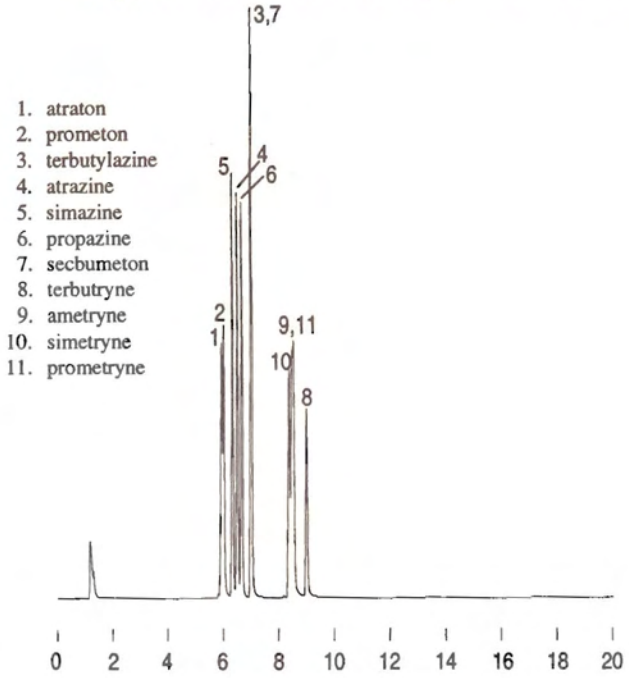
Figure 2 - The Rtx®-50 is an excellent choice for the primary column in triazine analysis.



Rtx®-50
30m, 0.53mm ID, 0.50µm
cat.# 10540, each

0.5µl direct injection of EPA 619 - triazine herbicides
on-column concentration: 50ng
Oven temp.: 150°C to 250°C @ 4°C/min. (hold 5 min.)
Inj./det. temp.: 250°C/275°C
Detector: TSD
Carrier gas: helium
Linear velocity: 40cm/sec. set @ 150°C

Figure 3 - The elution order change of the Rtx®-200 makes it a good secondary column choice.



Rtx®-200
30m, 0.53mm ID, 0.50µm
cat.# 15040, each

0.5µl direct injection of EPA 619 - triazine herbicides
on-column concentration: 50ng
Oven temp.: 150°C to 250°C @ 4°C/min. (hold 5 min.)
Inj./det. temp.: 250°C/275°C
Detector: TSD
Carrier gas: helium
Linear velocity: 40cm/sec. set @ 150°C

Used together, these columns combine to help the analyst positively identify and quantitate the compounds of interest. Since both columns are operated with the same temperature program conditions, simultaneous confirmation can be used. Several techniques for simultaneous dual column confirmation are available including a "Y" Press-Tight® Connector, a dual column direct injection tee, or a 2-hole ferrule.

Triazine herbicides are commonly applied to agricultural fields containing corn, apples, grapes, etc. Their stability increases their residence time in the environment, creating a general concern for their potential hazards. The Rtx®-50 and Rtx®-200 columns enhance the performance of triazine herbicide analyses. These columns offer a basis of quality that leads to accurate, reproducible results as required in EPA Method 619.

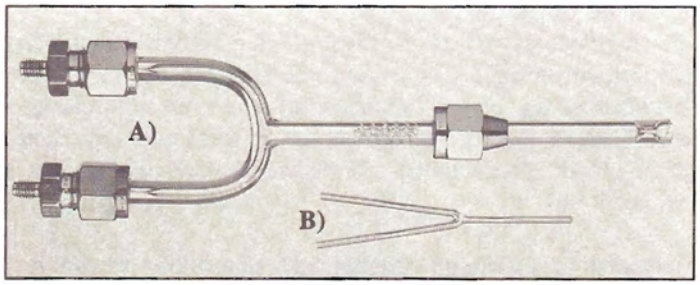
Reference
Kearney, P.C. and Kaufman, D.D. *Herbicides-Chemistry, Degradation, and Mode of Action.* 2ed., Marcel Dekker, Inc., 1969.

Product Listing

Rtx®-50
30m, 0.53mm, 0.50µm
cat.# 10540, each

Rtx®-200
30m, 0.53mm, 0.50µm
cat.# 15040, each

Products for Dual Column Analysis



A) Dual Column Direct Injection Tee Kit (includes all fittings/ferrules): cat.# 20412, kit
Replacement Tee: cat.# 20411, each
Replacement ferrules:

0.5mm ID graphite:	0.8mm ID graphite:
cat.# 20201, 10-pk.	cat.# 20202, 10-pk.
cat.# 20228, 50-pk.	cat.# 20224, 50-pk.

1/4" graphite cat.# 20210, 10-pk.

B) Universal "Y" Press-Tight® Connectors
- Split sample flow onto two different columns.
- Split a single column flow into two different detectors.
- Perform confirmational analysis with a single injection.

cat.# 20405, each
cat.# 20406, (3-pack)

Also Available:

Restek's new Dual Column *mini*-Lam™ Direct Injection Tee Kit
cat.# 20436, kit
Call your local distributor to request the October 1994 International Version of *The Restek Advantage* for more information.

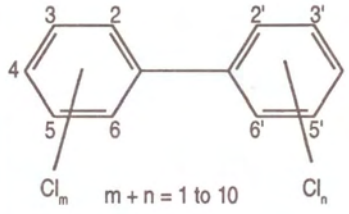
Polychlorinated Biphenyls in Used Engine Oil

Production of polychlorinated biphenyls began in the early part of this century and proved very useful in a variety of applications. They were used in transformers, paints, printing inks, and pesticides. PCBs are also by-products of many processes such as the manufacturing of chlorinated solvents and chlorinated benzenes. They were primarily synthesized by chlorination of biphenyl with chlorine gas.[1] The world's primary producer was Monsanto. They produced complex mixtures of PCBs under the tradename Aroclor®. A four digit numbering system was assigned to the mixtures. The first two numbers indicate the number of carbon atoms and the second numbers provide the weight percent of chlorine. PCBs were manufactured by other companies under trade names like Clophen (Bayer, GFR), Phenoclor (Caffaro, Italy), Fenclor (Prodelec, France), etc.[2] PCBs or Aroclors® are very stable species and do not readily degrade in the environment. Consequently, their wide use resulted in their regular presence in soil, water, and air. Due to this stability, they tend to be present in much of the food chain. The toxicity level of PCBs is still not clearly known--some being more lethal than others. In 1976, PCBs became regulated under the Toxic Substances Control Act[3] and disposal became a major consideration. Monsanto stopped producing Aroclors® in 1977.

Identification of PCBs

The general structure of PCBs is shown in Figure 1. There are 209 congeners, 10 homologs, and 1 to 46 isomers. The commercial production of PCBs resulted in mixtures or blends. Consequently, identification is based more on the type of mixture, eg., Aroclor® 1232, Aroclor® 1254 than on individual PCBs. This can be difficult and identification relies heavily on pattern recognition.

Figure 1 - Structure of PCBs.



Even though PCBs are relatively stable, some microbial and environmental degradation can occur making it more difficult to ascertain the parent Aroclor®. PCBs are hydrophobic and with their low vapor pressure tend to accumulate in soil sediments. In the soil, anaerobic microbes can attack the PCBs, dechlorinating them primarily at the meta and para positions. The extent of PCB dechlorination by microbial activity is dictated by environmental surroundings. Variables such as the presence of oils, grease, sulfur, and metals govern how and if anaerobic microbial dechlorination will occur.[4]

Environmental degradation involves photolysis. The monochlorobiphenyls can have half lives as little as a few days. On the other hand, the half life of pentachloro-biphenyl can be as long as a few months.[5] Any degradation can change the "fingerprint" of the Aroclor®, making identification a very difficult process.

Clean-up is Crucial

Since PCBs were widely used in a variety of applications and remain prominent in the environment, their presence is visible in almost every sample matrix--pesticide residue, milk, waste oils, fish, etc. Complicating a PCB blend with a waste oil matrix can make it virtually impossible to correctly identify and/or quantify. A series of clean-up steps are vital in order to properly identify the PCB mixture. A typical waste oil sample can include not only used motor oil, but sediment and other non-volatile components. The typical mode of PCB analysis is capillary GC using an Electron Capture Detector (ECD). In order to produce acceptable chromatography, the above interferents need to be removed and concentration levels should typically be in the ppb range. Even though the ECD is predominantly responsive to halogenated compounds, severe interferences, such as large amounts of hydrocarbons (ng concentrations), will disrupt the integrity of the chromatography. Figure 2 shows a 2µl injection of PCBs in waste oil with little sample preparation. Peak broadening is a dominant factor producing an almost packed column type chromatogram. There is no indication of an Aroclor® fingerprint. Figure 3 shows the same sample after several clean-up steps. Now the characteristic Aroclor® pattern is readily apparent. Both samples were spiked with decachlorobiphenyl as the internal standard. The 5-point calibration curve indicated the retention time of DCB to be 19.038 minutes (an additional amount of DCB was added to confirm the retention time of DCB). The chromatogram in Figure 2 shows a distinctive shift in the retention time of DCB indicating that significant amounts of interferents such as motor oils can cause peak shifting. This phenomenon can result in incorrect component identification and unreliable quantitation. After proper sample clean-up, the resulting chromatogram in Figure 3 can be properly identified and quantified. The retention time for DCB (19.038 minutes) is well within its retention window.

Common Clean-up Techniques

Several methods exist for the clean-up of PCB type samples and are well documented. The clean-up of waste oils can be particularly demanding. Figure 2 indicated a major source of interference from oil. Diluting the sample in hexane or isooctane, then mixing it with concentrated sulfuric acid removes much of the oil by oxidation.

Even after acid clean-up, contamination can still be present. It may be necessary to treat the sample with a magnesium silicate slurry (Florisil). Shake the sample mixture with Florisil, allow the magnesium silicate to settle, remove the final solution, and then analyze the sample. If the sample still exhibits interferences, the sample should undergo a silica gel slurry clean-up, following the same procedure as the Florisil method.

Figure 2 - Inadequate sample clean-up results in poor chromatography and loss of resolution.

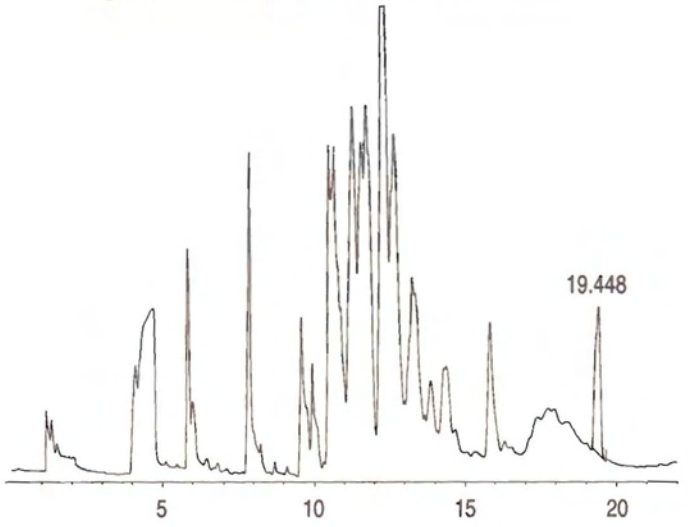
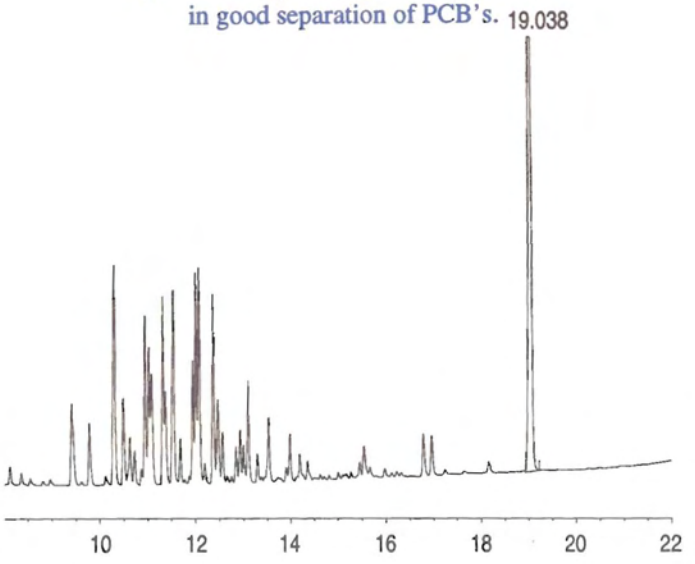


Figure 3 - Proper sample clean-up results in good separation of PCB's.



Another step that can be taken for dirty waste oils is a magnesium silicate column clean-up. A 10ml disposable pipet is plugged with glass wool, followed with anhydrous sodium sulfate, magnesium silicate, sodium sulfate, and moistened with some hexane. Filter the sample solution through the column. Collect the eluant in a vial. The sample should now be completely free of oil contamination.

If the sample also contains elemental sulfur, the impurity will be seen as a large peak on the chromatogram in the early portion of the analysis. Mixing the sample with copper powder or mercury and following it with an acid-wash will remove the sulfur. Depending on the degree of contamination some or all of these steps may be necessary to prepare the sample for analysis. More detail on these clean-up procedures is discussed in EPA Methods 3620, 3630, and 3660.

Column Choice

After clean-up, PCB samples can be effectively analyzed on a 30m, 0.32mm ID, 0.25µm XTI®-5 (5% phenyl / 95% dimethyl polysiloxane) capillary column. Since Aroclors® are very complex samples, a high resolution capillary column such as the XTI®-5 ensures that the Aroclor® will be identified correctly. The high degree of the XTI®-5 columns' reliability guarantees maximum resolution and efficiency. The thermal stability of the XTI®-5 (360°C maximum temperature) allows faster elution of higher boiling contamination.

The analysis of PCB mixtures can be additionally complicated by contamination, photodegradation of the PCBs, dechlorination by anaerobic microbes, and more than one Aroclor® present in the sample. Knowledge of the sample's origin, an effective clean-up procedure, and a high resolution capillary column will significantly decrease the problems associated with PCB analyses.

References:

1. Abramowicz, Daniel A., Brennan, Michael J., Van Dort, Heidi M., and Gallagher, Edith L., *Environ. Sci. Technol.*, Vol. 27, No. 6, 1993, p. 1125.
2. *Analytical Chemistry of PCBs*, Erickson, Michell D., Lewis Publishers,Inc.,1992, p.18.
3. *Analytical Chemistry of PCBs*, Erickson, Michell D., Lewis Publishers,Inc.,1992, p.2.
4. Alder, Alfredo C., Häggblom, Max M., Oppenheimer, Stephanie R., Young, L.Y., Environ. *Sci. Technol.*, Vol. 27, No. 3, 1993, pp. 530-538.
5. *Analytical Chemistry of PCBs*, Erickson, Michell D., Lewis Publishers,Inc.,1992, p.37.

PRODUCTS

XTI®-5 30m, 0.32mm ID, 0.25µm cat .# 12224, each

Aroclor® Standards

1000µg/ml each in 1ml hexane	Individual	Individual w/data pack	5-pk.	5-pk. w/data pack	10pk. w/data pack
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® 1016/1260	32039	32039-500	32039-510	32039-520	32139

Hints for the Capillary Chromatographer

Selecting the Proper Ferrule for Capillary Columns[1]

Proper ferrule selection is critical for capillary column installation. Characteristics such as thermal stability, ruggedness, and compressibility are determined by the different materials used to make ferrules. It is important to choose the right ferrule type and size to ensure proper column installation. The wrong ferrule type could cause damage to sensitive detectors such as ECDs, ELCDs, and MSDs. The wrong ferrule size or type can cause system leaks that result in decreased sensitivity and deterioration.

Ferrule Materials

Since metal ferrules would damage fused silica tubing, softer materials are used for capillary column ferrules. The two most common materials for capillary column ferrules are graphite and Vespel®. These materials can also be combined to form hybrid ferrules with the benefits of each material. Other ferrule materials, such as Teflon® and silicone, are commonly used with packed columns, but because of their limited thermal stability they are not typically used with capillary columns. Table I lists the maximum operating temperatures and the characteristics of common capillary ferrule materials.

Table I - Common Characteristics of Capillary Ferrules

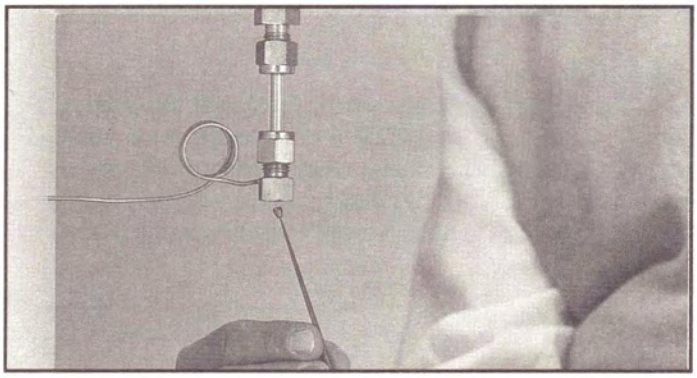
Material	*Max Temp.*	*Characteristics*
Graphite	450°C	Soft, easily conforms to all column sizes. Excellent for high temperature applications. Can flake or deposit particles in inlet & detector fittings. Easily deforms, resulting in limited reusability. Not recommended for vacuum interfaces.
Vespel®/Graphite	400°C	Hard, must be sized to exact column OD. Contracts when cooled causing leakage if not retightened after several thermal cycles. Excellent reusability.

Properties of Graphite Ferrules

Many chromatographers prefer graphite ferrules because they are soft and easily conform to any fitting dimension. Most graphite ferrules are made by tightly winding graphite ribbon around a pin and compressing it into a mold. The graphite ribbon increases ferrule pliability and allows it to deform easily. Increased pliability makes it possible to seal a 0.4mm OD (0.25mm ID) fused silica column with a 0.8mm ID ferrule. In addition, the ferrule can accomodate larger columns if the graphite bore is cored out. These features allow chromatographers to always have the right size ferrule on hand.

Graphite ferrules should be tightened using minimal force. Usually ¼-turn past finger-tight is sufficient to form a leak-tight seal. If a graphite ferrule is over-tightened, it will extrude out of the bottom of the nut, deform into the fitting cavity, and create ferrule fragments. These particles can be driven further into the inlet or make-up gas fitting, causing adsorption or peak tailing when a column is reinstalled. Graphite ferrules can also flake or abrade and emit particles that can clog small orifices. Because graphite is porous, graphite ferrules leak under vacuum. Therefore, graphite ferrules are not recommended for detectors operated under vacuum, such as MSDs.

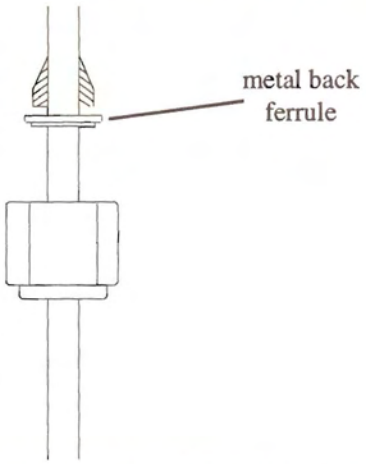
Graphite ferrules must be carefully removed, otherwise fragments and flakes remaining in the fitting can contaminate the GC system. Ferrules are easily dislodged by inserting a tapered needle file into the bore and moving it side-to-side. If the graphite ferrule does not come out in one piece, the inlet or detector fitting should be completely disassembled to ensure that no ferrule fragments remain.



Needle files easily remove graphite ferrules from injector and detector fittings or nuts. Gently insert the file into the ferrule bore and move it from side-to-side to dislodge the ferrule.

The life of a graphite ferrule is limited because they compress so easily. Some chromatographers obtain new life from a crushed ferrule by installing a reversed Swagelok®-type back ferrule between the fitting and the ferrule (Figure 1). The back ferrule raises the graphite ferrule higher in the fitting, allowing it to seal again.

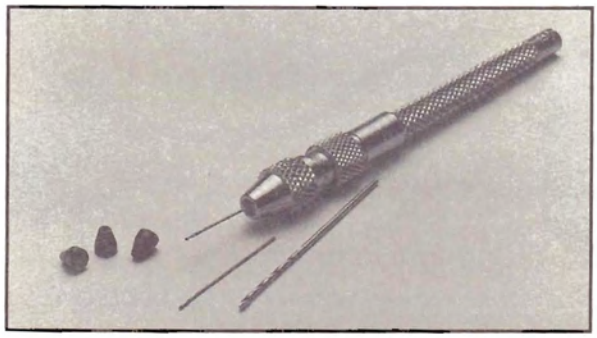
Figure 1 - Give a used graphite ferrule new life by installing a reversed metal back ferrule in the fitting.



Properties of Vespel® or Vespel®/graphite

Both 100% Vespel® and Vespel®/graphite ferrules are available. Vespel®-type ferrules are often preferred because they do not flake, deposit particles, or fall apart in a fitting. Most chromatographers choose the Vespel®/graphite ferrule combination. These ferrules are made by compressing a graphite/polyimide powder under high pressure in a heated mold. They retain their shape and can easily be removed intact. Vespel®/graphite has a higher thermal stability than Vespel® (400°C vs. 350°C) and the graphite impregnation makes the ferrule feel softer and seal with less torque. Vespel®/graphite ferrules are currently available in combinations ranging from 85% Vespel®/15% graphite to 60% Vespel®/40% graphite. The 60/40 Vespel®/graphite combinations are preferred by most chromatographers because they seal with the least amount of torque.

Unlike graphite, the inside diameter of Vespel®-type ferrules must be very close to the column OD in order to seal properly. If the ID of a Vespel®-type ferrule is too large for the column OD, it will not compress properly and allow a leak. Usually, the ferrule forms an oval shape, gripping the tubing but not sealing at the ends of the oval. If the ID of a Vespel®-type ferrule is too small to fit over the column, the bore must be enlarged with a small drill.



If the Vespel®/graphite ferrule's ID is too small to fit over the column, a pin vise drill can be used to enlarge the bore.

Vespel®/graphite ferrules will deform to the exact fitting dimension when heated. Usually this deformation process causes the ferrule to become loose and leak during the cool down cycle of a GC oven. Therefore, they must be subsequently retightened after several thermal cycles or carrier gas leakage will occur. No additional shrinkage or loosening occurs once the ferrule has conformed to the internal dimensions of the fitting cavity.

Vespel® ferrules can be removed from a fitting using a tapered needle file in the same manner as a graphite ferrule. Vespel® ferrules sometimes stick to the fitting and column after they have been in use for a prolonged period. Stuck ferrules can be removed by tapping the fitting with a solid object such as a wrench and gently pulling outward on the column. This problem is greatly minimized by using Vespel®/graphite combination ferrules.

What are common ferrule sizes?

Most column connections in the GC inlet and detector are made using 1/16" Swagelok®-type fittings. The ID or opening of the ferrule depends on the outside diameter of the column. Table II lists common fused silica capillary column IDs, ODs, and recommended ferrule sizes.

Table II - Common Ferrule Sizes for Fused Silica Capillary Columns

Column ID	*Column OD*	*Ferrule Opening*
0.18 to 0.25mm	0.35 to 0.40mm	0.4mm
0.32mm	0.45 to 0.48mm	0.5mm
0.53mm	0.69 to 0.72mm	0.8mm

The choice of ferrule material is often personal preference. If you are installing a capillary column for the first time, we suggest using a graphite ferrule. Graphite easily forms a leak-tight seal and conforms to any column OD. If you frequently install new columns, Vespel®/graphite is recommended to eliminate particle evolution and minimize maintenance downtime. However, when connecting columns to MSDs or Mass Spectrometer transfer lines, Vespel®/graphite is the only ferrule you should use to ensure a leak-free seal under vacuum. We recommend trying both ferrule types to choose a ferrule that best fits your needs. ■

[1]This article is a reprint from the *The Restek Advantage*, Vol. 4 No. 2, March 1993, p. 12-13.

Peak Performers

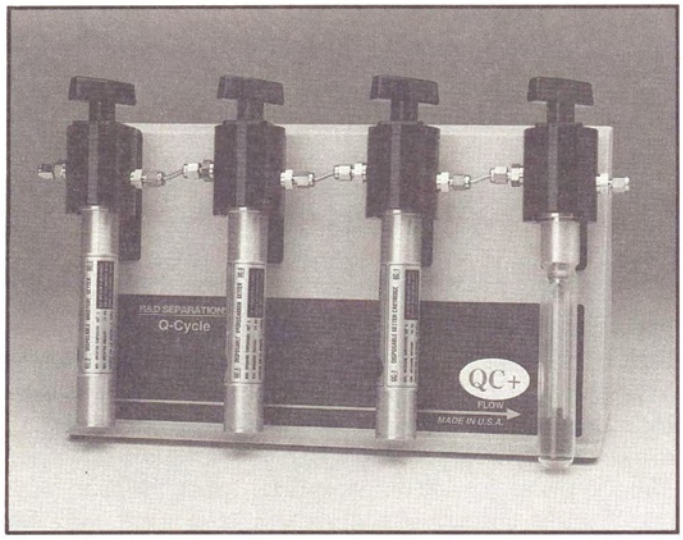
Simplify Gas Purification with the QC+ Purifier Panel

- Replace GC gas purifiers without flow interruption.
- Self aligning, trouble-free installation.
- Three and four panels units available.
- Impurities reduced to low part per billion levels.

GC carrier, make-up, and fuel gases require purification in order to provide stable baselines and longer column life. Traditional gas purifiers are installed at the gas source or in-line near the instrument, but require instrument downtime to be replaced. The new QC+ Purifier panels can be bench or wall mounted and allow a complete set of gas purifiers to be quickly changed without significant interruption in gas flow, eliminating instrument downtime.

Each QC+ filter mounting head is equipped with an internal valve that provides two flow paths. In the up position, it provides a closed gas path through the mounting head. As the valve is moved to the down position, it punctures the seal on the cartridge top and shifts the gas flow through the purifier cartridge. QC+ cartridges are self-aligning for trouble-free installation and are quickly replaced via a simple knurled retaining nut. As many as four cartridges can be replaced in less than two minutes without contamination of your GC gas system.

Restek offers two QC+ panels. The QC+ 3 cartridge panel features moisture, oxygen and indicating oxygen cartridges. The QC+ 4 cartridge panel is the ultimate in GC purification. It features the moisture, hydrocarbon, oxygen and indicating oxygen cartridges. The indicating oxygen cartridge can be regenerated to further increase the value of these units. Both panels will reduce gas impurities to low part per billion levels, regardless of source gas condition.



Product Listing

QC+ 3 head purifier panel
(with Moisture, O_2, & Indicating O_2 traps):
cat.# 21676

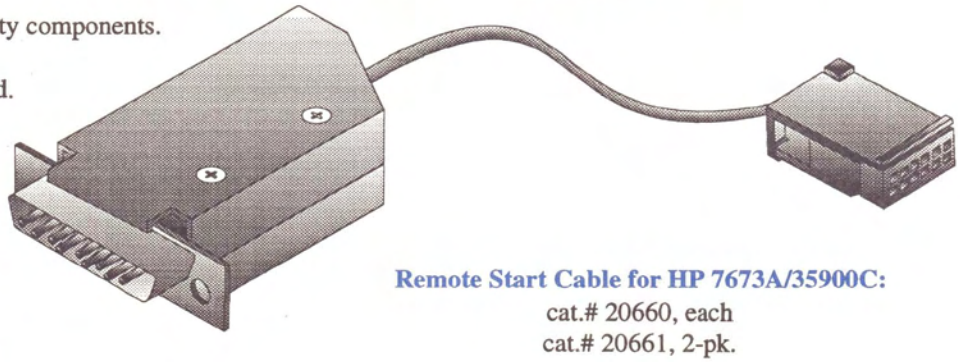
QC+ 4 head purifier panel
(with Hydrocarbon, Moisture, O_2, & Indicating O_2 traps):
cat.# 21677

QC+ Replacement Cartridges
Oxygen Cartridge: cat.# 21678
Moisture Cartridge: cat.# 21679
Hydrocarbon Cartridge: cat.# 21680
Indicating O_2 Cartridge: cat.# 21681

Remote Start Cable for HP 7673A/35900C

- Manufactured with only the highest quality components.
- Tested for 100% signal integrity.
- Instructions and wiring diagrams included.
- Priced less than original HP equipment.
- Instructions and wiring diagrams included.

Connect an HP 5890 GC to an HP 35900C Interface or an HP 7673 Autosampler to enable remote starts. Similar to HP part # 35900-60700.



Remote Start Cable for HP 7673A/35900C:
cat.# 20660, each
cat.# 20661, 2-pk.

MXT® Capillary Columns

The unbreakable alternative for process control and portable GCs

- Stationary phases available for most applications.
- High degree of inertness.
- Priced equivalent to fused silica.
- Extreme flexibility eliminates spontaneous breakage.
- Standard coil diameter approximately 5" OD, smaller custom coil sizes available.

What are MXT® columns?

MXT® columns are made by depositing a uniform, micron layer of flexible fused silica on the inner surface of thin walled stainless steel tubing. The surface is then deactivated by the same process used to treat our fused silica capillary columns.

MXT® columns are easy to use!

Available in both 0.28 and 0.53mm IDs (other IDs available upon request), MXT® columns can be installed in most instruments without modification. MXT columns are easy to cut using a small file or ceramic scoring wafer.

If the idea of an unbreakable capillary column sounds like the solution to your analytical problems, try an MXT® column. They are backed by Restek's 100% satisfaction guarantee. For a complete list of MXT® products, call your local distributor for a free copy of our *MXT® Columns & Accessories* catalog.

			0.28mm ID columns			0.53mm ID columns			
MXT®-1 Applications: Sim Dist, waxes, fuel oils, pharmaceutical, solvents	df (µm)	max. temp.	15-meter cat.#	30-meter cat.#	60-meter cat.#	15-meter cat.#	30-meter cat.#	60-meter cat.#	
	0.25	360°C	70121	70124	70127	70122	70125	70128	
	0.50	330°C	70136	70139	70142	70137	70140	70143	
	1.00	325°C	70151	70154	70157	70152	70155	70158	
	1.50	320°C	-------	-------	-------	70167	70170	70173	
	3.00	300°C	70181	70184	70187	70182	70185	70188	
	5.00	275°C	-------	-------	-------	70177	70179	70183	
	7.00	250°C	-------	-------	-------	70191	70192	70193	
MXT®-5 Applications: semi-volatiles, pesticides, PCBs, environmental samples, essential oils	df (µm)	max. temp.	15-meter cat.#	30-meter cat.#	60-meter cat.#	15-meter cat.#	30-meter cat.#	60-meter cat.#	
	0.25	360°C	70221	70224	70227	70222	70225	70228	
	0.50	330°C	70236	70239	70242	70237	70240	70243	
	1.0	325°C	70251	70254	70257	70252	70255	70258	
	1.5	320°C	-------	-------	-------	70267	70270	70273	
	3.0	300°C	70281	70284	70287	70282	70285	70288	
	5.0	275°C	-------	-------	-------	70277	70279	70283	
MXT®-Wax Applications: FAMEs, flavors, solvents, BTEX, essential oils, EPA Method 603	df (µm)	max. temp.	15-meter cat.#	30-meter cat.#	60-meter cat.#	15-meter cat.#	30-meter cat.#	60-meter cat.#	
	0.25	250°C	70621	70624	70627	70622	70625	70628	
	0.50	240°C	70636	70639	70642	70637	70640	70643	
	1.0	230°C	70651	70654	70657	70652	70655	70658	
	1.5	220°C	-------	-------	-------	70666	70669	70672	
	2.0	230°C	-------	-------	-------	70667	70670	-------	
MXT®-502.2 Applications: volatile organics	df (µm)	max. temp.	30-meter cat.#	60-meter cat.#	105-meter cat.#	30-meter cat.#	60-meter cat.#	105-meter cat.#	
	1.6	270°C	70919	70920	70921	-------	-------	-------	
	3.0	270°C	-------	-------	-------	70908	70909	70910	
MXT®-624 Applications: EPA Methods 502.2, 524, 624, 8240, and 8260	df (µm)	max. temp.	30-meter cat.#	60-meter cat.#	105-meter cat.#	30-meter cat.#	60-meter cat.#	75-meter cat.#	105-meter cat.#
	3.0	240°C	-------	-------	-------	70971	70973	70974	70975
Sim Dist MXT®-1	6m, 0.53mm ID, 0.15µm, cat.# 70101				cat.# 70101's are tested at 400°C, but may be run at higher temperatures and at a lower sensitivity with additional conditioning.				
Sim Dist MXT®-2887	10m, 0.53mm ID, 2.65µm, cat.# 70199				cat.# 70199's maximum temperature is 360°C.				

Note: All maximum operating temperatures are for 30m columns. Maximum temperatures may be slightly lower for longer lengths.

CHROMalytic TECHnology >>>

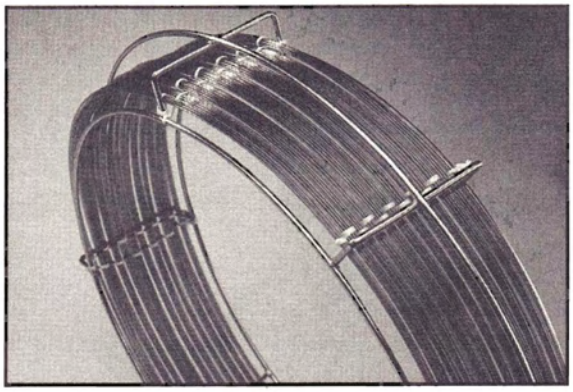
" some" **promos / Products / Offers** in the **ADVNews**
have been since been progressively superceded
/ UPDATED OR Since Discontinued
CHECK THE latest Restek ADVantage Newletter, Restek ESSENTIALS
. . . *Or* The Restek Catalog . . . *Or* other Resteb publications for updates
www.chromtech.net.au or NEW site 2015 > **www.chromalytic,net.au**

THE RESTEK ADVANTAGE

NEW! Rtx®-5 Amine Capillary Columns

Analyze Amines and Other Strongly Basic Compounds

- Eliminate the need to derivatize basic compounds.
- Minimal priming effects.
- For concentrations as low as 5ng on-column.



The reproducible analysis of basic compounds by capillary gas chromatography has always been difficult due to the presence of acidic silanol (SiOH) functional groups on the surface of fused silica tubing. Because common deactivation schemes do not completely remove or react with these silanols, the fused silica surface can remain acidic. When basic compounds, such as amines, are analyzed on an acidic surface, they are retained due to acid/base interactions. Chromatographically, this results in reduced response and severe peak tailing of the basic compounds.

The wizards at Restek have developed a new Rtx®-5 Amine column with unique deactivation technology to improve response and reduce tailing of basic compounds. Analyses that previously required derivitization or another analytical technique such as HPLC can now be performed on the Rtx®-5 Amine column. This column can also be used to analyze neutral compounds with the same efficiency as our standard Rtx®-5 columns.

We have developed a stringent quality assurance test mix containing several basic compounds such as pyridine, diethylenetriamine, diethanolamine, and 2,6-dimethylaniline. The on-column concentrations of these analytes range from 10-20ng. Specifications for the response and separation of these components ensure that each Rtx®-5 Amine column delivers consistent results.

New Deactivation Technology for Analyzing Basic Compounds

The analysis of basic compounds is most commonly accomplished with Carbowax® (PEG) columns doped with a basic salt. Certain compounds, such as alkylamines and diamines can be successfully analyzed on base deactivated Carbowax® columns. Unfortunately, other high pKa compounds such as

in this issue...

ethanolamines cannot. Also, the polar characteristics of the Carbowax® phase can often result in prolonged analysis time for many of these compounds.

Typical deactivation techniques used for less polar siloxane based stationary phases do not prevent the adsorption of amine-functional compounds. Figure 1a shows typical peak shapes for the analysis of ethanolamines on an Rtx®-5 capillary column. Note the reduced response and poor peak shape of the ethanolamines relative to an internal standard of equal concentration. Figure 1b shows the same compounds analyzed with the new Rtx®-5 Amine column. The peak height and shape are much improved compared to a standard Rtx®-5 column, indicating this new deactivation technology is effective for analyzing basic compounds.

The Rtx®-5 Amine column analyzes alkylamines, diamines, triamines, and nitrogen-containing heterocycles

Alkylamines, diamines, triamines, and nitrogen-containing heterocycles are common analytes found in lubricants, cosmetic products, and used as fuel additives. The Rtx®-5 Amine column delivers consistent analytical results for a wide variety of amine compounds found in many commercial products. Figure 2 shows an industrial sample which contains several different types of ethyleneamine and piperazine derivatives.

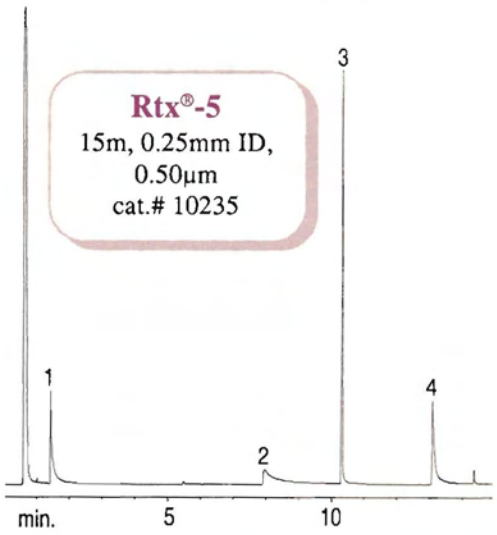
Tired of derivatizing your basic drug analytes?

The Rtx®-5 Amine column can save time and money by eliminating the need to derivatize basic pharmaceutical compounds. Figure 3 shows the analysis of seven basic drugs in their salt or free base form. Our studies have found that the dissociation of salt forms is most efficient in a hot injection port (315°C) with a slow injection rate (3-5sec./µl). Accurate qualitative and quantitative analyses of underivatized drugs can be achieved with the Rtx®-5 Amine column.

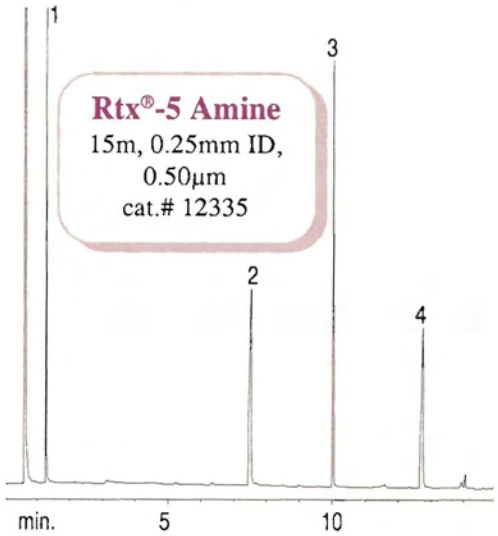
If your lab is analyzing amines or other strongly basic compounds, the new Rtx®-5 Amine columns give accurate and consistent results. These columns are available in 15 and 30 meter lengths; 0.25, 0.32, and 0.53mm IDs; and film thicknesses from 0.50 to 3.0µm. Each column is rigorously tested with a special mixture of amines to ensure column-to-column reproducibility.

Figure 1 - The new Rtx®-5 Amine column shows improved response and peak shape for ethanolamines over standard Rtx®-5 columns.

1a)



1b)



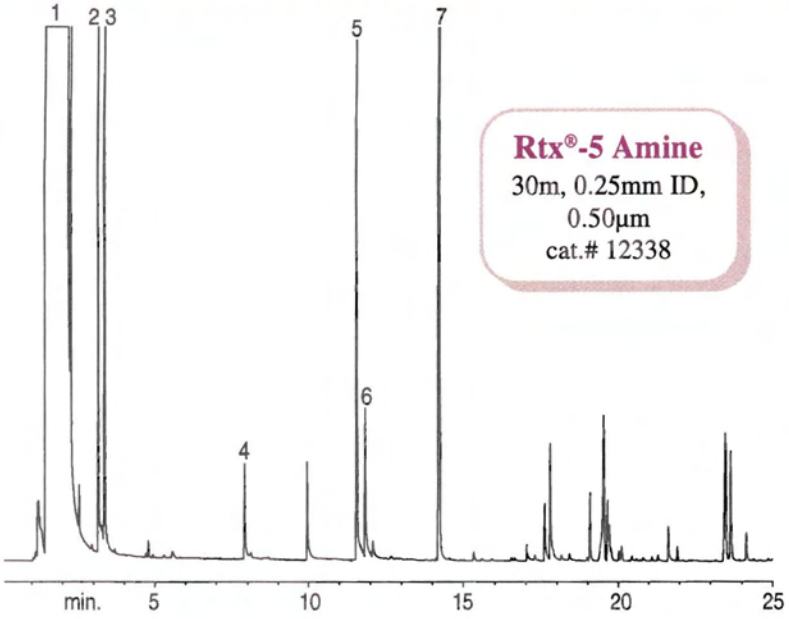
Peak List and Run Conditions for Figures 1a and 1b

1. monoethanolamine
2. diethanolamine
3. triethylene glycolmonomethylether (IS)
4. triethanolamine

1.0µl split injection of ethanolamine mix in methanol, on-column concentration=34ng

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
Split ratio:	58:1

Figure 2 - Obtain excellent peak symmetry and response of a wide range of industrial amines on the Rtx®-5 Amine column.



Rtx®-5 Amine
30m, 0.25mm ID,
0.50µm
cat.# 12338

ethyleneamines (and impurities) industrial sample:

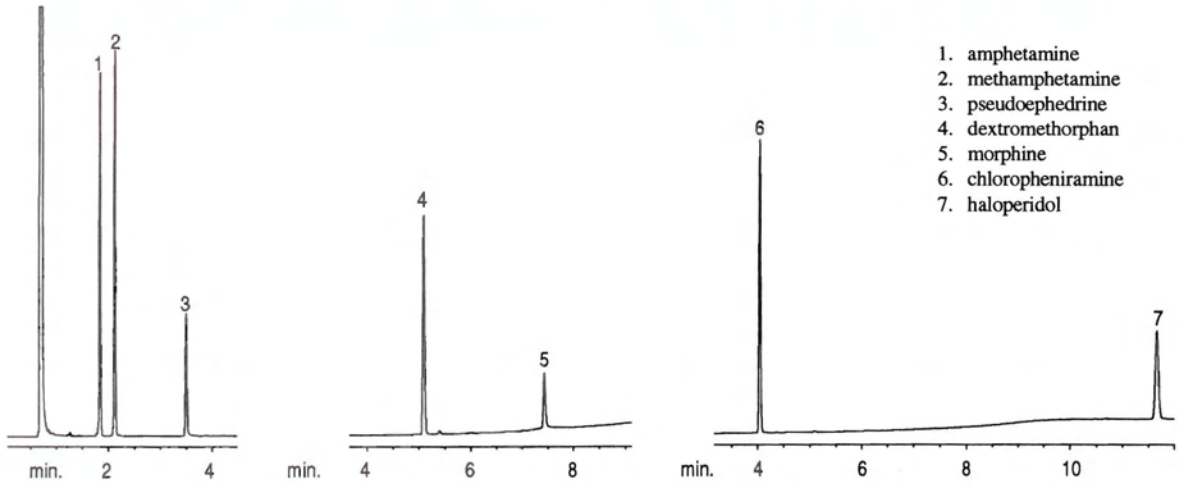
1. isopropanol
2. monoethanolamine
3. ethylenediamine
4. piperazine
5. diethylenetriamine
6. aminoethylethanolamine
7. aminoethylpiperazine

remaining impurities consist of ethyleneamine and piperazine derivatives

3.0µl split injection of Ethyleneamine Industrial Sample
concentration ~5-80ng on-column

Oven temp.:	40°C (hold 4 min.) to 315°C @ 10°C/min. (hold 5 min.)
Inj./det. temp.:	315°C
Carrier gas:	hydrogen
Linear velocity:	43cm/sec. set @ 40°C
FID sensitivity:	6.4 x 10^{-11}AFS
Split ratio:	20:1

Figure 3 - Eliminate the need for derivatization of basic drugs using the Rtx®-5 Amine column.



1. amphetamine
2. methamphetamine
3. pseudoephedrine
4. dextromethorphan
5. morphine
6. chloropheniramine
7. haloperidol

15m, 0.25mm ID, 1.0µm Rtx®-5 Amine column (cat.# 12350)
1.0µl split injection of basic drugs in methanol, on-column concentration=39ng

Oven temp.:	225°C to 315°C @ 10°C/min. (hold 10 min.)
Inj./det. temp.:	315°C
Carrier gas:	hydrogen
Linear velocity:	43cm/sec. set @ 225°C
FID sensitivity:	6.4 x 10^{-11}AFS
Split ratio:	58:1

New! Rtx®-5 Amine Columns

	df(µm)	15-meter	30-meter
0.25mm ID	0.50	12335	12338
	1.00	12350	12353
0.32mm ID	1.00	12351	12354
	1.50	12366	12369
0.53mm ID	1.00	12352	12355
	3.00	12382	12385

Analyzing Drinking Water Volatiles Listed in EPA Method 524.2, Rev. 4.0

In August 1992, EPA Method 524.2, Rev. 4.0 added 24 new compounds to the existing list of 60 volatile drinking water contaminants. The analytical procedures remained the same and include a purge and trap system to concentrate the volatile organics, a capillary gas chromatographic column to separate the components, and a mass spectrometer for the measurement and detection of the analytes.

Monitoring 84 compounds in a single analysis requires care in selecting the proper column and analysis conditions.[1] A 105 meter, 0.53mm ID, 3.0µm, Rtx®-502.2 column (cat.# 10910) was used for the separation. This long, thick film capillary column eliminates the need for sub-ambient cooling. The GC parameters were optimized using *ezGC™* method development software. The run conditions recommended by the software are 35°C for 8 minutes, then program to 220°C at 9°C/min. Using this temperature profile, the overall analysis time was reduced to 34 minutes. This fast analysis time is possible when using a mass spectrometer because it is not necessary to resolve all components. Even if coelutions occur, extracted ion profiles can be used to accurately identify and quantitate the individual compounds.

Figure 1 shows the analysis of the 84 component standard at a concentration of 100ppb in water. The standard was spiked into a 5ml purge vessel and purged for 11 minutes at a flow rate of 40ml/min. Once trapped, the sample was desorbed for 2 minutes at 250°C, with a desorb preheat temperature of 245°C.

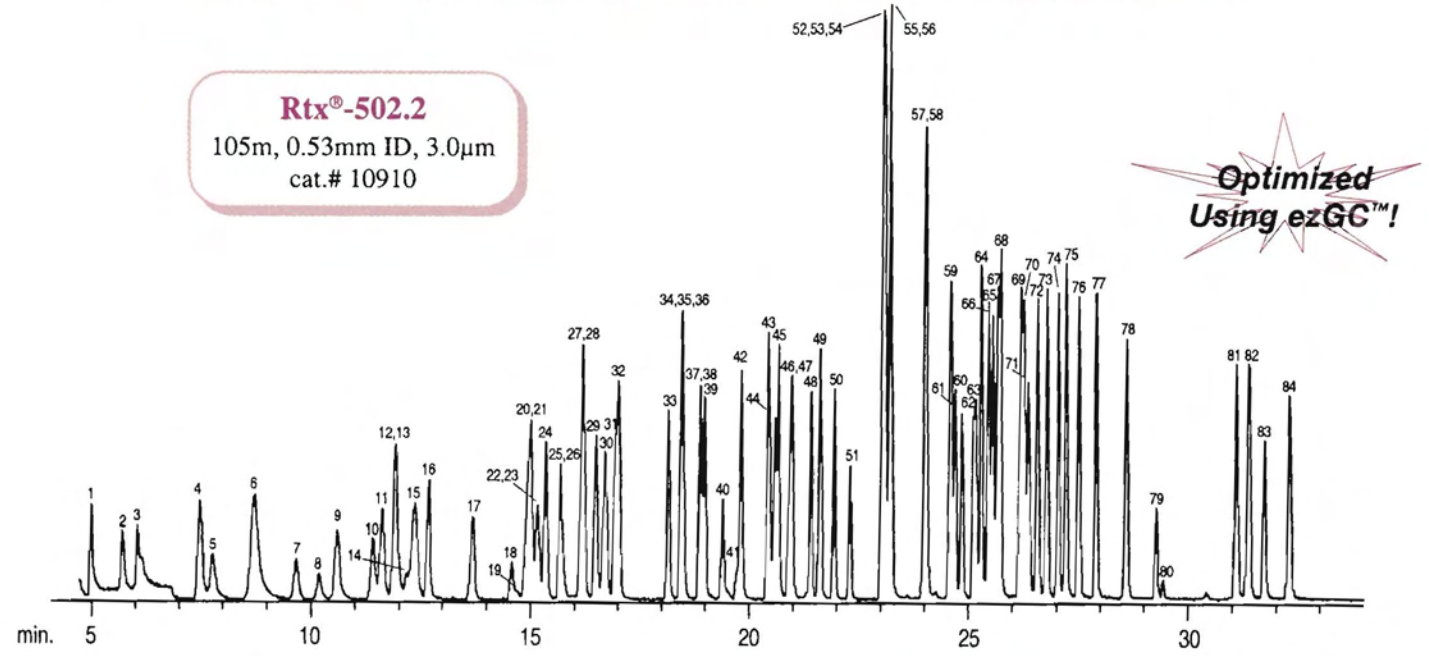
The desorb flow was set at 10ml/min. to transfer a narrow sample band to the capillary column. A low volume injector interface was used to connect the purge and trap transfer line to the GC. This interface prevents band broadening within the injection port. Before entering the vacuum system of the mass spectrometer, the column flow was split 10:1 using an open split interface. Alternatively, a jet separator can be used to reduce the carrier gas flow prior to entering the mass spectrometer. With the addition of 24 compounds, a scan range of 35 - 260 amu was required to obtain the primary quantitation ions of all 84 analytes. A solvent delay of 4.7 minutes was used to eliminate the CO_2 peak.

To assist in instrument calibration for EPA Method 524.2, Rev. 4.0, Restek now offers standards for the expanded target list. These standards are available from stock and include a convenient kit that covers the revised method. As with all Restek environmental mixtures, full data packs are available for each mixture to meet audit requirements.

EPA method 524.2, Rev. 4.0 has increased the total analyte list to 84 compounds. For the optimum analysis of these components, a 105 meter, Rtx®-502.2 capillary column is recommended. By using *ezGC™* method development software, run conditions were optimized to reduce analysis time to 34 minutes.

1. Munch, Jean W., Eichelberger, James W., *Journal of Chromatographic Science*, Vol. 30, Dec. 1992, pp 471-477.

Figure 1 -All 84 components listed in EPA Method 524.2, Rev. 4.0 can be analyzed in 34 minutes.



Standards for EPA Method 524.2, Rev. 4.0

For the 60 original compounds listed in EPA Method 524.2, Rev. 1.0, use Restek's 502.2 Calibration Mixes 1 through 6. For EPA Method 524.2, Rev. 4.0 compounds, add Restek's Calibration Mixes 7 & 8.

524 Calibration Mix #7

2000µg/ml each in 1ml P & T methanol, 1ml per ampule.

acetone	allyl chloride
acrylonitrile	2-butanone
methyl acrylate	tetrahydrofuran
methyl methacrylate	4-methyl-2-pentanone
ethyl methacrylate	2-hexanone
pentachloroethane	nitrobenzene

Cat.#	30202	each
	30202-500	each w/data pack
	30202-510	5-pack
	30202-520	5-pack w/data pack
	30302	10-pack w/data pack

524 Calibration Mix #8

2000µg/ml each in 1ml P & T methanol, 1ml per ampule.

diethyl ether	iodomethane
carbon disulfide	methyl tert-butyl ether
propionitrile	methacrylonitrile
1-chlorobutane	chloroacetonitrile
2-nitropropane	1,1-dichloropropanone
trans-1,4-dichloro-2-butene	hexachloroethane

Cat.#	30203	each
	30203-500	each w/data pack
	30203-510	5-pack
	30203-520	5-pack w/data pack
	30303	10-pack w/data pack

524 Internal Standard/Surrogate Mix

2000µg/ml each in 1ml P & T methanol, 1ml per ampule.

fluorobenzene	1,2-dichlorobenzene-d4
4-bromofluorobenzene	

Cat.#	30201	each
	30201-500	each w/data pack
	30201-510	5-pack
	30201-520	5-pack w/data pack
	30301	10-pack w/data pack

524 Revision 4.0 VOA Kit

Contains 1ml each of catalog #'s:

- 524 Internal Standard/Surrogate Mix (cat.# 30201)
- 524 Calibration Mix #7 (cat.# 30202)
- 524 Calibration Mix #8 (cat.# 30203)
- 502.2 Calibration Mix #1 (cat.# 30042)
- 502.2 Calibration Mix #2 (cat.# 30043)
- 502.2 Calibration Mix #3 (cat.# 30044)
- 502.2 Calibration Mix #4 (cat.# 30045)
- 502.2 Calibration Mix #5 (cat.# 30046)
- 502.2 Calibration Mix #6 (cat.# 30047)

Cat.#	30204	each kit
	30204-500	each kit w/data packs

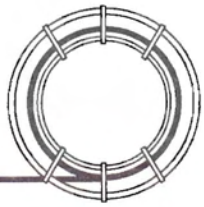
1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. diethyl ether
8. acetone
9. 1,1-dichloroethene
10. methyl iodide
11. allyl chloride
12. methylene chloride
13. carbon disulfide
14. acrylonitrile
15. MTBE
16. trans-1,2-dichloroethene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

Peak List and Run Conditions for Figure 1

105m, 0.53mm ID, 3.0µm Rtx®-502.2 (cat.# 10910)
HP 5971MSD, Tekmar LS-3000 concentrator with Tekmar Low Volume Interface
100ppb of 524 Revision 4.0 VOA Kit (cat.# 30204)

Oven temp.:	35°C (hold 8 min.) to 220°C @ 9°C/min. (hold 5 min.)
Det. temp.:	285°C
Carrier gas:	helium
Scan range:	35-260amu
Trap type:	VOCARB™ 3000
Purge time:	11 min. @ 40 ml/min.
Desorb preheat temp.:	245°C
Desorb temp.:	250°C
Desorb time:	2 min.
Desorb flow rate:	10 ml/min.

Hints for the Capillary Chromatographer



Using Electron Capture Detectors

Electron capture detectors (ECDs) are common GC detectors used to analyze compounds with electronegative functional groups such as nitroaromatics, halogens, and oxygenates. Many of these compounds are frequently encountered in analyses including pesticides, polychlorinated biphenyls, lead containing compounds, and clinical/forensic samples.

ECDs are very sensitive detectors providing accurate responses in the picogram and femtogram range. They are considered selective detectors because their response is not uniform and is very dependent on the individual component's affinity for electrons. Therefore, a polyhalogenated compound will have a much greater response than a monohalogenated compound.

Detector Operation

The ECD uses a radioactive source placed within the cell to emit beta particles. The carrier gas flows past the beta source and is ionized producing positive ions and a cloud of free electrons within the cell. These free electrons are captured by a positive electrode producing a stable background current which is amplified and used as a reference. When a sample component with an electron affinity enters the detector, it captures some of these electrons and decreases the current. This indicates the presence of the sample component within the detector. The ECD is the only common GC detector that uses a decrease in signal as a method of detection.

The ^{63}Ni pulsed detector is the most commonly used ECD. The ^{63}Ni foil contains a very small amount of radiation (usually less than 15 millicurie) and is sealed by the manufacturer inside the detector cell. Normally the GC manufacturer holds a general radiological license which covers their ECDs and a specific site license is not required by the NRC. To comply with NRC regulations, it is necessary to perform a radioactivity leak test every six months to verify the cell is not leaking radiation above the allowable limit. Arrangements can be made with the manufacturer or through an outside agency to obtain a leak test kit.*

Detector Gases

For proper ECD operation, the detector make-up gas must be ionizable. Helium and hydrogen, the two most common carrier gases used for capillary chromatography, do not readily ionize and, therefore, are not recommended as an ECD make-up gas. Since capillary column carrier gas flow rates are low (typically < 10cc/min), an ionizable make-up gas can be used to produce the desired electron cloud. The make-up gas is added at high flow rates to produce a stable signal (Table I). The two most common make-up gases used with ECDs are nitrogen and 5% methane in argon (Ar/CH_4). Nitrogen gives better sensitivity than Ar/CH_4. However, Ar/CH_4 yields a greater dynamic range than nitrogen. Both nitrogen and Ar/CH_4 are not recommended as carrier gases with capillary columns and should only be used as make-up gases.

Operating Hints

Because ECDs are extremely sensitive detectors, it is imperative the entire GC system be absolutely leak-free. Otherwise oxidation of the ^{63}Ni foil will occur and increased noise, baseline drift, and decreased lifetime of the detector will result. The best way to check the system for leaks is with a thermal conductivity leak detector (TCD) (cat.# 21605 or 20130). TCDs are recommended over liquid leak detectors for capillary chromatographic systems because they are very sensitive, easy to use, and there is no risk of contamination. Using a liquid leak detector like Snoop® can result in contamination of both the column and the detector if a leak is present. Even though the system is under positive pressure, liquid leak detectors can be drawn into the column at the leak point via the Venturi effect.

Moisture and oxygen traps are necessary for both the carrier gas and make-up gas or excessive detector noise will result. An indicating oxygen trap (cat.# 20624 or 20602) should be installed at the bulkhead inlet fitting to remove oxygen from

Table I
Operating Hints from Various Manufacturers

	Radiation Source	Detector Insertion Distance	Make-up Flow Rate
HP 5890	^{63}Ni	7.2cm (back of nut)	50-60ml/min.
Varian 3300/3400, 3600, 3700	^{63}Ni	13.2cm (back of nut) 11.5cm (back of nut)**	20-30ml/min.
Shimadzu 9A, 14A, 17A	^{63}Ni	9.0cm (tip of ferrule)	30-40ml/min.
PE Autosystem	^{63}Ni	6.5cm (back of nut)	30ml/min.

* Restek has been satisfied with the services of Detector Service Center (919)469-0259 and C.J. Bruyn & Co. (800)252-7896. Each wipe test costs approximately $20 to $25.
**metal column insertion distance

both the carrier gas and makeup gas lines. A molecular sieve trap (cat.# 20686) must be installed prior to the oxygen purifier to remove trace levels of water. Excessive noise and baseline instability will result if a molecular sieve trap is not used on an ECD, particularly if the GC does not come equipped with a small internal carrier/make-up gas line trap.* A hydrocarbon trap is not usually necessary since ECDs do not respond to hydrocarbon contamination. Also, be sure to use carrier gas and make-up gas regulators that are equipped with stainless steel diaphragms to avoid oxygen permeation.

Because ECDs are so sensitive, always precondition columns out of the detector. Install the column into the injector but not into the detector. Verify flow through the column and condition the column at the maximum test temperature for several hours, preferably overnight. Remember, the detector port must be capped to prevent air from oxidizing the ^{63}Ni foil. Before removing or installing a column into the ECD, always cool the detector below 100°C to prevent oxidation of the ^{63}Ni foil. *Never heat the ECD without a column installed or without capping the detector port!*

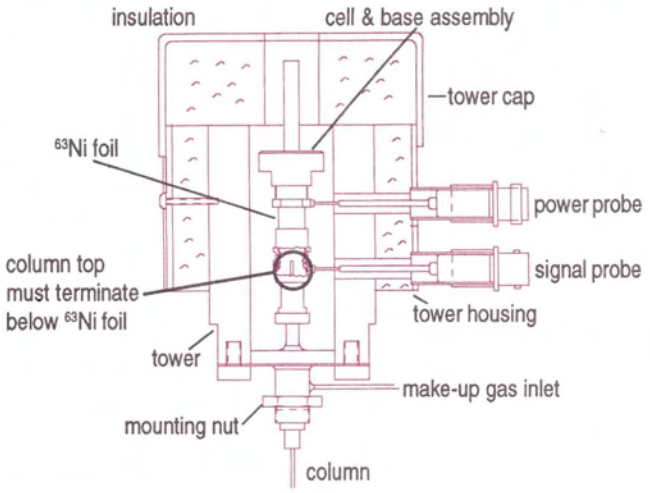
Detector Maintenance

Baseline instability or a high background signal is often an indication of a contaminated ECD cell.** With HP ECDs, a signal greater than 50 (500 Hz) indicates a contaminated system. A signal greater than 10 indicates contamination in Varian ECDs. Often, contaminants deposited on the radioactive foil can be removed by heating. For routine maintenance, thermal cleaning is recommended. To thermally clean an ECD, first cool the detector below 100°C, remove the column and cap off the detector port. Next, establish a make-up gas flow of 50 to 60 ml/min. and set the oven temperature to 250°C. For HP GCs, heat the ECD to 350°C for 3 to 12 hours. If the background signal continues to be high (>60), the detector should be returned to HP for cleaning. Varian recommends heating their ECD to 400°C for 6-12 hours. Monitor the output signal. It should initially increase in magnitude, then decrease. When the signal has reached a stable plateau, the foil has been cleaned as much as possible. Varian also suggests thermally cleaning their ECDs using hydrogen as a purging agent.†

Troubleshooting

Changes in sensitivity are sometimes related to the detector. A reduced response is often the result of an incorrect installation distance. It is critical that the column end terminates within the electron cloud located inside the cell. If the column end is installed too far into the detector and terminates above the cloud, or if the column is not installed far enough and terminates below the electron cloud, reduced response of sample components will be observed (Figure 1). Always install columns to the manufacturer's recommended insertion distance. Common detector insertion distances are shown in Table I.

Figure 1 - Adhere to manufacturer's insertion distances or optimize distance based on response.



Make-up gas flow also has a great impact on sensitivity. If the make-up gas flow is set improperly reduced sensitivity can result. Regularly check make-up gas flow and adjust if necessary. Set make-up gas flows according to the manufacturer's instructions.

Another common problem associated with electron capture detectors is caused by frequent heating and cooling of the detector when making changes to the system. The base screws and/or Vespel®/graphite ferrules can loosen with temperature changes. Always make sure all connections to the ECD are leak-free to prevent oxygen influx.

Once electron capture detectors are set up properly configured, they require little optimization. Remember, the ECD is a concentration dependent detector. Therefore, carrier gas flow rates must be kept constant and leaks eliminated. Since ECDs are very sensitive, they are easily affected by contamination. Molecular sieve and oxygen traps must be placed on all gas lines and changed on a regular basis. Every effort should be taken to prevent foil contamination which can lead to reduced sensitivity.

References:

1. *Varian 3300/3400 Gas Chromatograph Operator's Manual*, Vol. 2, Varian Associates, Inc. 1990.
2. *HP 5890 Series II Reference Manual*, Edition 2, Hewlett-Packard, October 1989.
3. *Gas Chromatograph GC-14A Instruction Manual*, Shimadzu Corporation.
4. Buffington, Rosemary and Wilson, Michael K., *Detectors for Gas Chromatography - A Practical Primer*, Hewlett-Packard Co., Avondale, PA, 1991.
5. Hill, Herbert and McMinn, Dennis, ed., Detectors for Capillary Chromatography, John Wiley & Sons, New York, 1992.
6. *Perkin Elmer Auto System GC Operator's Manual*, Perkin Elmer Corp. June 1991.

*Often excessive ECD noise or baseline instability can be traced to a contaminated internal trap. Routine replacement is highly recommended.
**Negative peaks in the baseline indicate an O_2 leak present in the system. A positive baseline rise is often indicative of column or detector contamination.
†For more information on hydrogen cleaning, refer to the electron capture detector section of the Varian manual.

NEW! Polar Deactivated Guard Columns

Provide Optimum Wettability for Polar Compounds

Chemists who are using polar analytical columns can now increase column lifetime and protect their expensive analytical column from harmful chemical damage. The life expectancy of a capillary column is greatly increased by using a 5-meter, deactivated, uncoated fused silica guard column. This prevents non-volatile contamination of the analytical column. Since the guard column is uncoated, sample components are allowed to enter the analytical column freely, while non-volatile contaminants are deposited in the guard column. Once contamination degrades performance, short lengths of the guard column can be removed. When the guard column is totally contaminated, replace it with a new one.

Polar Deactivated Guard Columns		
	5 meter length	10 meter length
ID	cat.#	cat.#
0.25mm	10065	10068
0.32mm	10066	10069
0.53mm	10067	10070

SFC/CE Narrowbore Tubing

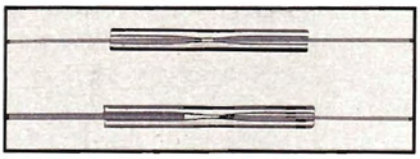
Restek now offers both untreated and deactivated narrow bore tubing for use in CE or as GC transfer lines, or as SFC restrictors. The IP deactivation process provides a phenyl methyl deactivated surface that gives optimum wettability and inertness for both non-polar and polar compounds. Deactivated tubing is available in continuous lengths up to 20 meters.

SFC/CZE Narrowbore Tubing			
ID	nominal OD	untreated	IP deactivated
		cat.#	cat.#
0.025mm	.36 mm	10091	10097
0.05mm	.36 mm	10092	10098
0.075mm	.36 mm	10093	10099
0.10mm	.36 mm	10094	10100
0.15mm	.36 mm	10095	10101
0.18mm	.36 mm	10096	10102

NEW! Deactivated, Universal Press-Tight® Connectors

- High temperature silanization for excellent inertness.
- Ideal for trace analysis of active compounds.
- Works with tubing ODs from 0.30 to 0.75mm (0.18 to 0.53mm ID)
- Available in economical 25 and 100 packs.

Restek's Universal Press-Tight® Connectors are made from rugged, highly inert fused silica and work with most common tubing diameters. However, if your application requires an extremely inert surface, we now offer connectors that have been treated with the same high temperature deactivation process used for our inlet sleeves. Ideal for the analysis of pesticides, semi-volatile pollutants, and clinical/forensic samples.



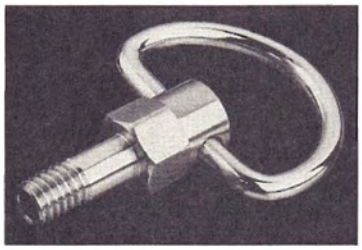
Deactivated Press-Tight® Connectors
cat.# 20429, 5-pk.
cat.# 20430, 25-pk.
cat.# 20431, 100-pk.

We're Nuts About HP!

Restek offers several uniquely designed fittings to fit HP GCs

Finger Tight Nut for HP 5890 GCs

- Rapidly tighten columns without wrenches.
- Avoid stripped threads from over-tightening.
- Two versions available, one for use with HP short ferrules and another for standard graphite ferrules.
- Both versions can be used with 0.25, 0.32, or 0.53mm ID columns.
- Wrench pad for use with Vespel®/graphite ferrules.
- 316 stainless steel body.
- Similar to HP part# 5020-8293 & 5020-8292 except Restek's can be used with Vespel® ferrules.

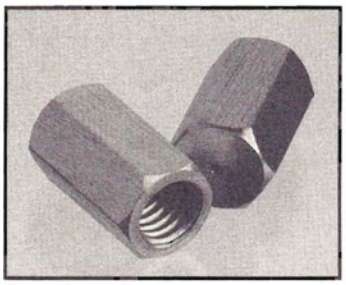


Finger Tight Nut*:
Standard Swagelok®-type ferrules:
cat.# 21312, each

HP-type "short" ferrules:
cat.# 21311, each

**purchase ferrules separately*

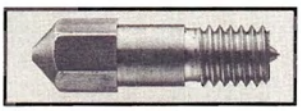
MSD Source Nut



The MSD source nut bore has been changed from 0.8mm to 1.2mm to permit easy removal of stuck ferrules with a standard tapered needle file (cat.# 20106). The nuts still match the specifications of the manufacturer's original part and are made of brass to prevent thread stripping on the transfer line. Similar to HP part # 05988-20066.

MSD Source Nut: cat.# 20643, 2-pk.

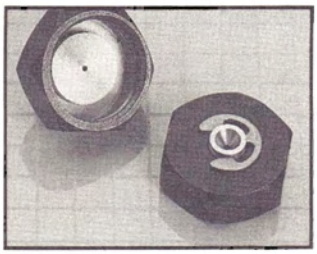
Detector Plug Nut for HP GCs



Need to cap off or thermally clean a dirty detector? Need to check detector or make-up gas flow rates? Want to prevent H_2 from accidentally diffusing into the oven from an unused detector base? Use Restek's brass Plug Nut for HP GCs. Similar to HP part # 5020-8294.

Detector Plug Nut: cat.# 21883, 2-pk.

Septum Nut for HP 5890 GCs (Autosampler Injections)



Ensure a leak-free injection port by using Restek's septum nut. This new high quality stainless steel nut is similar to HP part # 18740-60835. The thread design and needle guide allow easy penetration and prevent premature septum coring.

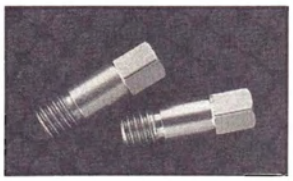
Septum Nut: cat.# 20631, each

Needle Guide Septum Nut for HP 5890 GCs (Manual Injections)

Increase septa lifetime and decrease maintenance requirements with Restek's septum nut for 26 gauge needle. This new septum nut directs the needle through the same hole, minimizing coring and leakage. For additional details on how our Needle Guide Septum Nut can extend septa lifetime, request Restek's technical tip, "Extending Septa Life", from *The Restek Advantage*, Vol. 4 No. 6. This nut is similar to HP part# 18740-60835 except with a 26 gauge hole.

Needle Guide Septum Nut: cat.# 21309, each

Stainless Steel Capillary Nuts for HP GCs



Restek now offers two stainless steel nuts for HP 5890 GCs. One version incorporates a deeper recess that allows the use of longer-based, Swagelok®-type graphite or Vespel®/graphite capillary ferrules. The other version has a shallow recess for use with shorter HP-type ferrules. This version is similar to HP part# 05921-21170.

Recessed Capillary Nut: cat.# 20883, 2-pk.
Standard Capillary Nut: cat.# 21884, 2-pk.

For more information on Restek's Inlet Supplies for HP GCs, please call your local distributor.

0.32mm ID Rt-Alumina™ PLOT Columns

For fast and efficient analysis of light hydrocarbons

Rt-Alumina™ PLOT columns have proven to be fast and reliable for the analysis of light hydrocarbons. Now, the Restek wizards have developed a 0.32mm ID Rt-Alumina™ PLOT column which improves efficiency and reduces analysis time for many applications.

The 0.32mm ID and 0.53mm ID Rt-Alumina™ PLOT columns exhibit the same unique selectivity, but the 0.32mm ID offers faster analysis times. Figure 1 shows the comparison of a 50 meter, 0.53mm ID and a 60 meter, 0.32mm ID Rt-Alumina™ PLOT column. Both columns achieve baseline separation of all 17 saturated and unsaturated hydrocarbons. The thinner alumina layer on the 0.32mm ID column results in a significant savings in analysis time. Even though the 0.32mm ID column is 10 meters longer, the analysis time is only 10 minutes, compared to 16 minutes with the 0.53mm ID column.

In addition to shorter analysis time, the 0.32mm ID offers improved resolution due to the higher plate count. The 0.32mm ID columns exhibit over 1600 plates/meter, compared to ~1000 plates/meter for the 0.53mm ID columns. The big advantage of the 0.53mm ID column is capacity. Since the 0.53mm ID column has a thicker alumina layer, its capacity is higher than the 0.32mm ID column. For trace level analysis, capacity may not be an important consideration. However, for purity determinations, the increased capacity of the 0.53mm ID columns can prevent column overload.

Effects of Water on the Rt-Alumina™ PLOT Column

The selectivity of the Rt-Alumina™ PLOT column can be affected by water contamination. This will cause changes in elution and retention of some components. By conditioning the column for 8 hours at 200°C with carrier gas, water can be removed and the proper selectivity restored.

Column Reproducibility is Guaranteed

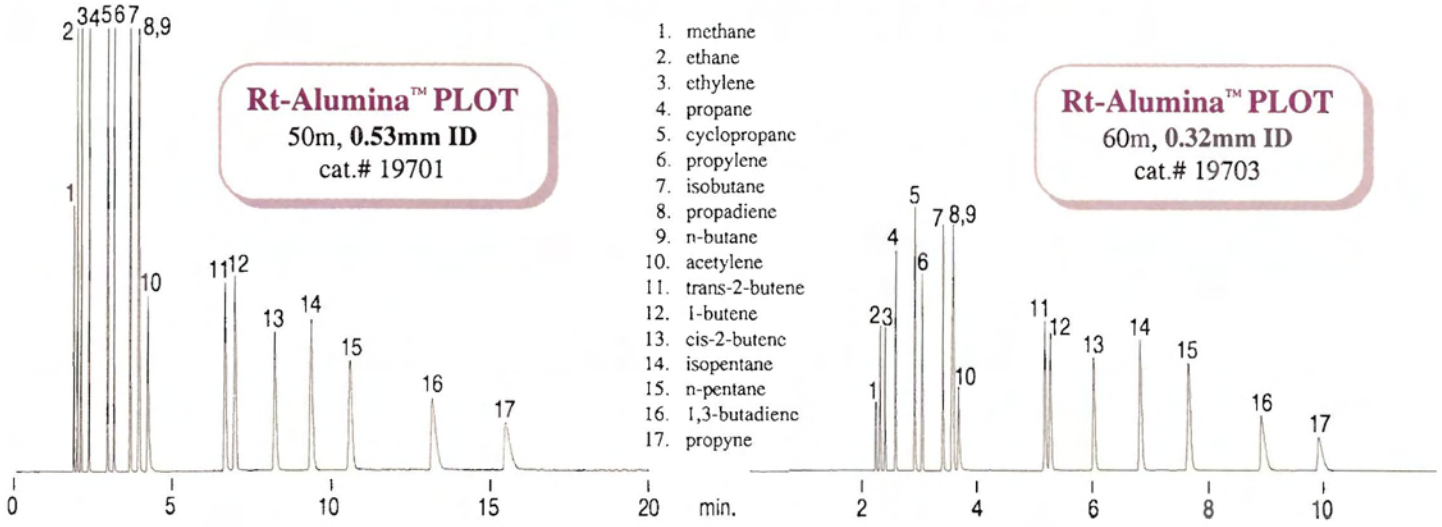
The chromatograms shown in Figure 1 are examples of the test mix used to evaluate every Rt-Alumina™ PLOT column. Pentane is used to calculate k (capacity factor) which verifies proper film thickness. A ratio of isobutane to acetylene retention is used to monitor the deactivation of the Alumina Oxide layer. The number of plates per meter is also calculated to determine column efficiency. Finally, the retention indices for acetylene and 1-butene are used to verify the selectivity of the Al_2O_3 phase. Every Rt-Alumina™ PLOT column is guaranteed to meet stringent QA specifications.

Restek's Rt-Alumina™ PLOT columns offer a choice for fast and reproducible analysis of hydrocarbon streams or purity analysis. With the introduction of 0.32mm ID Rt-Alumina™ PLOT columns, analysts have the option of enhanced efficiency and faster analysis times for light hydrocarbon separations.

Rt-Alumina™ PLOT Columns

60m, 0.32mm ID	Cat.# 19703
30m, 0.32mm ID	Cat.# 19702
50m, 0.53mm ID	Cat.# 19701
30m, 0.53mm ID	Cat.# 19700

Figure 1 - The 0.32mm ID Rt-Alumina™ PLOT column exhibits a faster analysis time than the 0.53mm ID column for a mixture of C1 to C5 hydrocarbons.



50µl split injection
Oven temp.: 80°C isothermal
Inj./det. temp.: 200°C
Carrier gas: helium
Linear velocity: 42cm/sec. set @ 80°C (5.6ml/min.)
Split flow: 40ml/min.
FID sensitivity: 1.28 x 10^{-10}AFS

30µl split injection
Oven temp.: 80°C isothermal
Inj./det. temp.: 200°C
Carrier gas: helium
Linear velocity: 46cm/sec. set @ 80°C (2.4ml/min.)
Split flow: 60ml/min.
FID sensitivity: 1.28 x 10^{-10}AFS

Optimizing the Analysis of Chlorophenoxy Herbicides

Chlorine substituted phenoxyacetic acids, such as 2,4-D, MCPA, and 2,4,5-T, were introduced as selective weed killers in the 1940's. Due to their growth-regulating and herbicidal activities against broadleaf weeds, they have been commonly used for weed control on cereal crops, grasslands, and lawns. 2,4-D and 2,4,5-T were also the primary defoliant agents used in Agent Orange in Vietnam. Today, chlorophenoxy herbicides are still used as commercially available lawn weed killers.[1]

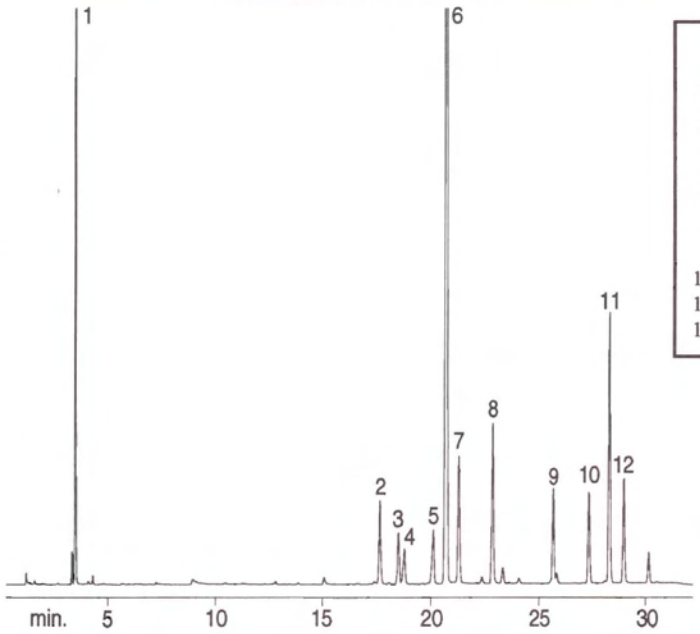
Chlorophenoxy herbicides are applied as either esters or salts which are easily metabolized by plants. The esters are oil soluble, but can also be applied as emulsions in water. The salts are typically highly soluble in water and are used as aqueous concentrates. Because the chlorophenoxy herbicides are spread on top of the soil or grass and then leach into the ground, there is great potential for ground water contamination. Chlorophenoxy herbicides readily degrade in the environment and, for many years, were not considered an environmental or public concern. However, potential hazards to public health and environmental quality led to the development of methods for the analysis of these herbicides. US EPA Methods 615 (municipal/industrial waste water), and 8150 (solid waste) were developed to monitor chlorophenoxy herbicides in environmental samples.[2,3]

Difficulties exist in the analysis of chlorophenoxy herbicides by gas chromatography. In their free acid form, these herbicides have limited volatility and are prone to irreversible adsorption. Therefore, they are most frequently analyzed as methyl esters. Since these herbicides can be applied as several different types of esters or as a salt, they must first be converted to the free acid form, then derivatized into methyl esters for analysis by gas chromatography. Methylation increases herbicide volatility and overcomes matrix interferences of herbicides extracted from soil. Despite this derivatization step, problems such as poor resolution, matrix interference, and peak misidentification are still experienced by analysts.

Using *Pro ezGC*™, computer modeling software, the analysis of chlorophenoxy herbicides was optimized with 30m, 0.53mm ID, 0.5µm Rtx®-35 (cat.# 10440) and Rtx®-5 (cat.# 10240) columns. Figures 1 and 2 show chromatograms of 10 derivatized chlorophenoxy herbicides listed in EPA Methods 615 and 8150B. All compounds are baseline resolved on the Rtx®-35 and the analysis is complete in 30 minutes. The Rtx®-35/Rtx®-5 dual column confirmational system produces a different elution pattern on each phase, offering a fast, positive peak identification.

Another important consideration when analyzing environmental samples for chlorophenoxy herbicide contamination is accurate instrument calibration. The key to accurate calibration is high quality chemical standards. Restek offers chlorophenoxy herbicide calibration, internal, and surrogate standards that can be used for EPA Methods 515.1, 615 and 8150. Each mixture is available in either the free acid form or the methyl ester form. The free acid mixtures can be used to verify analyte recovery or derivatization procedures. The derivatized mixtures can be used for instrument calibration. Compounds are separated into mixtures based on their

Figure 1 - The Rtx®-35 gives baseline resolution for all 12 chlorophenoxy herbicides.



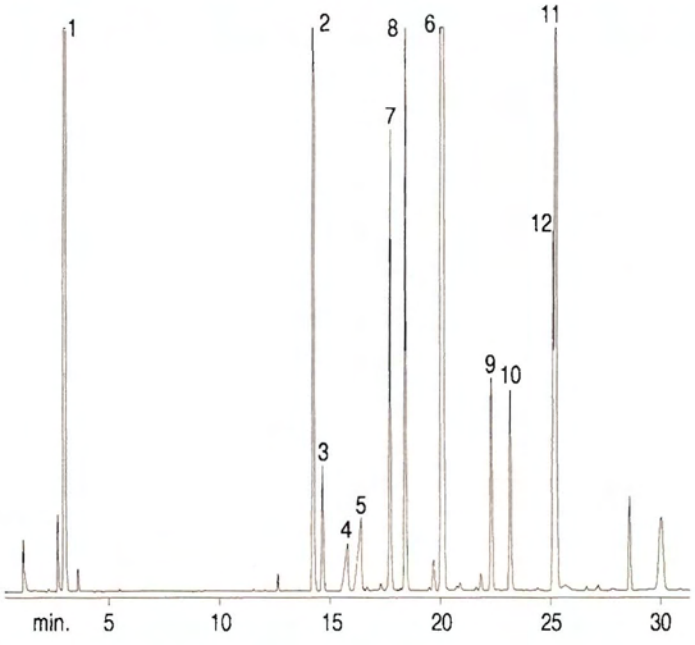
30m, 0.53mm ID, 0.50µm **Rtx®-35** (cat.# 10440)

0.5µl direct injection of Chlorophenoxy Herbicides, on-column concentration=10-10,000µg/ml

Oven temp.:	60°C to 150°C @ 8°C/min. (hold 5 min.), then to 210°C @4°C/min.
Inj./det. temp.:	250°C/275°C
Carrier gas:	helium
Linear velocity:	35cm/sec. set @ 60°C
ECD sensitivity:	60kHz FS

Optimized Using Pro ezGC™

Figure 2 - Use the Rtx®-5 for confirmation of peak identification.



30m, 0.53mm ID, 0.50µm **Rtx®-5** (cat.# 10240)

0.5µl direct injection of Chlorophenoxy Herbicides,
on-column concentration=10-10,000µg/ml

Oven temp.:	60°C to 150°C @ 8°C/min. (hold 5 min.), then to 210°C @ 4°C/min.
Inj./det. temp.:	250°C/275°C
Carrier gas:	helium
Linear velocity:	35cm/sec. set @ 60°C
ECD sensitivity:	160kHz FS

Please see Figure 1 for peak list.

Optimized Using Pro ezGC™!

response to allow labs to custom tailor their working calibration standards. As with all Restek environmental standards, these herbicides are thoroughly tested for purity and quantitative accuracy. All raw materials are tested by several analytical techniques including DSC (Differential Scanning Calorimetry), GC-FID, and GC/MS. The final product mixtures are tested in replicate using GC-FID.

The continued use of herbicides increases concerns of contamination. Laboratories must be able to determine the presence or absence of herbicide contamination in environmental samples. Capillary columns such as the Rtx®-35 and Rtx®-5 improve the quality of chlorophenoxy herbicides analysis by offering high resolution, inertness, and reproducible results. In addition to these columns, chemical standards are available for EPA methods 515.1, 615 and 8150B. Together, these products enhance the analysis of chlorophenoxy herbicides.

References

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