

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

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: 8x10⁻¹¹ AFS

Split vent: 40:1

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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.

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

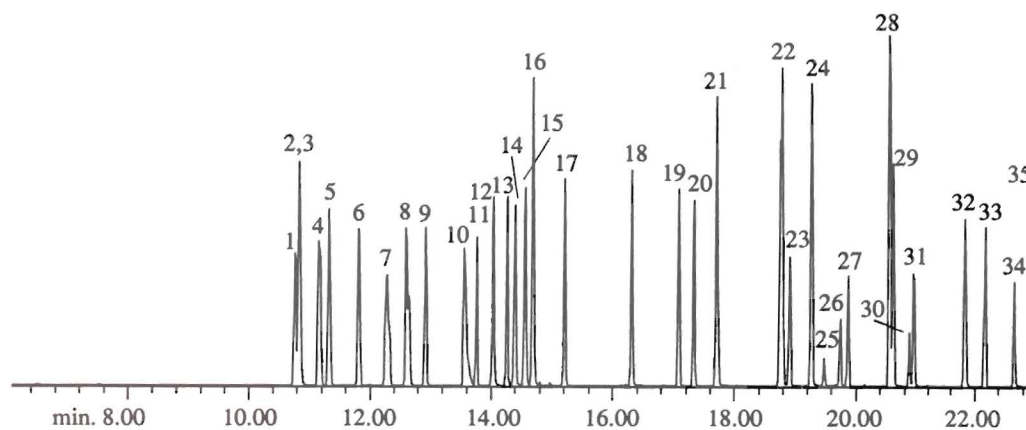
200ng without overload. This 0.50µm XTI®-5 column has a 330°C maximum operating temperature, which reduces the analysis time to 38 minutes.

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, 0.50µm 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.	Carrier gas:	helium	Splitless hold time:	0.5 min.
Inj. temp.:	280°C	Linear velocity:	80cm/sec. set @ 40°C	Scan mode:	TIC
Detector:	MSD	Purge flow:	25mls/min.	Scan range:	35-500 AMU
Det. temp.:	300°C				

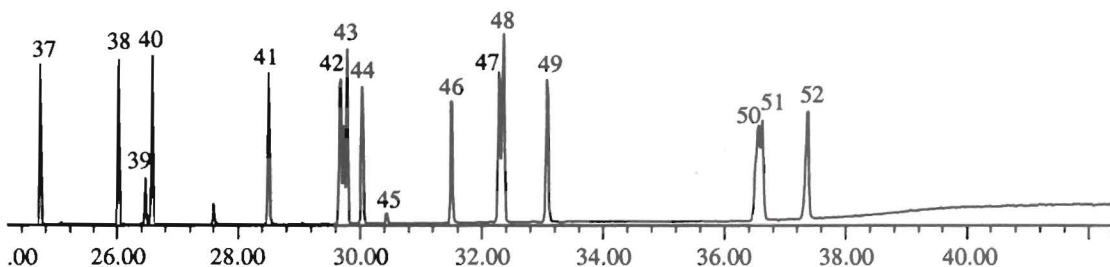
Product Listing

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.

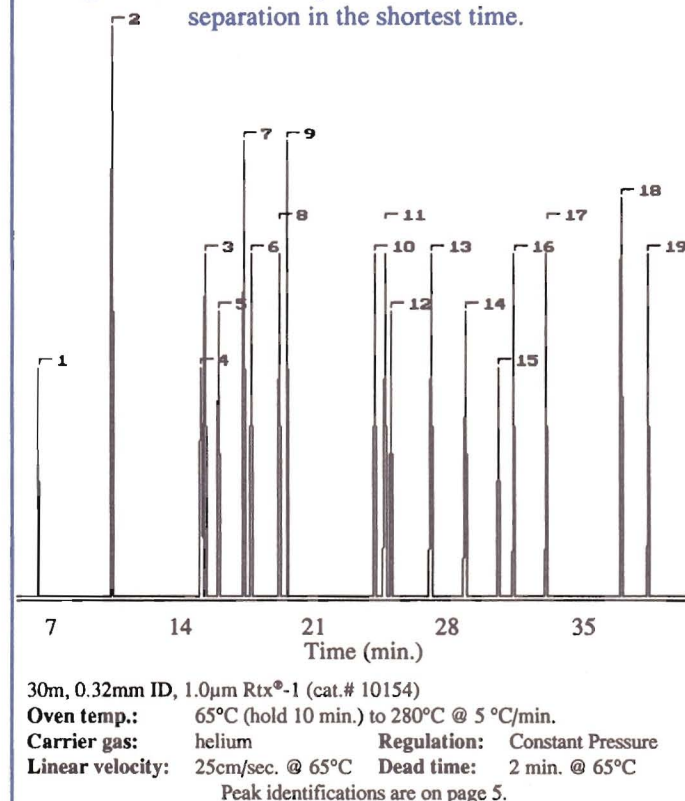
Figure 1 - Easily enter information into ezGC™ from menu driven screens.

The screenshot shows the 'Pro ezGC 1.07b' software interface. The 'Column' screen displays: Name: Rtx-1, Comment: Food Volatiles #10120, Length: 30.00 meters, Diameter: 0.320 mm, Film: 1.000 microns. The 'Operating Conditions' screen displays: Carrier Gas: Helium, Control: Constant Pressure, Gauge Pressure: 7.31 PSI, Flow Rate: 1.311 mL/min, Dead Time: 2.000 min, Linear Velocity: 25.00 cm/sec, Test Temp: 65.00 °C, Atm. Pressure: 14.69 PSI, Outlet Pressure: 14.69 PSI. The interface includes buttons for 'OK', 'Cancel', 'Help', and 'View Summary'.

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.



Determine how changes in column and carrier gas will improve separations

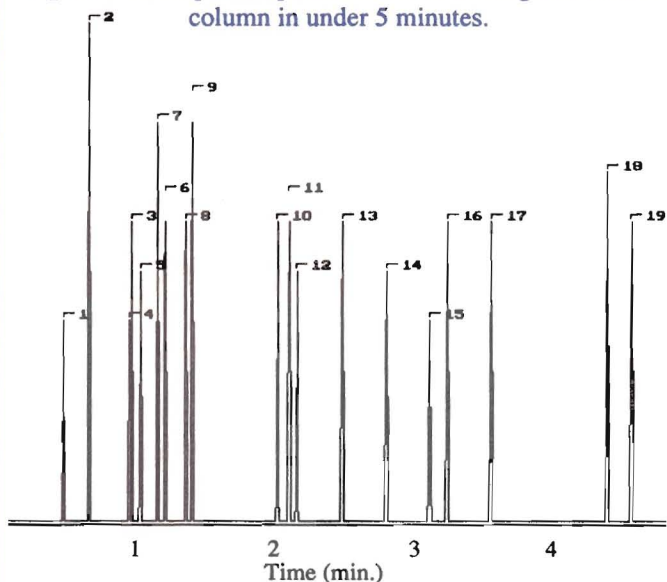
GC method development can be very costly when considering the expense of evaluating different stationary phases, column dimensions, and oven temperatures. Many chromatographers empirically optimize their chromatographic conditions for a particular column and then accept these as the "best separation" 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, H₂, N₂, 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.

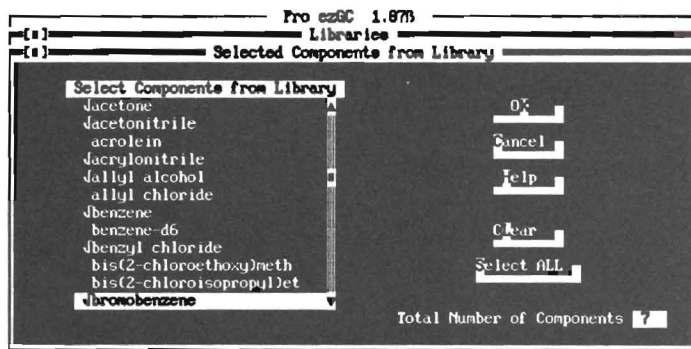
1 ethyl butyrate	8 p-cymene	15 eugenol
2 trans-2-hexenol	9 D-limonene	16 vanillin
4 benzaldehyde	10 methone	17 ethyl vanillin
3 α-pinene	11 menthofuran	18 ethyl laurate
5 camphene	12 menthol	19 amyl cinnamic aldehyde
7 β-pinene	13 carvone	
6 octanal	14 cinnamic alcohol	

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

AI 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

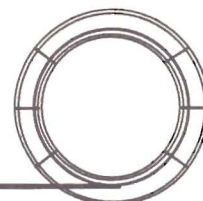
Environmental - Volatiles: A complete collection of the retention indices for the volatile organic compounds specified in EPA 500, 600, and 8000 series methods. A total of 137 components are included from the target analyte lists, recommended surrogates and internal standards for EPA methods 502, 524, 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 C₁ and C₁₂ 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



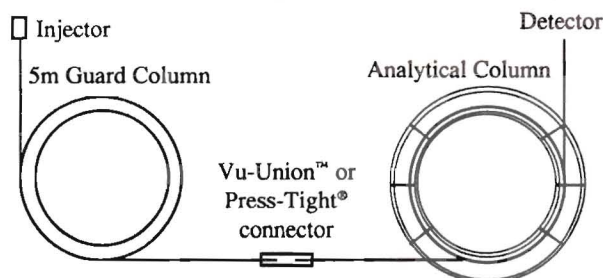
The Benefits of Guard Columns for Capillary Gas Chromatography

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

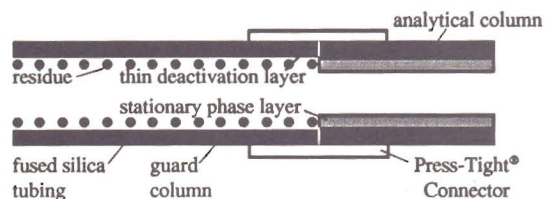


What advantages do guard columns offer?

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.

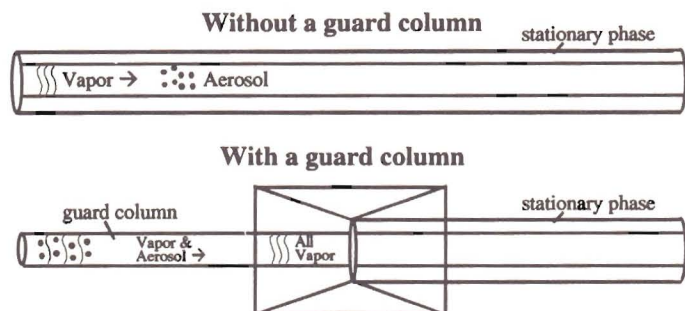
Decrease maintenance requirements

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.



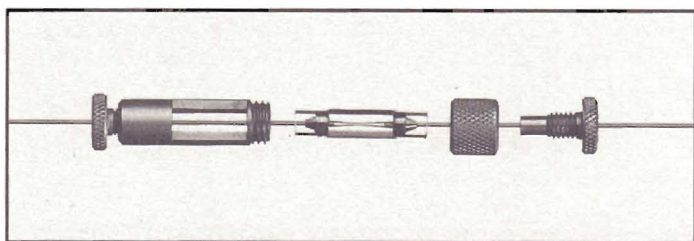
**Table I - Column Efficiency Data
(1µl split injection of 2,6-dimethylphenol)**

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.

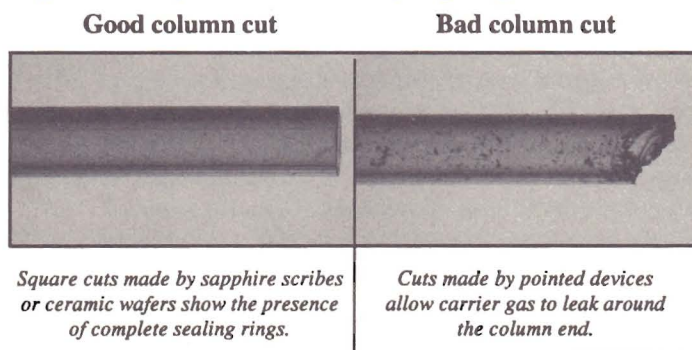


The metal housing holds the tapered glass insert securely. Two thumb screws are positioned at the end of each union to tightly compress the ferrule in the insert. The primary, low dead volume seal occurs as the column ends are compressed in the press-tight taper region.

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.



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?

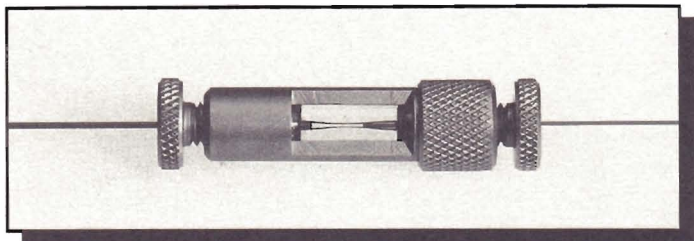
A guard column should be long enough to keep non-volatile residue from entering the column, but short enough so that the analysis time is not significantly increased. Five meter guard columns are more cost effective, reduce the frustrations of making the connection between the guard column and analytical 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

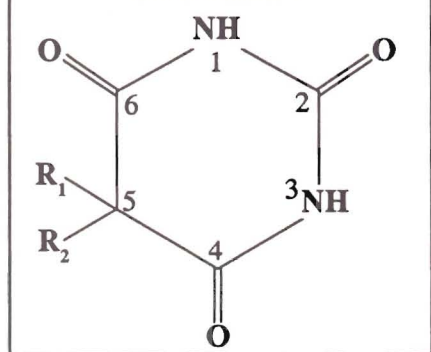
* Patent Pending



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

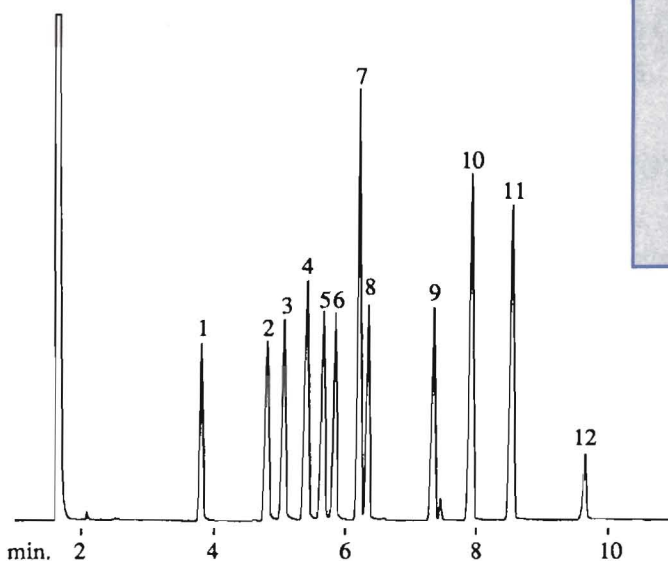
tetramethylammonium hydroxide (TMAH) and trimethyl-anilinium hydroxide (TMAH) can be used for on column derivatization of the barbiturates. While derivatization can improve the peak shape and response, extraneous peak formation can interfere with some analyses. Proper injection port set-up is important 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 Cycloplitters®, 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.



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™!**

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



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:

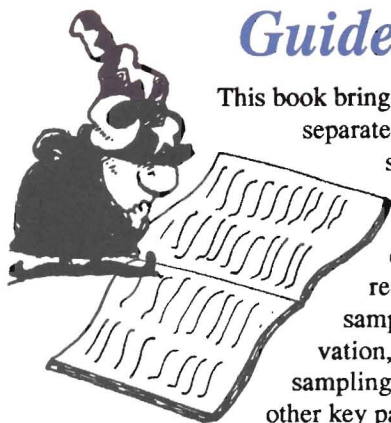
Rtx®-502.2, 105m, 0.53mm ID, 3.0µm (cat.# 10910)
MXT®-502.2, 105m, 0.53mm ID, 3.0µm (cat.# 70910)
Rtx®-624, 105m, 0.53mm ID, 3.0µm (cat.# 10975)
MXT®-624, 105m, 0.53mm ID, 3.0µm (cat.# 70975)

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 O₂ and H₂O 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.

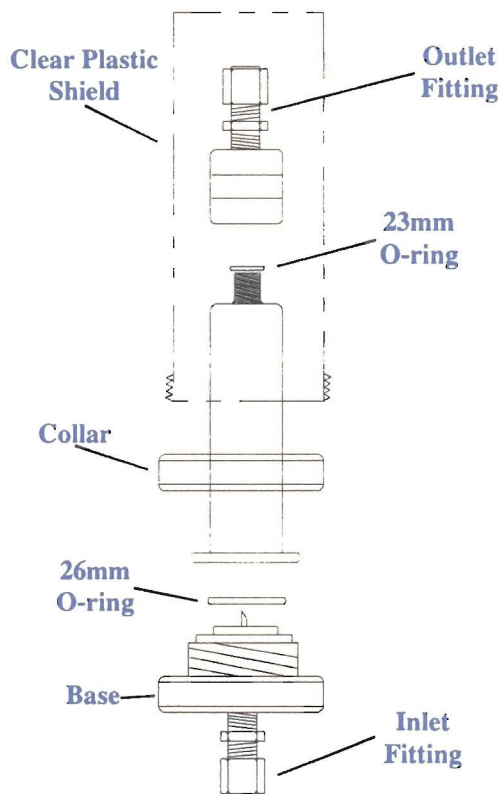
- Indicator changes from dark blue to brown as O₂ and H₂O are trapped.
- Heavy duty, sturdy body design virtually eliminates breakage.
- High capacity (>300s.c.f.) design lasts longer than three smaller traps.
- Economic replacement cartridges easily changed.
- Useable with multiple GC systems.
- Removes impurities at flow rates up to 230s.c.f./hr.
- Removes O₂ and H₂O 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



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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
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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.

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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.



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