

ENVIRONMENTAL

CHROMATOGRAPHY

PRODUCTS



From
Chromalytic

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Chromalytic Technology Pty Ltd



RESTEK
CORPORATION

Why choose Restek for environmental analysis supplies?

In just six years, Restek has grown into a leading supplier of environmental analysis products. We have accomplished this by developing innovative products to meet the needs of constantly changing EPA methodology requirements. Our dynamic, customer service oriented organization is always working to provide the best solutions to environmental analysis problems. And, we support our products with service after the sale. Restek's knowledgeable technical staff is always ready to assist with column selection, standard selection, methodology selection, or troubleshooting problems.

What products are available for environmental analyses?

Many products are available for environmental analyses including fused silica capillary columns, calibration standards, glassware, and deactivated inlet sleeves. Our fused silica capillary columns are widely used in environmental labs because of their excellent inertness, low bleed, high thermal stability, and long lifetime. Our environmental standards are specifically blended to meet EPA methodology requirements, and are manufactured and tested to rigorous quality control specifications. We supply sample extraction and concentration glassware designed to conform to EPA method protocols. Deactivated inlet sleeves, which reduce breakdown of active components, are available for most GC designs.

Columns

What do Restek columns offer over other brands of capillary columns?

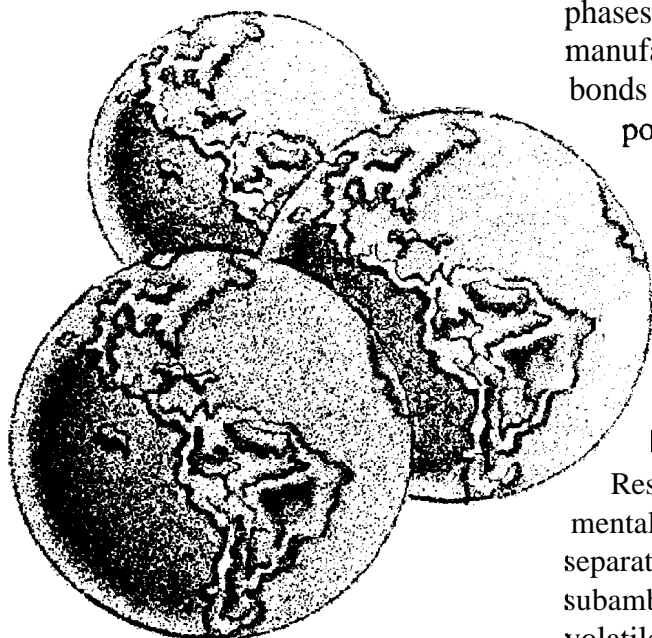
Although many companies sell fused silica capillary columns, only a few actually manufacture them. The features that set Restek columns apart from other column manufacturers are:

H Polymer Technology

Restek's polymer laboratory synthesizes all of its deactivants and stationary phases. This allows us to carefully control the materials that are used to manufacture our capillary columns. We have developed a unique process that bonds the liquid phase of the column to the tubing wall, and cross-links the polymer to itself. Most capillary columns are made using free radical generators, such as peroxides. Columns produced with these techniques have limited life expectancy, higher bleed, and decreased inertness. Restek's CROSSBOND" capillary columns are made using a proprietary process that eliminates the use of free radical generators. This process reduces column bleed, improves inertness, and produces columns that last longer.

■ Application Specific Capillary Columns

Restek has developed several capillary columns specifically for environmental analysis. The Rtx"-502.2 column was engineered to give maximum separation of volatile organic compounds without the need for cryofocusing or subambient cooling. XTI"-5 columns were developed specifically for semi-volatile organic analysis by GC/MS. They exhibit very low bleed, even with sensitive ion trap detectors. They also exhibit excellent response for active compounds such as phenols and benzidines.



Extensive Quality Control

Restek is the only capillary column manufacturer that uses temperature programmed test mixes to test column integrity. Although this method of column evaluation involves longer QA testing time, it is much more demanding than the faster isothermal test used by other column manufacturers. A programmed test mixture confirms the inertness, efficiency, polarity, and film thickness of each column and monitors bleed at the column's maximum operating temperature to ensure high column quality and reproducibility from column-to-column.

Technical Support

Our knowledgeable technical support staff is available to answer your environmental analysis questions Monday through Friday, 7:30 a.m. to 8 p.m., and Saturdays, 10 a.m. to 2 p.m. EST. Just call us toll-free at 800-356-1688.

■ 100 % Satisfaction Guarantee

Restek is committed to supplying the highest quality products available. If you are not satisfied with any of our products, we will do whatever is necessary to resolve the problem.

How do you select the column that is right for your application?

Choosing the right column for a particular analysis and equipment can be very difficult. If, after reviewing the applications guide, you are still uncertain which column is the best for your needs, call our experienced technical service department at 800-356- 1688.

Chemical Standards

How do Restek's environmental standards compare to other commercially available materials?

There are several companies that supply standards for environmental analysis. The features that set Restek standards apart from other reference material suppliers are:

Quality Control Documentation

Restek promotes a total quality program that begins before the raw materials arrive at our facility. 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 have 98% or greater purity.

Our analytical balance calibrations are verified daily at six mass levels using ASTM class 1 weights. In addition, all balances are certified traceable to NIST weights annually. Two chemists independently prepare identical mixtures for each product. These mixtures are then analyzed in triplicate. 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.

The latest Superfund statement of work requires laboratories to have detailed quality documentation on file for commercially purchased standards. Restek's data packs provide the necessary information for EPA audit documentation. Many companies do not have the time or capacity to perform all of the QA testing for each mixture used in the lab. We start with the QA documentation on each raw material used in a mixture. This includes the measured melting point (solids) or the refractive index (liquids) for each compound. Copies of the GC/FID purity check chromatograms, GC/MS total ion chromatograms, and mass spectra are also in the data pack. Each mass spectrum is compared to a library spectrum to ensure correct compound identification.

Our data packs contain more than just raw material tests. We include a lot sheet showing the exact gravimetric weight of each analyte and the volume of solvent used for dilution, a copy of the analytical balance printout with each analyte and lot number clearly identified, the statistical results from both mixtures, copies of all the chromatograms, and a copy of the Certificate of Analysis for the product purchased.

For laboratories not performing Superfund analyses, the data pack can be used by internal QA departments to verify the standard's integrity.

High Concentration for Maximum Value

Most chemical standards are produced at concentrations from 1000ug/ml to 2000ug/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.

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 so that there is little chance that active analytes can adsorb to the glass walls. We package every ampule in a flex square containing an ampule breaker to safely open the ampule, a deactivated vial to store unused portions of the mixture, and an extra label. Several mixtures are offered in varying concentrations and package sizes for different size laboratories.

Custom Standards

Restek manufactures custom chemical standard mixtures. We can perform custom testing and include a data package if requested.

Quotations for custom chemical standards are normally supplied within 48 hours after receiving your request. Most custom chemical standards can be made and shipped within 7-10 days after receipt of your order. (Extremely complicated mixtures may take slightly longer.) If your list of components is extensive, we ask that you fax us the list at 814-353-1309 to expedite your quotation.

When requesting a quotation for custom chemical standards, please have the following information available:

- list of components & concentration levels
- solvent to be dissolved in
- type and extent of QA required on final mixture
- documentation needed
- number of ampules and volume per ampule
- required delivery date

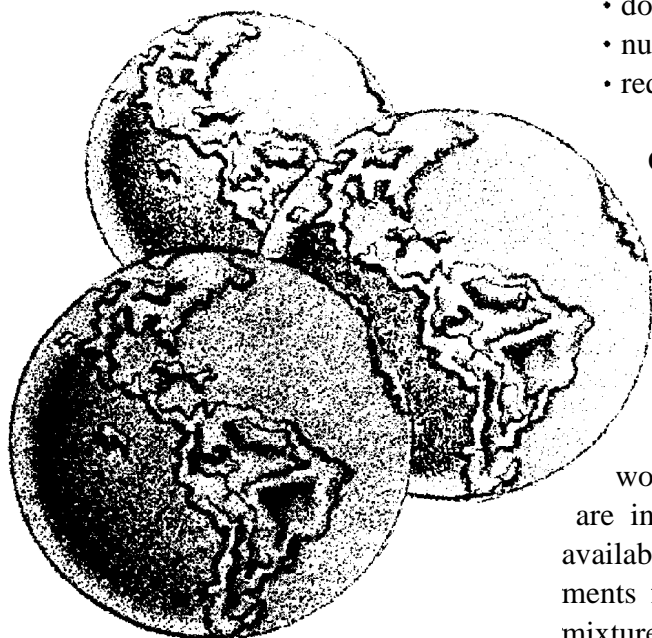
Call us and we'll be your #1 source for custom chemical standards!

Suggestion: The cost per ampule is significantly reduced when ordering 10+ ampules of the same mixture. This is because a minimum volume of the mixture must be prepared to accurately weigh all of the components.

Comply With all EPA Specifications

Our line of standards for the EPA Contract 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.

At Restek, in stock means exactly that! We ship over 99% of chemical standard orders the same day. And, our standards are 100% Satisfaction Guaranteed.



Commonly Asked Questions About Environmental Analysis

Can capillary columns be used for EPA packed column methods?

Capillary columns provide superior results compared to packed columns and many newer EPA methods include the use of capillary columns. All packed column methods developed by the US EPA include a disclaimer which allows other columns, including capillary columns, to be used if certain quality control criteria are met. The final decision on whether data generated from a capillary column is acceptable to either a state or a federal environmental agency rests with the quality control officer involved in the program. If you are unsure whether you can use capillary columns, consult the QA officer from your regional EPA office or your state's environmental agency.

what EPA methods specify capillary columns?

Many new methods developed by the EPA include the use of capillary chromatography. Table I is a brief listing of these capillary methods.

Table I - EPA methods specifying capillary columns.

EPA Method(s)	Method Description
502.2 & 8021	volatile organic compounds in water by purge & trap capillary gas chromatography
504 & 8011	EDB & DBCP in water by microextraction and capillary gas chromatography
505	organohalide pesticides in water by microextraction and capillary gas chromatography
507	nitrogen & phosphorus-containing pesticides in water by capillary gas chromatography
508	chlorinated pesticides in water by capillary gas chromatography
515.1	chlorinated acids in water by capillary gas chromatography
524.2 & 8260	volatile organic compounds in water by purge & trap capillary GC/MS
525	organic compounds in drinking water by liquid-solid extraction and capillary GC/MS
8141	organophosphorus pesticides by capillary gas chromatography
8270	semi-volatile organics by capillary GC/MS

Where are EPA methods obtained?

There are three sets of environmental analytical methods developed by the U.S. EPA based upon sample matrix. The 500 series methods were developed for the analysis of drinking water and source water. The 600 series methods were developed for the analysis of municipal and industrial discharge water. The EPA's solid waste testing manual (SW-846) contains the 8000 series methods. EPA methods are available from the following locations:

800 Series Methods

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
(703) 487-4600
Document Numbers:
PB89-220461 (Volume 1)
PB90-2 15039 (Volume 2)

600 Series Methods

Environmental Monitoring and Support
Laboratory
US EPA
Cincinnati, OH 45268
(513) 569-7562

8000 Series Methods

U.S. Government Printing Office
Washington, DC 20402
(202) 783-3238
Document number:
955-001-00000-1

Can Restek columns be used in place of the columns recommended in the EPA methods?

The capillary methods developed by the EPA allow equivalent columns to be used in place of the brand of column cited in the methods. Restek offers capillary columns that are equivalent to virtually all the capillary columns cited in EPA methods. If you are unsure of which Restek column is equivalent to the one listed in the method, just call our technical service department at 800-356-1688.

How long does a capillary column usually last?

Columns can last as little as two weeks or as long as two years depending upon sample matrices and column care. Non-volatile residue from the sample can deposit on the front end of the column, reducing the performance of the column and eventually making it unusable. Columns that are heated beyond their maximum temperature or operated with impure carrier gas will degrade rapidly. Even minor leaks on the inlet side of the carrier flow path can introduce air into the column, oxidizing the stationary phase and causing premature failure.

Can a capillary column be rejuvenated?

Normally, non-volatile residue tends to accumulate in the first meter or so of a column. Removing one or two loops from the inlet side of the column can usually restore a column's performance. This can only be done a few times before column length is reduced enough to affect resolution.

Another way to remove the soluble, non-volatile residue is to rinse the column with an appropriate solvent. Since Restek capillary columns are Crossbonded, solvent rinsing with most solvents will not remove the liquid phase. Recommendations for solvent rinsing capillary columns can be found in Restek's *Fused Silica Capillary Column Installation Guide*.

Can a capillary column be protected from non-volatile residue?

Rinsing the column with solvent is only effective in removing the soluble residue. Often the residue left inside the column is no longer soluble because it has been pyrolyzed inside the column. Better protection and longer column lifetime can be obtained by using a guard column connected to the front of your analytical column (Figure 1).^{*} A guard column is an uncoated length of deactivated fused silica tubing. It can easily be attached to the analytical column with a universal Press-Tight Connector (cat.# 20400). Table II shows a listing of guard columns available from Restek.

Since the guard column is uncoated, interaction time between the sample and residue buildup is minimized. This allows more injections before a section of the guard column must be removed. As the guard column becomes contaminated, one or two loops can be removed from the front to restore performance without remaking the "Press-Tight" connection. Once the guard column is expended, a new one can be attached to the analytical column. Using guard columns precludes the need to shorten the analytical column and results in longer column life.

^{*} Choose a guard column with the same diameter as your analytical column.

Other useful technical information can be found in the following guides available from Restek:

*Helpful Hints for Analyzing
Volatile Organics*

*Operating Hints for
Split/Spitless Injectors*

*Guide to Minimizing
Septa Problems*

*A Guide When Injecting
Dirty Samples*

*Fused Silica Capillary Column
Installation Guide*

Wizard Reference Wall Chart

Call 800-356-1688 to request
these publications.

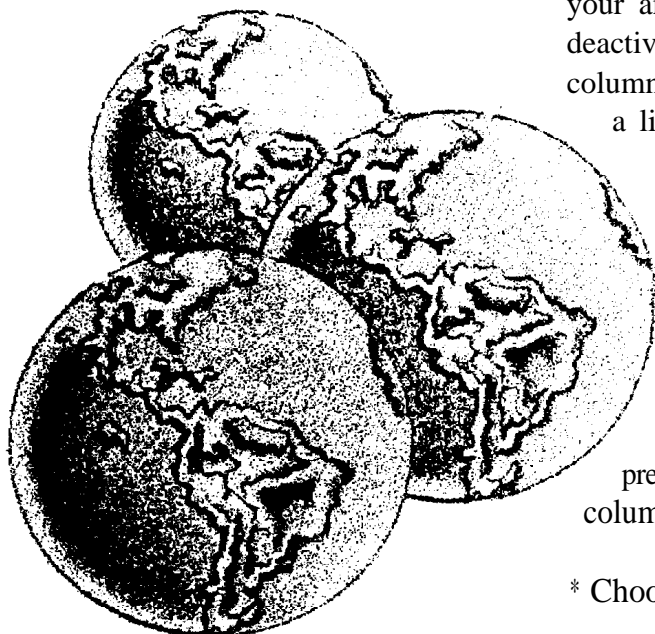


Figure 1- A guard column connected to an analytical column increases column lifetime and protects the analytical column from contamination.

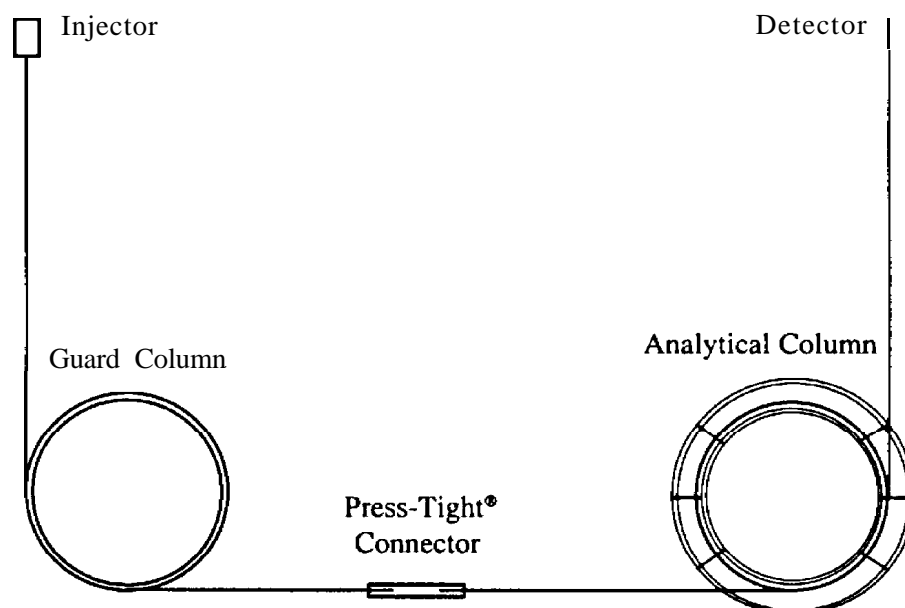


Table II - S-meter length Guard Columns & Press-Tight" Connectors

5meter Length Guard Columns

ID	OD	cat.#	price
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	

Universal Press-Tight" Connectors

cat.#
20400, \$35, 5-pack
20401, \$150, 25-pack
20402, \$425, 100-pack

Volatile Organics by Capillary Gas Chromatography

(EPA Methods 502.2, 524, 524.2, 601, 602, 8010, 8020, 8240)

Column Selection

Volatile organic pollutants are generally analyzed by purge & trap techniques, Choosing the most appropriate capillary column for purge & trap analyses is very dependent on the compounds you plan to analyze and type of equipment you are using. Different EPA methods for volatile organics contain different analyte lists. The EPA 600 Series methods for waste water contain only 35 volatile organic analytes, the CLP method contains 42, and the 500 Series methods for drinking water contaminants contain 60. Your choice of columns may differ depending upon the method you are following.

The type of purge & trap system you use effects column choice. If your purge & trap system has the capability of secondary trapping or cryofocusing, either narrow (0.25 & 0.32mm ID) or wide bore (0.53mm ID) capillary columns can be used. Purge & trap systems without these features are limited to wide bore capillary columns.

Narrow Bore Columns

The flow constraints of a purge & trap system require a high desorption flow to rapidly transfer the sample from the trap onto the analytical column. Since narrow bore (0.25 & 0.32mm ID) capillary columns are most efficient when operated under low flow conditions, interfacing capillary columns to purge & trap systems has been difficult. Several techniques have been developed for doing this. These techniques usually involve either cryofocusing, subambient cooling, splitting the desorb flow, or secondary trapping on a smaller diameter trap to reduce the desorb flow to a range that is compatible with narrow bore capillary columns (1 to 2cc/min.).

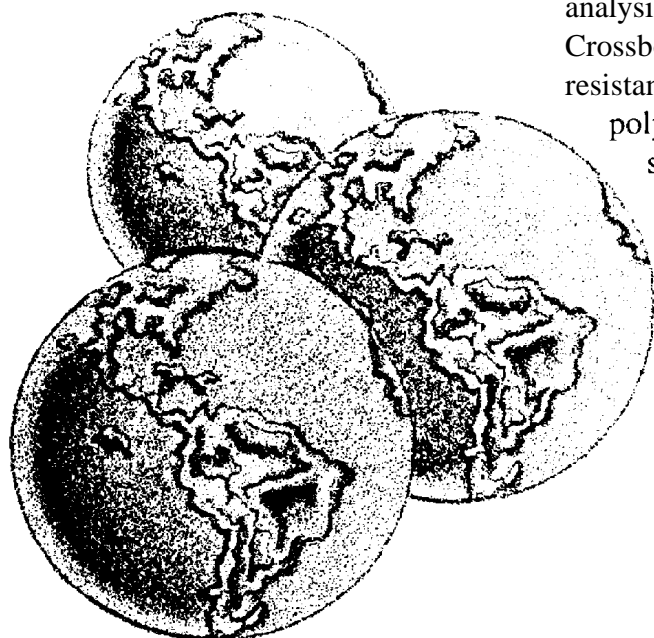
Figure 2 shows the analysis of EPA Method 524 compounds on a 30m, 0.25mm ID, 1.8µm RtxYj02.2 column using a cryofocusing interface between the purge & trap system and the GC. The cryofocusing interface steps down the desorb flow to a level that is compatible with a narrow bore column. Cryofocusing interfaces require the use of liquid nitrogen, which makes automation more difficult.

Another technique used to interface narrow bore columns to purge & trap systems is subambient cooling of the column. Liquid nitrogen or carbon dioxide is used to cool the GC oven so that the volatile organic compounds can condense at the front of the column. Figure 3 shows the analysis of EPA CLP volatiles on a 60m, 0.32mm ID, 3.0µm Rtx"-Volatiles that was run with the GC oven cooled to 10°C. By subambiently cooling the GC oven, the separation of the early eluting gases can be achieved, even with slow trap desorption flows.

Wide Bore Columns

Wide bore (0.53mm ID) capillary columns can be operated at higher flow rates, allowing them to be directly interfaced to purge & trap systems. Higher column flow rates result in faster trap desorption flows, producing narrower band widths and sharper peaks. By directly interfacing the purge & trap system to the column, the chance for sample loss is minimized. Although less efficient than narrow bore columns, wide bore columns provide greater sample capacity and are the choice of most analysts. This larger capacity allows calibration over a wider range of concentration before column overloading occurs. To overcome the reduced efficiency of wider bore columns, longer length columns can be used. In addition, specialized phases, tailored to the analysis of volatile organic pollutants, have been developed to improve separations. However, the separation of very volatile gases can be poor and requires subambient cooling (Figure 4). To combat this problem, Restek has developed a thick film, 105-meter column to eliminate the need for cryofocusing or subambient cooling. This 0.53mm ID, 3.0µm Rtx"-502.2 column never needs liquid nitrogen or CO₂. Figure 5 shows the analysis of EPA Method 502.2 compounds separated on this column using a Tekmar® purge & trap system interfaced to a photoionization detector (PID) in series with an electrolytic conductivity detector (ELCD). Figure 6 shows the same analysis using an OI purge & trap system. Rtx"-502.2 columns are Crossbonded with a dimethyl diphenyl polysiloxane stationary phase that is resistant to water vapor or solvent damage. The thermal stability of this polymer exceeds 290°C, and exhibits low bleed with sensitive detectors such as PIDs, ELCDs, and MSDs.

Restek's Rtx"-502.2 columns are Crossbonded with dimethyl diphenylsiloxane stationary phase that is resistant to water vapor or solvent damage. The thermal stability of this polymer exceeds 290°C, and exhibits low bleed with sensitive detectors such as PIDs, ELCD and MSDs.



0.25 & 0.32mm ID columns

Advantages

- higher efficiency
- directly interfacable to mass spectrometers

Disadvantages

- not directly interfacable to purge & trap systems
- limited sample capacity

0.53mm ID columns

Advantages

- directly interfacable to purge & trap systems
- column flow rates compatible with trap desorption flow rates
- large sample capacity

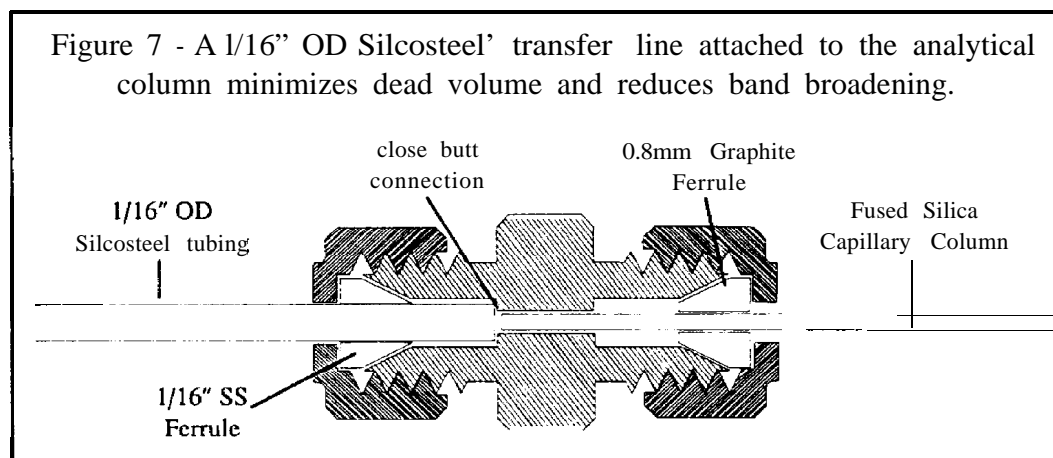
Disadvantage

- requires jet separator or open split for mass spectrometer interface

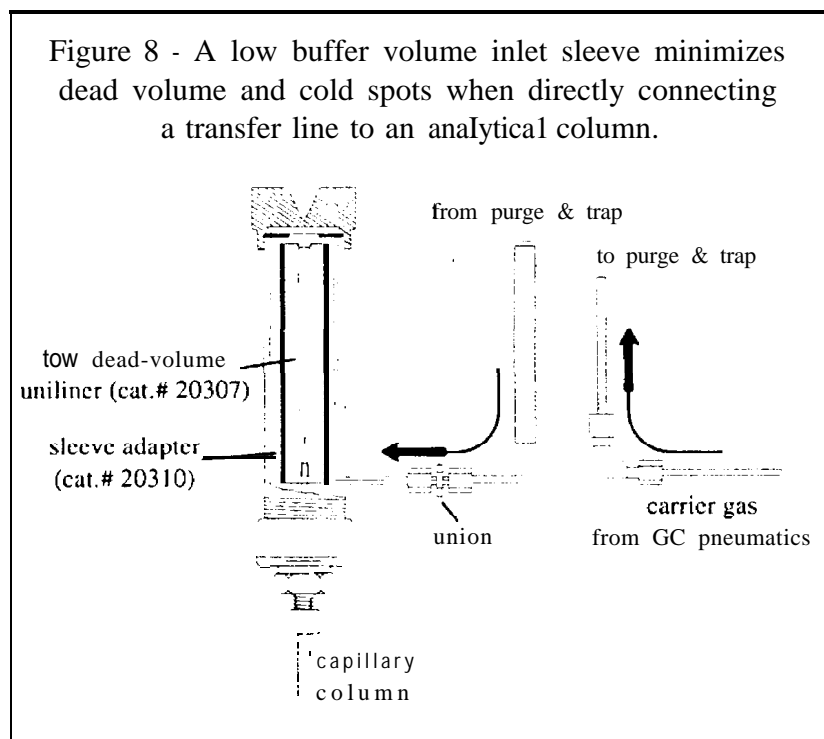
Interfacing Capillary Columns to Purge & Trap Systems

The connection between the transfer line from the purge & trap and the column can effect the resolution of the volatile organic compounds. Any excess dead volume in a capillary system can cause loss in resolution and should be avoided. The internal volume of the transfer line should be kept to a minimum. The use of 0.53mm ID Silcosteel" or fused silica tubing will help reduce band broadening in the transfer line.

The preferred interfacing method, shown in Figure 7, is to directly couple the transfer line to the column using a 1/16" stainless steel union (cat.# 21918) and 1/16"OD x 0.020"ID Silcosteel" cat.# 20524). This method prohibits direct injections from being made, but eliminates dead volume problems that can be caused by the injection port. Resolution and peak shapes will be greatly improved with direct coupling.



If you find it necessary to make direct injections onto the column, minimize any dead volume that may exist in the injection port. This method can be accomplished by using a sleeve with a small inner diameter (Figure 8). We recommend using a Uniliner" without a buffer volume (cat.# 20307) in conjunction with a metal sleeve adaptor (cat.# 20310). Dead volume can also be reduced by making the transfer line connection as close to the injector body as possible. This also reduces the chance for cold spots that can cause condensation of water or high molecular weight compounds in the transfer line connection.

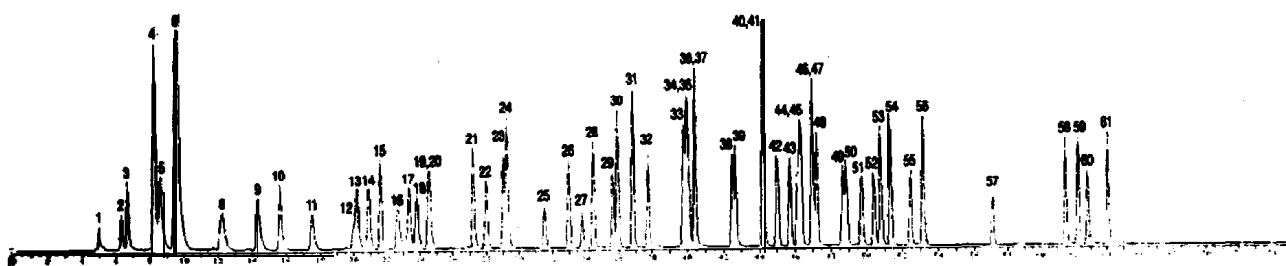


This also reduces the chance for cold spots that can cause condensation of water or high molecular weight compounds in the transfer line connection.

Interfacing Capillary Columns to Detection Systems
Column choice is also dependent upon the type of detection system you plan to use. The analysis of volatile organic pollutants is generally done with either PID/ELCD detectors in series (methods 601/602, SOIO/8020, 502.2), or by MS (methods 624, 8240, 524.2). Because band broadening can also occur in a detection system, it is important to minimize dead volume within the detection system when using capillary columns. The transfer line between the PID and ELCD detectors must be minimized with the use of narrow bore tubing. It is also critical that the ELCD detector be designed with a micro detector cell for capillary analysis. In addition, make-up gas must be added to ensure that the sample is swept through the detector quickly.

The low flow rates of narrow bore capillary columns are ideal for the vacuum systems of mass spectrometers (1 or 2cc/min. flows), but they require cryofocusing or secondary trapping on a smaller diameter trap. If the purge & trap equipment does not have these capabilities, or you prefer to use wide bore capillaries with your mass spec, then an open split interface or a jet separator will be required. With an open split interface, as much as 90% of the sample can be lost, significantly reducing sensitivity. Interfacing with a jet separator is the preferred approach since it will reduce the carrier flow without a significant loss in sensitivity. (Note, it is very important to use make-up gas for proper operation of a jet separator.) Since MS detection provides the added ability of separating coeluting compounds by their unique quantitation ions, shorter length columns with less chromatographic resolution can be used. Figure 9 shows a comparison of the separation of EPA Method 524.2 compounds on a 60- and a 105meter Rtx-502.2 column using GC/MS detection. Although the 60-meter column does not chromatographically resolve as many components as the 105meter column, quantitation with the mass spectrometer can still be accomplished using ion profiles unique to each compound. The 60-meter column also produces faster analysis times resulting in increased sample throughput.

Figure 9 - Use a 60m, 0.53mm ID, 3.0um Rtx"-502.2 for EPA Method 524.2 to increase analysis times and sample throughput. 1



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

Tekmar purge & trap concentrator

Silica gel / tenax trap - 11 min. purge 4 min. desorb, 7 min. bake

Oven temp.: 35°C (hold 10 min.) to 200 @ 4C/min.

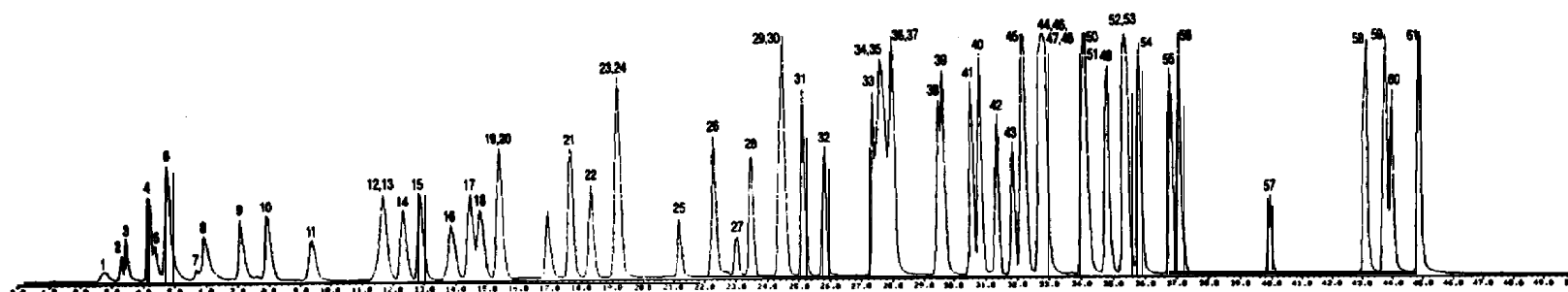
Carrier gas: helium

Linear velocity: 80cm/sec.

Preheat temp.: 198C

Desorb temp: 200C

Trap bake: 225C



105m, 0.53mmID, 3.0um Rtx-502.2(cat.# #0910).

Tekmar purge & trap concentrator

Silica gel / Tenax trap - 11 min. purge, 4 min. desorb

Oven temp.: 35°C (hold 10 min.) to 220°C @ 3C/min.

Carrier gas: helium

Flow rate: 7cc/min.

Preheat temp.: 198C

Desorb temp.: 200°C

Trap bake: 225C

Peak List for Figures 9-10

502.2 Volatile Organics Kit (cat.# 300 0)			
1	carbon dioxide	22	1,2-dichloropropane
2	chloromethane	23	bromodichloromethane
3	vinyl chloride	24	dibromomethane
4	bromomethane	25	cis-1,3-dichloropropene
5	chloroethane	26	toluene
6	trichlorofluoromethane	27	trans-1,3-dichloropropene
7	acetone	28	1,1,2-trichloroethane
8	1,1-dichloroethene	29	1,3-dichloropropane
9	methylene chloride	30	tetrachloroethene
10	trans-1,2-dichloroethene	31	dibromochloromethane
11	1,1-dichloroethane	32	1,2-dibromoethane
12	2,2-dichloropropane	33	chlorobenzene
13	cis-1,2-dichloroethene	34	1,1,1,2-tetrachloroethane
14	chloroform	35	ethyl benzene
15	bromochloromethane	36	p-xylene
16	1,1,1-trichloroethane	37	m-xylene
17	1,1-dichloropropene	38	o-xylene
18	carbon tetrachloride	39	styrene
19	1,2-dichloroethane	40	isopropylbenzene
20	benzene	41	bromoform
21	trichloroethene	42	1,1,2,2-tetrachloroethane
43	1,2,3-trichloropropane		
44	n-propylbenzene		
45	bromobenzene		
46	1,3,5-trimethylbenzene		
47	2-chlorotoluene		
48	4-chlorotoluene		
49	tert-butylbenzene		
50	1,2,4-trimethylbenzene		
51	sec-butylbenzene		
52	p-isopropyltoluene		
53	1,3-dichlorobenzene		
54	1,4-dichlorobenzene		
55	n-butylbenzene		
56	1,2-dichlorobenzene		
57	1,2-dibromo-3-chloropropane		
58	1,2,4-trichlorobenzene		
59	hexachlorobutadiene		
60	naphthalene		
61	1,2,3-trichlorobenzene		

Permission to publish these chromatograms granted by Centre Analytical Laboratories, Inc.

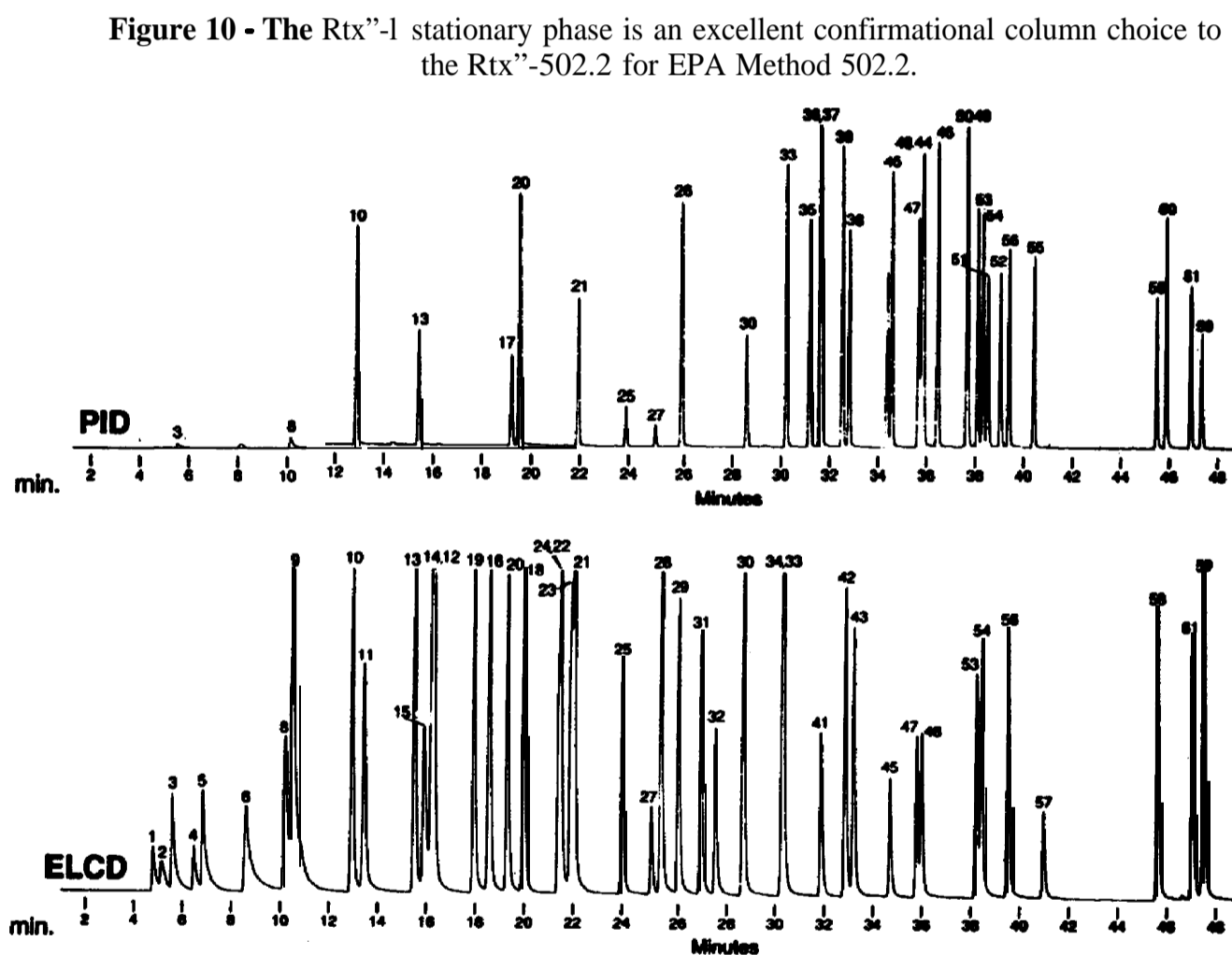
Confirmational Analysis

Confirming the identity of volatile organic compounds can be accomplished several ways. With mass spec analysis, confirmation is accomplished by matching the spectral pattern and the retention time to a known standard. With PID & ELCD detectors in a series, confirmation is more difficult. Often selective response, or lack of response on one or both of the detectors, can be useful in confirming the identity of some compounds. A more accurate confirmation technique is to use a different polarity column and match retention time shifts. Figure 10 shows the analysis of EPA Method 502.2 compounds on an Rtx"-1 column. The elution order and pattern for the volatile organic compounds is significantly different than the Rtx"-502.2 column, allowing more positive quantitative and qualitative confirmations.

Other Factors Affecting Capillary Analysis by Purge & Trap

Trapping Materials

There are various types of trapping materials used for purge & trap analysis. The trapping materials recommended in EPA methods include Tenax", silica gel, and charcoal. Tenax" is not capable of trapping the very light gases, so unless silica gel or charcoal is included in the trap, these components will not be recovered. Silica gel is capable of trapping the volatile gases except dichlorodifluoromethane, which can be trapped by charcoal. The drawback to silica gel is that it retains large amounts of water vapor. This water vapor, when desorbed from the trap, can cause problems with some detection systems including PID and GC/MS.



105m, 0.53mm ID, 3.0um Rtx"-1 (cat.# 10189).
Tekmar UC-2000 purge & trap concentrator
Carbosieve"/Carbopack" B trap - 11 min. purge, 3 min. desorb
Oven temp.: 35C (hold 10 min.) to 220°C @ 3C/min. (hold 5 min.)
Inj.det. temp.: 200°C/250°C
Carrier gss: helium
Linear velocity: 80cm/sec. (flow rate: 10cc/min.)
Preheat temp.: 245C **Desorb temp.:** 250C
Tra bake: 270C

Acknowledgment: Varian 3600 GC and Tekmar purge & trap provided through the courtesy of Varian.
Trapping materials provided through the courtesy of Tekmar.

Alternative trapping systems have been developed that employ more hydrophobic materials so water vapor can be eliminated from the trap by using a dry purge cycle between the purge and desorb steps. One of these systems uses Carbosieve[®], SIII and Carbopac[®] B to trap the volatile organic compounds. The thermal stability of these materials allows them to be desorbed at higher temperatures (250°C) than the EPA recommended materials (180°C). The higher desorb temperatures speed the release of the volatile organic compounds, resulting in sharper peaks and better resolution of the early eluting gases. Other trapping systems have been developed that employ other types of adsorbents to improve recoveries and desorption profiles.

Desorb Flow Rates:

The desorb flow rate controls the speed in which the compounds are removed from the trapping materials and transferred onto the column. Since the desorb flow is also the carrier gas flow, it controls the speed in which the compounds move through the column. If the desorb flow is set too low, the compounds will not be released from the trap rapidly enough, resulting in broad peaks and loss of sensitivity. If the desorb flow is set too high, the column will be less efficient and poor resolution will result. When operating a wide bore column with a purge & trap system, a flow of 8 to 10cc/min. allows the compounds to be transferred from the trap rapidly enough without significant loss in resolution.

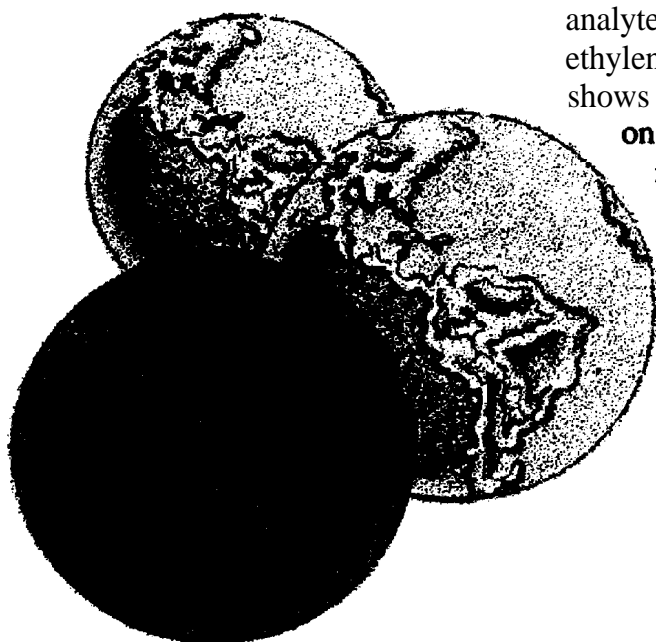
Transfer Line & Valve Oven Temperatures:

The transfer line and valve oven temperature should be kept above the boiling point of water so that water is kept in the vapor state and does not condense within the purge & trap system. (Note, under normal pressures used for P&T systems and .53 capillary columns, the boiling point of water is elevated to -120°C, so the transfer line should be set at -125°C.)

Liquid/Liquid Extraction Methods

EPA Methods 501.3 and 504

In addition to purge & trap methods, there are some liquid/liquid extraction methods for volatile organic contaminants in drinking water. These methods have limited applicability and were developed for a small number of specific analytes. EPA Methods 501.3 and 504 were developed for the analysis of ethylene dibromide (EDB) & dibromochloropropane (DBCP). Figure 11 shows the separation of these two compounds plus the four trihalomethanes on a 30m, 0.53mm ID 2.0µm Rtx[®]-Volatiles column in less than 13 minutes.



Restek's Technical Service Department is ready to help you Monday-Friday, 7:30a.m.-8p.m., and Saturdays, 10a.m.-2p.m.

**501 Trihalomethane Mix (cat.# 30036)
& 504 EDB/DBCP Mix (cat.# 30034)**

1	chloroform
2	bromodichloromethane
3	dibromochloromethane
4	dibromoethane (EDB)
5	bromoform
6	1,2dibromo3-chloropropane (DBCP)

30m, 0.53mmID, 2.0um Rtx"-Volatiles (cat.# 10902).

1.0ul direct injection

Concentration approximately : 200pg/ul

Oventemp.: 50C (hold 6 min.) to 200C @ 1 0C/min_

Inj. & det.temp.: 250°C

Carrier gas: hydrogen

Linear velocity: 80cm/sec. (flow rate: 10cc/min.) set @ 50C

ECD sensitivity: 256 x 10-11AFS

Figure 11 - Achieve baseline resolution of EPA Method 501.3 and 504 analytes on a 30m, 0.53mm ID, 2.0um Rtx"-Volatiles in less than 13 minutes.



Semi-Volatile Organics by Capillary Gas Chromatography

(EPA Methods 604, 605, 606, 607, 609, 610, 611, 612, 625, 8040, 8060, 8090, 8100, 8120 8250, 8270)

Column Selection for Semi-Volatile Compounds

Semi-volatile organic pollutants encompass a broad range of compound classes. There are several EPA methods for the analysis of different semi-volatile compound classes. These GC methods use specific detectors (FID, ECD, NPD) depending upon the type of compounds to be analyzed. There are also GC/MS methods which were developed for the simultaneous analysis of several compound classes. These GC/MS methods allow the combination of acid and base/neutral extracts into a single analysis.

Capillary columns provide the inertness and resolution necessary to analyze this wide range of semi-volatile compounds. A 5% diphenyl/95% dimethyl polysiloxane liquid phase (SE-54) has been found to be the best choice for the analysis of most semi-volatile environmental contaminants. Restek offers three types of columns with this polarity stationary phase. The regular Rtx"-5 is a bonded phase column with good inertness and thermal stability to 325C. The XTI"-5 is also a bonded phase column that has improved inertness characteristics for active compounds, and increased thermal stability to 360C. The latest innovation has been to combine the XTI" technology with Silcosteel" tubing to produce MXT"-5 metal capillary columns. These metal columns have the same inertness as fused silica but are virtually unbreakable. Both the XTI"-5 and MXT"-5 columns are tested with a special mix containing many environmental compounds that are difficult to chromatograph.

Internal Diameter

Capillary columns are available in a range of internal diameters from 0.18 to 0.53mm. Column efficiency increases as the inside diameter decreases. In general, smaller diameter columns provide better separations than wider bore columns, but sample capacity decreases as the column's diameter decreases. This affects the linear calibration range since column overload will occur. The choice of column diameter is often dependent upon the type of equipment used for analysis. Narrow diameter columns are generally operated at lower flow rates which make them more compatible with the limited pumping capacity of bench-top mass spectrometers, such as MSDs and ITDs. However, the lower flow rates used with narrow bore columns can tend to prolong analysis time. Mass spectrometers with larger pumping systems can use 0.32mm ID columns to improve Sample capacity and reduce analysis time. The higher flow rates used with 0.53mm ID columns will overload the vacuum of a GC/MS system, necessitating the use of a jet separator or open split interface. Although a 0.53mm ID column has half the theoretical plates of a 0.25mm ID column, it offers acceptable resolution and faster analysis times for many EPA methods. Either the 0.32 or the 0.53mm ID column can be operated in the splitless or direct injection mode, offering more versatility than the smaller diameter columns.

Length

Most semi-volatile pollutant analyses are done with a 30meter column. This length provides the necessary resolution without prolonging analysis time, When analyzing a limited number of semi-volatile compounds or certain specific classes of compounds, a 15meter column may be sufficient.

Film Thickness

Film thickness effects sample capacity, analysis time, and usable upper temperature limits of the liquid phase. Increasing the liquid phase's film thickness increases the sample capacity but also lengthens analysis time. Thicker film columns tend to exhibit higher bleed, which reduces their maximum operating temperature. Since narrow diameter columns (0.18, 0.25, and 0.28mm ID) are operated at low flow rates (<1cc/min.), thinner films (<0.5µm) are used to minimize analysis time. Because higher flow rates are normally used with 0.32mm ID columns, film thicknesses up to 1.0µm can be used without prolonging analysis time. Film thicknesses from 0.5 to 1.5µm are generally used when analyzing semi-volatile compounds with 0.53mm ID columns.

Confirmational Analyses

When analyzing samples using non-GC/MS methods it is important to confirm the identity of specific analytes. This can be accomplished by running the sample and reference standards on two columns of differing polarity. Identification of an unknown peak can be made more accurately from two sets of data. A different polarity can often resolve the target compound from interfering components in the sample, improving quantitative accuracy. When selecting the confirmational column, choose a column with very different polarity to maximize the retention time shift. Columns coated with Rtx™-200, a methyl trifluoropropyl siloxane stationary phase, or Rtx™-50, a 50% dimethyl/50% diphenyl polysiloxane stationary phase, are both excellent choices for confirmation of many EPA methods,

Recommendations for primary analytical columns and confirmational columns are listed in the convenient wall chart located at the center of this catalog (Ask for it!)

Factors Influencing the Analysis of Semi-Volatile Pollutants

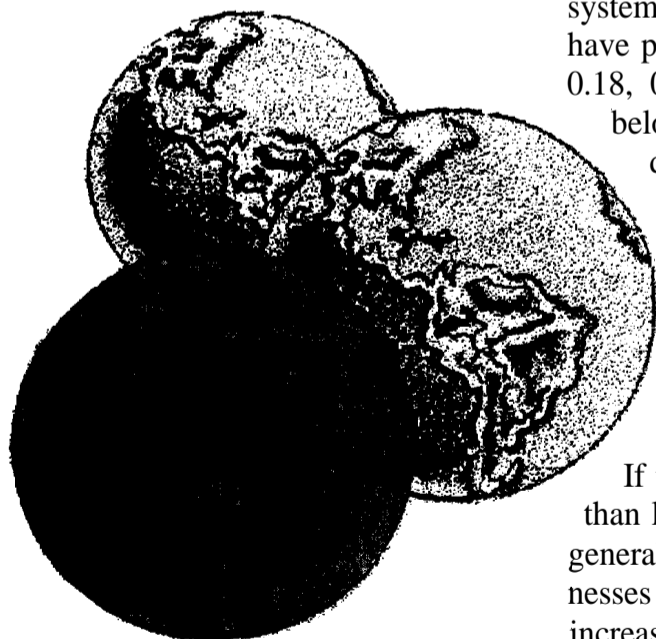
If your GC is equipped with a split/splitless injection port, you can use any diameter column. Instruments equipped with only a packed column injector are limited to 0.32 or 0.53mm ID columns. For most of the non-GC/MS methods, a 30m, 0.53mm ID, 1.5µm column has the necessary resolution and the capacity for handling most samples.

For GC/MS methods, the vacuum system adds a flow constraint to your choice of columns. If you wish to directly insert the column into the mass spec source then you are limited to the pumping capacity of your vacuum system. In general, small bench-top GC/MS systems, such as MSDs and ITDs have pumping capacities limited to ~1cc/min. This limits column choice to 0.18, 0.25, or 0.28mm IDs exhibit optimal resolving power at flows

below 1cc/min. We suggest that the film thickness of these narrow diameter columns be increased to 1.0µm to improve sample capacity.

However, thicker films combined with the low flow rates of narrow bore columns can result in very long analysis times. One way to overcome prolonged analysis times is to use XTI™-5 or MXT™-5 columns. These columns have higher thermal stability allowing them to be programmed to higher temperatures. Higher final operating temperatures result in shorter analysis times.

If the mass spectrometer vacuum system has a pumping capacity greater than 1cc/min., then 0.32mm ID columns can be used. These columns are generally operated with flow rates between 1.2 and 2.0cc/min. Film thicknesses of 1.0µm can be used with 0.32mm ID columns without a significant increase in analysis time. A 30m, 0.32mm ID, 1.0µm XTI™-5 column is ideal for the analysis of semi-volatile pollutants by GC/MS. It has ample capacity for calibration over a wide concentration range and thermal stability to 325°C, producing fast analysis times.



Confirmational Analysis

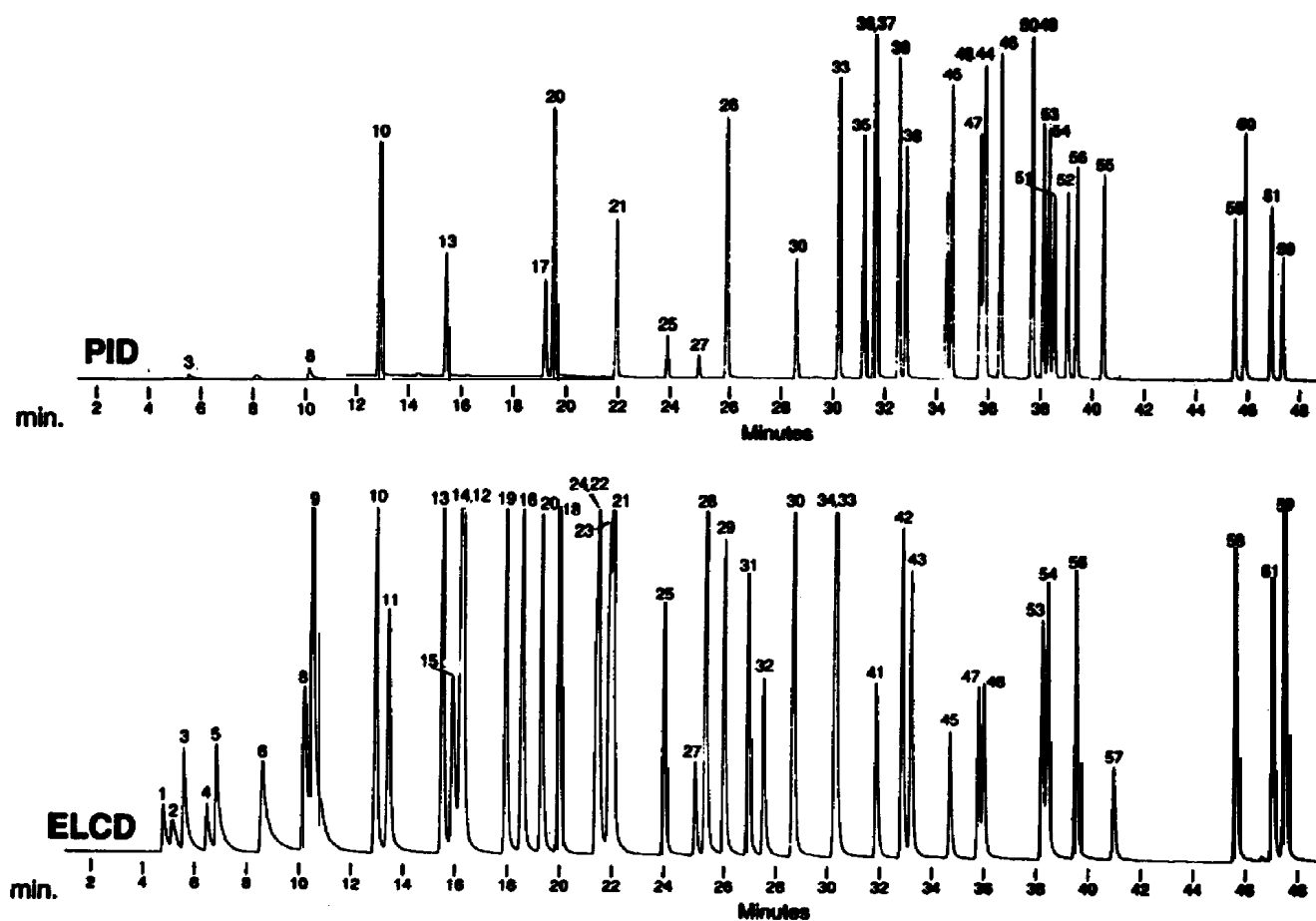
Confirming the identity of volatile organic compounds can be accomplished several ways. With mass spec analysis, confirmation is accomplished by matching the spectral pattern and the retention time to a known standard. With PID & ELCD detectors in a series, confirmation is more difficult. Often selective response, or lack of response on one or both of the detectors, can be useful in confirming the identity of some compounds. A more accurate confirmation technique is to use a different polarity column and match retention time shifts. Figure 10 shows the analysis of EPA Method 502.2 compounds on an Rtx"-1 column. The elution order and pattern for the volatile organic compounds is significantly different than the Rtx"-502.2 column, allowing more positive quantitative and qualitative confirmations.

Other Factors Affecting Capillary Analysis by Purge & Trap

Trapping Materials

There are various types of trapping materials used for purge & trap analysis. The trapping materials recommended in EPA methods include Tenax", silica gel, and charcoal. Tenax" is not capable of trapping the very light gases, so unless silica gel or charcoal is included in the trap, these components will not be recovered. Silica gel is capable of trapping the volatile gases except dichlorodifluoromethane, which can be trapped by charcoal. The drawback to silica gel is that it retains large amounts of water vapor. This water vapor, when desorbed from the trap, can cause problems with some detection systems including PID and GC/MS.

Figure 10 - The Rtx"-1 stationary phase is an excellent confirmational column choice to the Rtx"-502.2 for EPA Method 502.2.



105m, 0.53mm ID, 3.0um Rtx"-1 (cat.# 10189).

Tekmar UC-2000 purge & trap concentrator

Carbosieve"/Carbopack" B trap - 11 min. purge, 3 min. desorb

Oven temp.: 35C (hold 10 min.) to 220°C @ 3C/min. (hold 5 min.)

Injdet. temp.: 200°C/250°C

Carrier gss: helium

Linear velocity: 80cm/sec. (flow rate: 10cc/min.)

Preheat temp.: 245C

Desorb temp.: 250C

Tra bake: 270C

Acknowledgment: Varian 3600 GC and Tekmar purge & trap provided through the courtesy of Varian.
Trapping materials provided through the courtesy of Tekmar.

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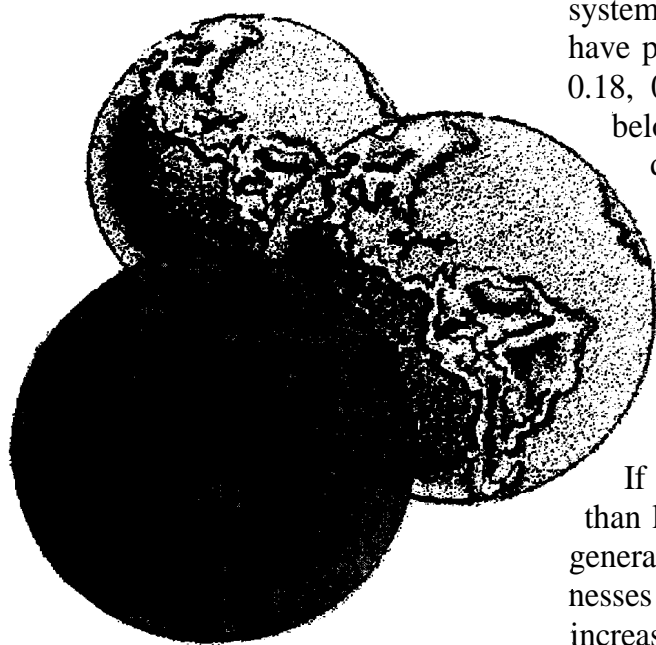
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30m, 0.53mmID, 2.0um Rtx"-Volatiles (cat.# 10902).

1.0ul direct injection

Concentration approximately: 200pg/ul

Oventemp.: 50C (hold 6 min.) to 200C @ 1 0C/min_

Inj. & det.temp.: 250°C

Carrier gas: hydrogen

Linear velocity: 80cm/sec. (flow rate: 10cc/min.) set @ 50C

ECD sensitivity: 256 x 10⁻¹¹AFS

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Length

Most semi-volatile pollutant analyses are done with a 30meter column. This length provides the necessary resolution without prolonging analysis time. When analyzing a limited number of semi-volatile compounds or certain specific classes of compounds, a 15meter column may be sufficient.

Alternative trapping systems have been developed that employ more hydrophobic materials so water vapor can be eliminated from the trap by using a dry purge cycle between the purge and desorb steps. One of these systems uses Carbosieve[®], SIII and Carbopac[®] B to trap the volatile organic compounds. The thermal stability of these materials allows them to be desorbed at higher temperatures (250°C) than the EPA recommended materials (180°C). The higher desorb temperatures speed the release of the volatile organic compounds, resulting in sharper peaks and better resolution of the early eluting gases. Other trapping systems have been developed that employ other types of adsorbents to improve recoveries and desorption profiles.

Desorb Flow Rates:

The desorb flow rate controls the speed in which the compounds are removed from the trapping materials and transferred onto the column. Since the desorb flow is also the carrier gas flow, it controls the speed in which the compounds move through the column. If the desorb flow is set too low, the compounds will not be released from the trap rapidly enough, resulting in broad peaks and loss of sensitivity. If the desorb flow is set too high, the column will be less efficient and poor resolution will result. When operating a wide bore column with a purge & trap system, a flow of 8 to 10cc/min. allows the compounds to be transferred from the trap rapidly enough without significant loss in resolution.

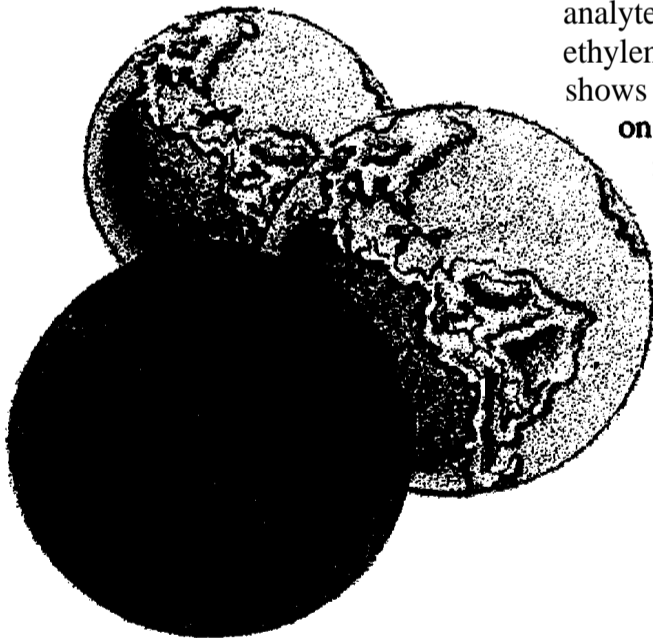
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