

Guide to Preparing and Analyzing Semivolatile Organic Compounds



Inside:

Introduction pg. 2

Extraction

Liquid samples pg. 2

Soil samples pg. 4

Cleanup pg. 5

Gel Permeation

Chromatography pg. 5

Analysis

Calibration standards pg. 6

Injection port

configuration pg. 6

Column selection pg. 9

Reducing

Discrimination pg. 15

Quantitation pg. 16

Summary pg. 16

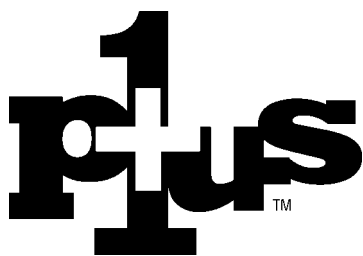
Product Listing pg. 17-27



www.restekcorp.com



The industry's best technical service
is only a phone call away! Call
800-356-1688, ext. 4
(814-353-1300, ext. 4) or email us at
support@restekcorp.com



Restek's Customer Commitment

Plus 1™ Service means we will surpass your expectations every time you contact us! You'll get Plus 1™ service when you ask our experienced Technical Service Team to help solve a difficult analytical problem. Our helpful, efficient Customer Service Team provides Plus 1™ service even when you place a late-day order. Keep reaching for Restek products and service, and we will provide you with Plus 1™ quality and attention.

Introduction

This technical guide addresses the preparation and gas chromatographic (GC) analysis of semivolatile organic compounds such as those listed in US Environmental Protection Agency (EPA) Methods 8270, 525, and 625; and polycyclic aromatic hydrocarbons (PAHs) such as those listed in US EPA Methods 610 and 8100. These analyses are some of the most common tests performed by environmental laboratories, yet there are many analytical challenges of which the analyst needs to be aware. For example, the samples often are highly contaminated with non-target compounds (e.g., hydrocarbons) and quality assurance/control (QA/QC) of the methods is rigorous. There are several procedures and techniques that can be employed, however, to make these analyses simpler to perform. Review this guide to learn these techniques and to troubleshoot analytical problems associated with the methods.

The compounds addressed in this guide are listed in Table I, but many additional compounds are also amenable to these semivolatile methods. Table I includes the compounds cited in the US EPA Methods, as well as some other compounds typically analyzed in environmental samples.

Extraction

The compounds listed in Table I may be difficult to extract because they fall into different chemical classes (i.e., acidic, basic, neutral, halogenated, oxygenated, polar, non-polar, low-boiling, and high-boiling compounds). Therefore, the extraction method will need to solvate a wide variety of compounds. It also must recover the analytes of interest while removing the interfering non-target contaminants. This limits the choices of cleanup options. A number of sample extraction methods can be applied to these compounds, but only the most common will be addressed in this guide.

Liquid Samples

For liquid samples, either separatory funnel extraction (US EPA Method 3510) or automated liquid-liquid extraction (US EPA Method 3520) may be used. Separatory funnel extraction is faster and less expensive to set up than the other methods, but it requires continuous operator attention. Automated liquid-liquid extractors run unattended, but are more expensive and, if analyte recovery is lower than allowed, re-extraction by separatory funnel may be required. Alternatively, if the sample forms an emulsion to the degree that acceptable solvent recovery is not possible using a separatory funnel, then some samples will require automated liquid-liquid extraction. Solid phase extraction (US EPA Method 3535) also is an option for aqueous samples.

For **separatory funnel extraction**, measure up to 1L of water into a 2L separatory funnel and adjust the pH to >11 using 10M NaOH; be careful not to add too much base. Then extract the sample by adding 60mL of dichloromethane and shaking for two minutes. It is critical to shake all samples consistently or variations in extraction efficiency will be observed. The best way to ensure consistency is to use a mechanical separatory funnel shaker, as there often is considerable variation with manual extractions. Allow the dichloromethane layer to settle to the bottom of the funnel and then decant that layer into a collection vessel (i.e., a Kurdena Danish [KD] concentrator, or a Turbo vap or Rapid vap® container if using automated concentrators). This extraction step is repeated twice more to get quantitative recovery of all analytes. Collect all three extractions into the same collection vessel and label as base/neutral.

Then adjust the water sample to a pH of slightly less than 2 using sulfuric acid (1:1, v/v). Avoid over-acidification because it can result in an acidic extract. Repeat extraction procedure on the water sample as described above, collecting extracts in a separate collection vessel and labeling it as acid fraction.

It is critical to remove water from the dichloromethane before you concentrate the extract to final volume. Dichloromethane can hold approximately 11mL of water per liter of dichloromethane. If this water remains in the extract, it will partition out when the volume is reduced. This will result in the dichloromethane boiling off first, leaving water in the collection vessel, and the formation of a two-layer extract. The analyte recoveries will be



Table I. Semivolatile organic compounds listed in US EPA Methods 8270, 525, and 625.

<i>Peak Number</i>	<i>Compound</i>	<i>Peak Number</i>	<i>Compound</i>
1	2-fluorophenol	50	fluorene
2	phenol-d6	51	4-chlorophenyl phenyl ether
3	phenol	52	4-nitroaniline
4	bis(2-chloroethyl) ether	53	4,6-dinitro-2-methylphenol
5	2-chlorophenol-d4	54	N-nitrosodiphenylamine
6	2-chlorophenol	55	2,4,6-tribromophenol
7	1,3-dichlorobenzene	56	4-bromophenyl phenyl ether
8	1,4-dichlorobenzene-d4 (ISTD)	57	α -HCH
9	1,4-dichlorobenzene	58	hexachlorobenzene
10	benzyl alcohol	59	β -HCH
11	1,2-dichlorobenzene-d4	60	pentachlorophenol
12	1,2-dichlorobenzene	61	γ -HCH (Lindane)
13	2-methylphenol (o-cresol)	62	phenanthrene-d10 (ISTD)
14	2,2'-oxybis-(1-chloropropane)	63	phenanthrene
15	4-methylphenol (p-cresol)	64	anthracene
16	N-nitrosodi- <i>n</i> -propylamine	65	δ -HCH
17	hexachloroethane	66	carbazole
18	nitrobenzene-d5	67	heptachlor
19	nitrobenzene	68	di- <i>n</i> -butyl phthalate
20	isophorone	69	aldrin
21	2-nitrophenol	70	heptachlor epoxide
22	2,4-dimethylphenol	71	fluoranthene
23	bis(2-chloroethoxy)methane	72	endosulfan I
24	benzoic acid	73	pyrene
25	2,4-dichlorophenol	74	4,4'-DDE
26	1,2,4-trichlorobenzene	75	<i>p</i> -terphenyl-d14
27	naphthalene-d8 (ISTD)	76	dieldrin
28	naphthalene	77	endrin
29	4-chloroaniline	78	endosulfan II
30	hexachlorobutadiene	79	4,4'-DDD
31	4-chloro-3-methylphenol	80	endrin aldehyde
32	2-methylnaphthalene	81	butyl benzyl phthalate
33	hexachlorocyclopentadiene	82	4,4'-DDT
34	2,4,6-trichlorophenol	83	endosulfan sulfate
35	2,4,5-trichlorophenol	84	endrin ketone
36	2-fluorobiphenyl	85	methoxychlor
37	2-chloronaphthalene	86	3,3'-dichlorobenzidine
38	2-nitroaniline	87	benzo(a)anthracene
39	dimethyl phthalate	88	chrysene-d12 (ISTD)
40	2,6-dinitrotoluene	89	chrysene
41	acenaphthylene	90	bis(2-ethylhexyl)phthalate
42	3-nitroaniline	91	di- <i>n</i> -octyl phthalate
43	acenaphthene-d10 (ISTD)	92	benzo(b)fluoranthene
44	acenaphthene	93	benzo(k)fluoranthene
45	2,4-dinitrophenol	94	benzo(a)pyrene
46	4-nitrophenol	95	perylene-d12 (ISTD)
47	dibenzofuran	96	ideno(1,2,3-cd)pyrene
48	2,4-dinitrotoluene	97	dibenz(a,h)anthracene
49	diethyl phthalate	98	benzo(g,h,i)perylene

The optimum way to remove the water is to decant the dichloromethane through granular sodium sulfate, which is held in a funnel with a high-quality grade filter paper (e.g., Whatman® 541). Approximately 30g of sodium sulfate are sufficient for most samples. This step must not be skipped. Methods may call for powdered sodium sulfate, but some analytes can be adsorbed onto the smaller particles so it is recommended that only 10-60 mesh or similar granular sodium sulfate be used. Also, it is important that this material be contaminant-free, so it should be purchased as American Chemical Society (ACS) pesticide residue-grade in glass containers or baked in a muffle furnace if purchased in bulk packages where exposure to plastic is an issue (see Restek Tip). If a muffle furnace is not available, the sodium sulfate can be washed or extracted with dichloromethane prior to use; however this technique uses large amounts of solvent.

**Restek
Tip**

How to Bake Sodium Sulfate

To bake the sodium sulfate, spread it into a glass pie plate no more than 1" thick and place into a muffle furnace at 400°C for a minimum of two hours. After this time, the sodium sulfate should be placed into a glass container while still hot and sealed with a Teflon®-lined cap to prevent the material from readsorbing contaminants from the atmosphere.



Restek Tip

Clean Glassware

It is important to properly clean glassware used during sample extraction. Contaminated glass surfaces can react with samples and cause breakdown or adsorption of active compounds. Verify cleanliness by running blanks through all glassware.

Automated liquid-liquid extraction can run unattended once the samples are ready and the solvent is added. This extraction is performed at a single pH. Generally, you will need to adjust the sample pH to 2, but some methods call for adjusting the pH to 4. In any event, it is critical to not let the pH go below 2 when using a liquid-liquid extractor. If this happens, an acidic extract will form and may cause damage to the GC column. Acidic extracts also will cause low recoveries for the late-eluting internal standards, possibly due to isotope exchange (e.g., perylene-d12).

Automated liquid-liquid extractors are available in two versions—conventional and accelerated. The conventional types use 1L of sample and extract using 100 to 500mL of dichloromethane. These extraction vessels typically are operated for 16 to 24 hours in order to achieve complete extraction. The accelerated extractor uses a hydrophobic membrane to separate the aqueous from the organic phases, and the extraction time can be reduced by 25 to 30% compared to the conventional extractor. However, the membranes are expensive, so it is important to analyze the cost versus the number of samples extracted to determine if there is a cost benefit to using this accelerated technique.

Finally, **solid phase extraction (SPE)** also may be used to extract semivolatile organic compounds from aqueous samples. When using SPE, it is extremely important to follow the manufacturer's recommendations for product use. There are several manufacturers of C18 cartridges and disks, which are the typical media for these compounds. The specific steps to extract these compounds will vary somewhat depending on the manufacturer. One of the biggest problems with SPE is plugging of the disk or tube with suspended solids, so this method only works reliably for drinking water samples. If contamination levels are low and the samples are free of solids, SPE provides very fast extraction times and low solvent usage. It is used easily for field extractions. And, generally, the disks are preferred for the extraction of 1L sample volumes, but recoveries are not uniform for all of the compounds in Table I. The compounds listed in US EPA Method 525.2 exhibit good extraction recoveries using this technique. For detailed information on this extraction, request the Applications Note "*SPE Extraction for US EPA Method 525.1*" (lit. cat.# 59557).

Soil Samples

Soxhlet and ultrasonic extraction are the most common extraction techniques for solid samples; although pressurized fluid, microwave, and supercritical fluid extraction (SFE) also can be used.

Because the soil and biota samples essentially are wet particles, acetone and dichloromethane (1:1) usually are used as the extraction solvents. Acetone is needed to adequately penetrate the soil particle so that compounds in the particle can be extracted. Several other solvent systems are used for more specialized extractions, but for most applications this combination works well.

All solvents used for extractions must be ACS pesticide-residue grade, and a solvent assay should be performed to verify purity prior to use. To perform a solvent assay, evaporate 300 to 400mL of solvent to a final volume of 1mL and analyze by GC/mass spectrometry (MS). The resulting chromatogram should have no compounds quantitated above $1/2$ the detection limit for any target compound.

Soxhlet and ultrasonic extraction work well for the semivolatile compounds listed in Table I. Sonication is a faster technique but requires constant operator attention. In both techniques, problems usually are caused by contaminated reagents (especially sodium sulfate) or by inconsistent handling from sample to sample. Sodium sulfate must be treated to remove water as described in the Restek Tip on page 3, and the sample must be mixed with the sodium sulfate to achieve a sandy consistency.

Pressurized fluid extraction (US EPA Method 3545A) can be run in an unattended fashion with multiple samples across a wide sample size range. Extraction vessels with volumes of 1 to 100 mL are available. Instruments like the Dionex ASE 200 accommodate wet samples from 1 to 15 grams, and the Dionex ASE 300 will accommodate wet samples from 15 to 50 grams. The volume of the cell needed for wet samples is generally twice the gram weight of the sample being used. For example, if 30-g wet samples are needed, the 66-mL and 100-mL vessels will be adequate for these extractions. This is necessary because a drying agent such

as diatomaceous earth is added to the sample prior to being loaded into the extraction vessels. The type of samples being extracted as well as the required method detection limits should be considered as part of the evaluation of pressurized fluid extraction.

Microwave extraction (US EPA Method 3546) can be useful for automated extraction as well. This method typically performs the extraction of 12 samples simultaneously, but requires slightly more operator handling than the pressurized fluid extraction instruments. Microwave extraction instrumentation is less expensive, but can suffer from the same sample size limitations.

Supercritical fluid extraction (SFE) has been promoted for a number of years as a means of “solventless” extraction for environmental samples. SFE has been added to SW-846 as Methods 3560, 3561, and 3562 but its application is limited. SFE suffers from severe matrix-related variation, requiring modification of its conditions depending on soil type, water content, sample size, and type of analytes. Doing so ultimately requires additional sample preparation prior to the actual extraction. These requirements, added to the high cost of the instrument, have virtually precluded the use of SFE for environmental sample preparation.

Cleanup

Sample extract cleanup may be the most important step in maintaining long-term instrument performance. Many times, when instrument problems arise, they are caused by exposure of the injection port and the column to material in the sample extracts other than the target compounds. While all contaminants cannot be eliminated, reducing them will minimize injection port and column maintenance. Most semivolatile extracts, especially those extracts from soil and biota samples, contain high-boiling hydrocarbons and lipids. The difficulty in attempting to remove these compounds using one of the common solid-liquid cleanup techniques (e.g., Florisil® and silica gel) is that the cleanup technique also removes some of the target compounds. In addition, because the analytical method usually calls for the reporting of several tentatively identified compounds (TICs), it is not desirable to clean the extracts of compounds that would normally elute in the range of the target compounds. For these reasons, gel permeation chromatography (GPC) is the only universal cleanup technique for semivolatile extracts.

Gel Permeation Chromatography

Gel permeation chromatography (GPC) is a preparative scale chromatographic method of separation based on molecular size. Because the target compounds are similar in molecular size, they elute as a band of material and are easily separated from lighter and heavier contaminants. However, GPC systems are expensive and the processing time per sample is between 30 to 70 minutes. For these reasons many laboratories choose not to use GPC. However, it is very efficient for removing sulfur, high molecular weight hydrocarbons, and lipids from semivolatile extracts; and may be prudent for soil and biota samples.

Although sulfur can be removed using other techniques such as mercury or activated copper powder, these procedures, especially copper powder, may degrade some of the target compounds and will not remove the high-boiling hydrocarbons or lipids. The lipid content of biota extracts can be significant and may overload most SPE clean-up techniques. If a sample extract with a high lipid content is injected into the GC, the injection port and front of the column will become contaminated quickly. This will result in failure of check standards and the loss of active compounds such as nitroanilines, nitrophenols, carbazole, and pentachlorophenol (PCP). In spite of the added expense and time required for GPC, it is the best alternative for extract cleanup.

US EPA Method 3640 details the requirements for GPC cleanup of extracts for semivolatile analysis. One of the important steps of GPC cleanup is to ensure each day that the instrument is within its retention time calibration. Although not required by the method, it is good practice to run a daily calibration check standard before processing the next batch of samples. If a number of samples have been processed that contain large amounts of contamination, the front of the GPC column can become reactive. This typically is observed in the loss of 2,4,6-tribromophenol for semivolatile extracts. If the column becomes reactive, injecting blanks may return the system to control and save the time required to change the column.

**Restek
Tip**

Stabilizing Dichloromethane

Dichloromethane requires a stabilizer to prevent the formation of hydrochloric acid (HCl). Without a stabilizer, HCl will form and injection of acidic dichloromethane will cause inlet liners and columns to become reactive. There are two types of stabilizers: stabilizers that keep HCl from forming, and stabilizers that eliminate HCl upon formation. Methanol is a stabilizer that prevents HCl from forming; whereas cyclohexane, cyclohexene, 2-methylbutene, and amylene scavenge the HCl after its formation.

Dichloromethane used in liquid extractors should contain both types of stabilizers. Methanol is a better stabilizer, acting as a free radical inhibitor, but methanol partitions into the water phase. This could leave an unstabilized extract unless a scavenger stabilizer also is used.

Obtaining consistent GPC results begins with the extraction and concentration procedures because slight changes in mobile phase and sample solvent composition can result in some target compounds being uncollected. Because the typical sample solvent for GPC is pure dichloromethane, it is critical that all extracts be reduced to as small a volume as possible before reconstitution in dichloromethane to avoid large amounts of acetone being applied to the column. Soil and biota samples typically are extracted with a solvent mixture of acetone and dichloromethane. It is critical that all extracts be reduced to as small a volume as possible before reconstitution in dichloromethane to avoid large amounts of acetone being applied to the column. Dichloromethane has a lower boiling point than acetone, so it will evaporate first during sample concentration, which will leave nearly 100% acetone in the concentration vessel. If dichloromethane is then added to adjust the extract to volume, there will be significant amounts of acetone introduced to the GPC column. This will lead to “solvent shock” and the formation of a void will be observed at the front of the column. This, in turn, will affect the retention times of the compounds eluting from the GPC column and ultimately will result in some target compounds being uncollected. Table II lists the common semivolatile compound elution volumes using GPC.

Analysis

Calibration Standards

Calibration standards are purchased as mixtures and usually are divided among three to seven separate ampuls due to the cross-reactivity of several compounds. It is important when making the actual working standard that the solution be stored under refrigerated conditions in a Mininert™ vial (Restek cat.# 21050 and 21051) due to the volatility of some of the compounds. Failure to properly store the calibration standards will result in evaporative loss of the early-eluting compounds and the solvent. This will, in effect, concentrate the late-eluting compounds and cause continuing calibration failure and quantitation errors. Even when stored under the correct conditions, there still will be degradation of some compounds due to cross-reactivity. This is observed as a loss of the target compound and commonly occurs with benzidine, 3,3'-dichlorobenzidine, 4-chloroaniline, N-nitrosodiphenylamine, and to a lesser extent with the phenols and other anilines. These standards are stable in the separate ampuls supplied from the manufacturer, but problems arise when all of the compounds are mixed together to make the working calibration standard. Therefore, it is important to monitor the response of the more active compounds and make fresh mixtures when the calibration standards degrade.

Restek Tip

Mixing Calibration Standards

When blending several ampuls to produce a calibration standard, it is important that all the compounds are completely dissolved in the solvent. This is particularly important with some of the high molecular weight polycyclic aromatic hydrocarbons (PAHs) and pesticides that can separate from solution during refrigerated storage. Before opening ampuls containing semivolatile compounds, allow them to warm to room temperature. Some mixtures may require sonication to ensure complete solubility. Follow the manufacturer's recommendations for proper handling of the standard mixture. Because some semivolatile compounds are light sensitive, it is recommended that calibration standards be stored in amber vials.

Injection Port Configuration

Several of the compounds listed in Table I are prone to breakdown or adsorption on active surfaces. Typically this will occur in the injection port; therefore, careful attention must be given to the configuration and maintenance of the injection system.

On-column injection techniques can eliminate breakdown or adsorption in the injection system and improve chromatographic analysis for drinking water extracts or extracts with little or no non-volatile residues. However, we do not recommend on-column injections for soil and biota extracts or extracts that contain large amounts of non-volatile residue, because the analytical column can be contaminated quickly.

The preferred injection technique for analyzing highly contaminated extracts is **direct injection**, but direct injection can cause solvent peak tailing and result in some of the target compounds eluting close to the solvent peak.

To reduce solvent peak tailing, **splitless injection** is most commonly used for GC/MS analysis of semivolatile compounds. There are some drawbacks to splitless injection including molecular weight discrimination, incomplete sample transfer, and reactivity. These problems can be minimized if the technique is properly optimized. Splitless injection requires an injection system that is equipped with a solenoid valve controlling the flow to a split vent. The solenoid valve is closed during the injection process, so the majority of the vaporized sample moves to the front of

Table II. Mobile phase volumes for elution of semivolatile compounds by GPC.

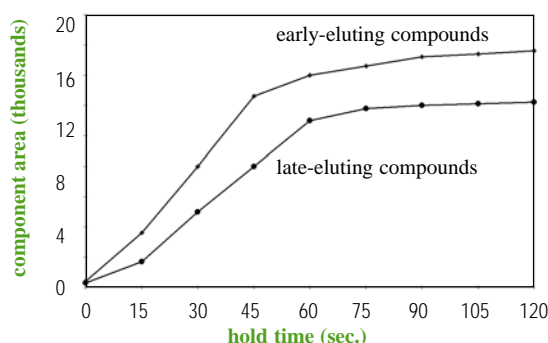
Compound	Elution Volumes (mL)		Compound	Elution Volumes (mL)	
	Start	End		Start	End
2-fluorophenol	201	241	acenaphthene-d10	225	249
phenol-d5	201	249	3-nitroaniline	209	225
phenol	209	225	acenaphthene	225	249
bis(2-chloroethyl)ether	201	225	2,4-dinitrophenol	201	225
2-chlorophenol-d4	209	249	dibenzofuran	225	249
2-chlorophenol	209	241	4-nitrophenol	201	217
1,3-dichlorobenzene	225	257	2,4-dinitrotoluene	201	225
1,4-dichlorobenzene-d4	225	257	diethylphthalate	186	209
1,4-dichlorobenzene	225	257	fluorene	225	241
1,2,-dichlorobenzene-d4	225	257	4-chlorophenyl-phenylether	217	241
1,2-dichlorobenzene	225	257	4-nitroaniline	201	225
2-methylphenol	201	241	4,6-dinitro-2-methylphenol	201	225
2,2'-oxybis(1-chloropropane)	201	225	N-nitrosodiphenylamine	201	233
4-methylphenol	201	241	2,4,6-tribromophenol	217	249
N-nitroso-di-n-propylamine	186	217	4-bromophenyl-phenylether	217	241
hexachloroethane	233	257	hexachlorobenzene	233	257
nitrobenzene-d5	209	233	pentachlorophenol	209	249
nitrobenzene	209	233	phenanthrene-d10	225	257
isophorone	193	217	phenanthrene	225	257
2-nitrophenol	217	233	anthracene	225	257
2,4-dimethylphenol	201	225	carbazole	225	257
bis(2-chloroethoxy)methane	193	225	di-n-butylphthalate	178	201
2,4-dichlorophenol	201	241	fluoranthene	225	257
1,2,4-trichlorobenzene	225	257	pyrene	225	257
naphthalene-d8	225	249	terphenyl-d14	217	233
naphthalene	225	249	butylbenzylphthalate	178	201
4-chloroaniline	217	241	benzo(a)anthracene	225	257
hexachlorobutadiene	225	249	3,3'-dichlorobenzidine	209	241
4-chloro-3-methylphenol	201	225	chrysene-d12	225	257
2-methylnaphthalene	225	249	chrysene	225	257
hexachlorocyclopentadiene	225	241	bis(2-ethylhexyl)phthalate	162	186
2,4,6-trichlorophenol	201	249	di-n-octylphthalate	162	186
2,4,5-trichlorophenol	201	249	benzo(b)fluoranthene	225	265
2-fluorobiphenyl	217	241	benzo(k)fluoranthene	225	265
2-chloronaphthalene	233	249	benzo(a)pyrene	225	265
2-nitroaniline	209	233	perylene-d12	249	273
dimethylphthalate	193	217	indeno(1,2,3-cd)pyrene	241	273
acenaphthylene	225	257	dibenz(a,h)anthracene	225	257
2,6-dinitrotoluene	193	225	benzo(g,h,i)perylene	233	273

Standard prepared and loaded as 1/4 acetone, dichloromethane; 5mL sample loop;
Column: 70g SX-3 silica size exclusion packing; **Guard column:** 5g same packing;
Flow rate: dichloromethane at 5.3mL/min. at 13 psi; Samples analyzed by GC/MS.
 (US EPA 8270)

the column. After a short time the solenoid valve is opened to allow excess solvent vapor to exit the split vent. The process of transferring the sample onto the column is relatively slow during splitless injection, so the sample must recondense at the front of the column through solvent or analyte focusing. This is accomplished by having the starting oven temperature 20°C lower than the boiling point of the solvent or the first eluting compound.

The time period that the solenoid valve is closed is referred to as the splitless hold-time. The hold-time must be optimized to obtain the best performance from the analytical system. If the solenoid valve is opened too quickly, some of the sample will be lost causing reduced response. If the solenoid valve is open too long, the solvent peak will tail. The splitless hold-time will vary depending on column flow rate, injection port geometry, injection port temperature, and volatility of the analytes. It is impossible to predict the optimum splitless hold-time without performing some experimentation under the exact conditions of your analysis.

Figure 1. Optimizing splitless hold time.



To optimize the splitless hold time for a particular instrument, prepare a standard that contains both an early- and a late-eluting compound (e.g., fluorophenol and benzo(g,h,i)perylene). Inject this standard over a range of splitless hold times from 0.1 to 2.0 minutes and plot the data. An example of this optimization is shown in Figure 1.

In this example the optimum splitless hold time is 60 seconds. This is the point on the graph where the response of the late-eluting compound levels off. Holding the solenoid valve closed longer will not appreciably increase the response of this compound, but will greatly increase the size of the solvent peak. Because the lower boiling compound will transfer onto the column faster, its response will level off sooner (in this example ~45 seconds). Once this data has been plotted, it is possible to observe the correct splitless hold-time (once again, the point at which the response of the late-eluting compounds levels off). The net effect of this optimization is to maximize response of late-eluting compounds while minimizing solvent tailing.

In addition to optimizing the splitless hold-time, fused-silica wool should be used in the injection port liner to improve vaporization of higher molecular weight compounds. While there are different theories regarding the placement of fused-silica wool, consistency in the amount of packing and location of the packing is most important. Restek recommends placing the plug of wool below the point that the syringe needle reaches, but above the inlet of the column. We also recommend using a gooseneck liner to minimize contact between the injected sample and the bottom of the injection port. This will help improve the response of the more reactive compounds such as 2,4-dinitrophenol, PCP, and the nitroanilines. The gooseneck liner also makes the greatest improvement in response and minimization of endrin breakdown for US EPA Method 525.

Another technique to minimize molecular weight discrimination is to perform the splitless injection under a higher column head pressure. A high inlet pressure is advantageous during injection to control the rapidly expanding vapor cloud in the inlet. By using a momentary pressure pulse for the time that the split vent line is closed, the sample vapor cloud is controlled and sample backflash into the gas lines entering and exiting the injection port is minimized. The effect of the pressure pulse is to increase the amount of analyte transferred to the column, especially the late-eluting components. This can lead to stationary phase overload, however, so it may be necessary to increase the capacity of the column when using

Any injection technique can suffer from reactivity (i.e., breakdown) and splitless injection is no exception. The splitless technique has two primary mechanisms for compound reactivity: sample backflash into the gas lines that enter and exit from the injector; and exposure of the sample extract to active sites on the wool, liner, and tip of the column. In general, the same set of compounds break down regardless of which mechanism is occurring: 2,4-dinitrophenol, PCP, 4-nitrophenol, carbazole, and 3-nitroaniline.

Daily maintenance of the injection port will help decrease this problem. Replace the inlet liner and fused silica wool plug, and the septum every day. Weekly, or more often depending on the extract contamination level, replace the inlet seal and remove a short section from the front of the column. The length of column removed will vary depending on the level of contamination in the extracts, generally 6 to 12 inches is adequate. When cutting the column and re-installing it into the injection port, be sure to make a square cut and be consistent with the installation distance. The installation distance varies by manufacturer. Refer to Table III for a list of recommended insertion distances.

Table III. Recommended installation distances.

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

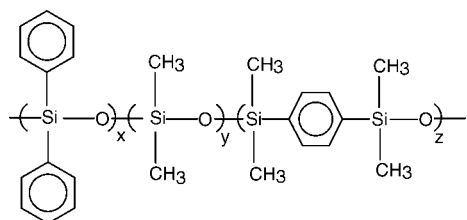
Column Selection

Due to the wide variation in functionality, volatility, and polarity of semivolatile compounds, it is not possible to select a column that is highly selective for all of them. As a result, this analysis is performed on a general-purpose stationary phase. The Rtx®-5Sil MS column has the best combination of low bleed, high inertness, and efficiency for semivolatile applications. (The Rtx®-5MS column also has been successfully used for analysis of semivolatile compounds.) The Rtx®-5Sil MS column features a silarylene phenyl/methyl phase that was developed to provide lower bleed and greater efficiency than other “5-type” phases for improved separation of the PAHs (Figure 2).

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

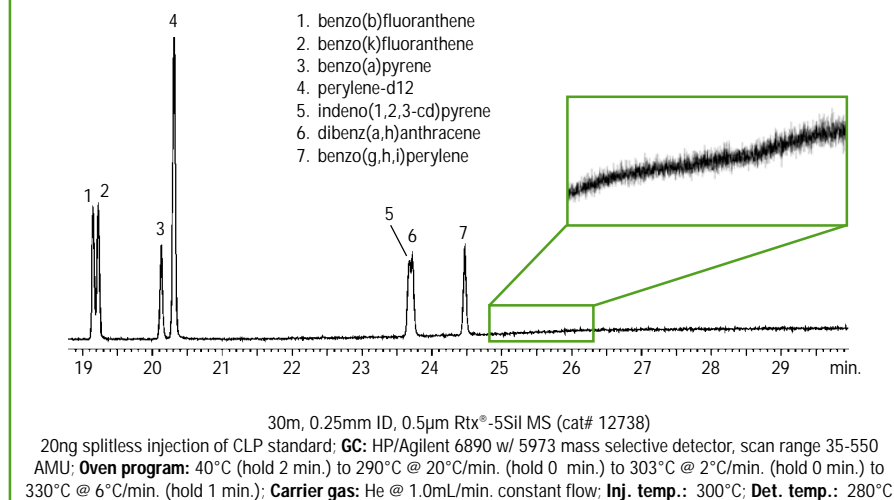
signal on the detector and can cause electron multiplier saturation. If this occurs, calibration curves may show non-linearity at higher concentrations. This is sometimes referred to as high-end roll-off, when the signal for a given concentration is lower than expected due to detector saturation. (See *Quantitation* section for more information on high-end roll-off.)

Figure 2. The Rtx®-5Sil MS column structure.



To diagnose column bleed problems, make an injection that allows the bleed to be measured relative to the concentration of the analyte in the method. Many data systems will normalize the display to the largest peak in the analysis. If no compounds are injected, the display will falsely indicate a high background. The Rtx®-5Sil MS column shows a minimal bleed level for a 20ng per component standard (Figure 3).

Figure 3. The Rtx®-5Sil MS column exhibits low bleed at 20ng concentration level.



Detector saturation also can be caused by the concentration of the analytes. It was common practice on older, less sensitive GC/MS systems to increase the multiplier voltage above the tune value to improve sensitivity of low-concentration standards. This technique can lead to problems with the newer, more sensitive instruments. It is much more likely the higher concentration calibration standards will saturate the new GC/MS systems. It may be necessary to reduce the multiplier voltage below the tune value if high-end roll-off is observed. High-end roll-off also may be observed when using pressure-pulsing injection techniques to minimize high molecular weight discrimination. If this is observed, you may either increase the stationary phase film thickness, or increase the column diameter. Alternatively, you may modify the injection conditions to eliminate the source of the overload.

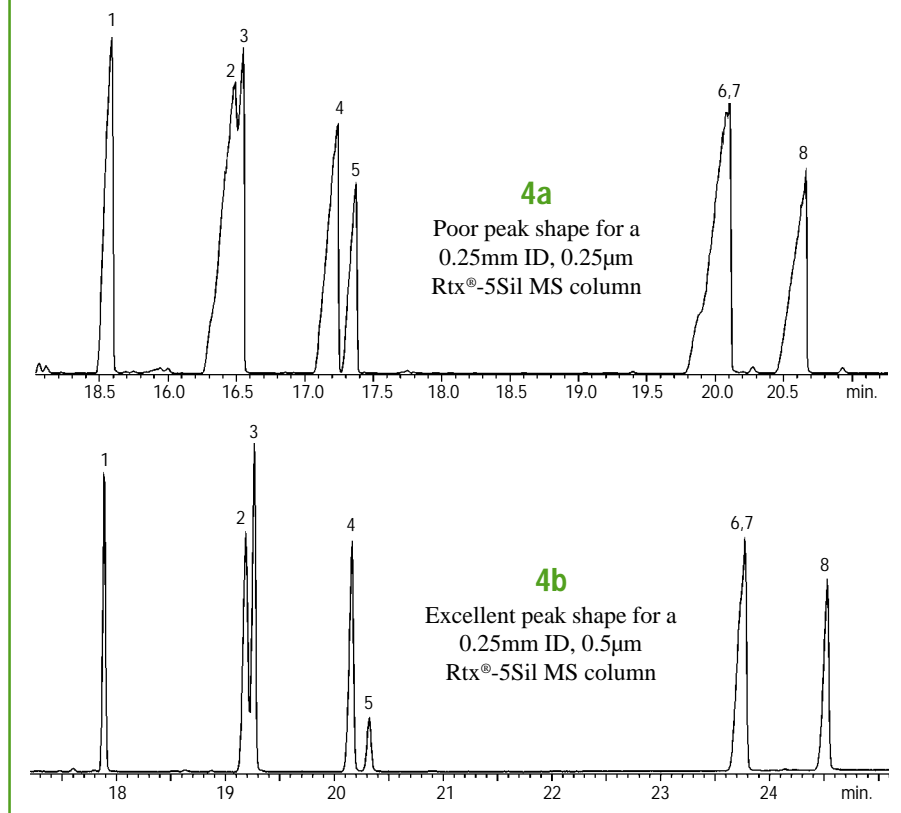
Column capacity also must be addressed when optimizing the analysis. The typical calibration range for many of these methods is 20 to 160ng per compound. This requires a column stationary phase and diameter that will not overload with a 160ng or larger injection. Because there is a loss of analyte in any splitless injection, calculation of the necessary column capacity is not simple. If the injection has been optimized for splitless hold-time and fused silica wool is being used in the liner to minimize high molecular weight discrimination, then it is easier to overload the analytical column. Possibly the biggest cause of overload is from pressure-pulsing the injection port, as this improves the transfer of all compounds to the column. The required capacity for your system will be a function of the specific calibration standards and, more importantly, the injection port.

From a capacity consideration, a 0.25mm ID column with 0.25µm film thickness does not have sufficient capacity for a 160ng per component standard. Figure 4a shows the poor peak shape observed when a column is overloaded. Increased capacity can be achieved by increasing column diameter or film thickness.

When increasing column diameter, the flow rate of the column can be a concern with bench-top GC/MS systems. Many bench-top GC/MS systems do not have the pumping capacity for the carrier gas flow that is needed with a 0.32mm ID column. A 0.28mm ID column can increase sample capacity without exceeding the pumping capacity of most bench-top GC/MS systems, making it ideal for calibrating semivolatile compounds from 20 to 160ng without overload. Alternatively, a 0.25mm ID column with a 0.5µm film thickness also has sufficient capacity to handle a calibration from 20 to 160ng without exhibiting overload. Figure 4b shows excellent peak shape for a 160ng-per-component standard on a 30m, 0.25mm ID, 0.5µm Rtx®-5Sil MS column.

The total **analysis time** should be as short as possible without sacrificing separation or resolution between compounds with similar mass spectra. Pay particular attention to the separation between benzo-b- and benzo-k-fluoranthrene—they tend to be the most difficult-

Figure 4a & 4b. Avoid overload by selecting a column with the proper capacity.



Peak List for Fig. 4a & 4b

1. di-*n*-octyl phthalate
2. benzo(b)fluoranthene
3. benzo(k)fluoranthene
4. benzo(a)pyrene
5. perylene-d12
6. indeno(1,2,3-cd)pyrene
7. dibenz(a,h)anthracene
8. benzo(ghi)perylene

Conditions for Fig. 4a

30m, 0.25mm ID, 0.5µm Rtx®-5Sil MS (cat.# 12738)
160ng splitless injection of CLP standard; **GC:** HP/Agilent 6890 w/ 5973 mass selective detector, scan range 35-550 AMU; **Oven program:** 40°C(hold 2 min.) to 290°C @ 20°C/min. (hold 0 min.) to 303°C @ 2°C/min. (hold 0 min.) to 330°C @ 6°C/min. (hold 1 min.); **Carrier gas:** He @ 1.0 mL/min. constant flow; **Inj. temp.:** 300°C; **Det. temp.:** 280°C

Conditions for Fig. 4b

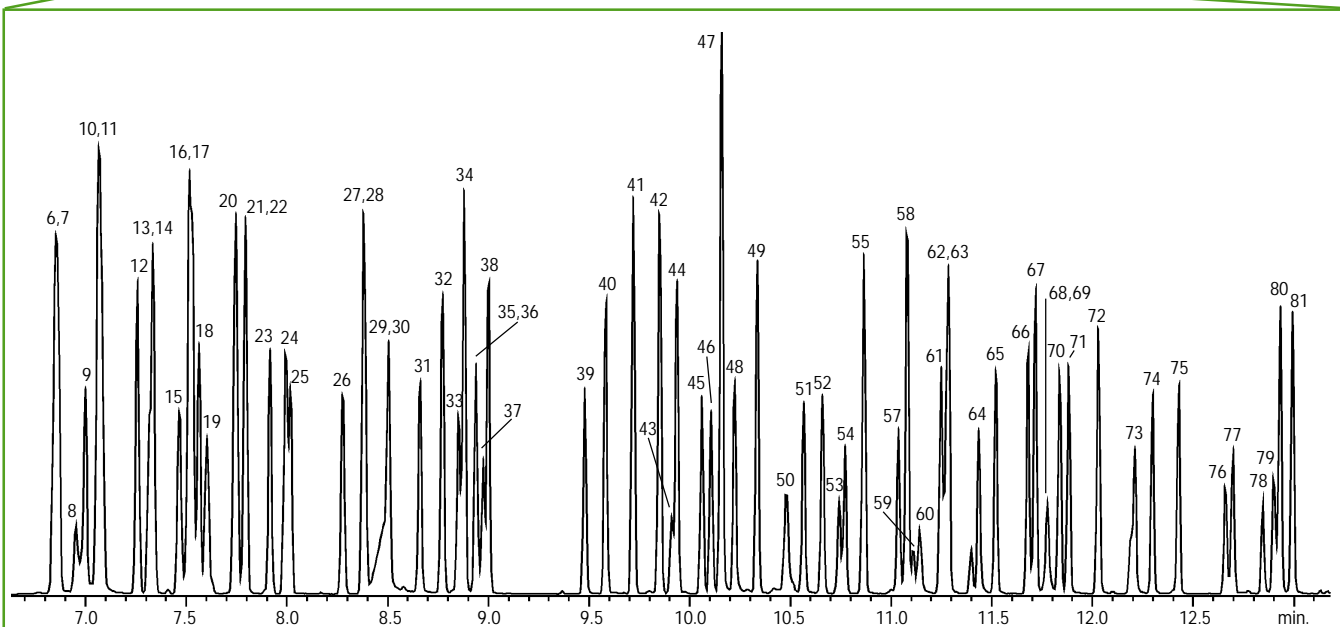
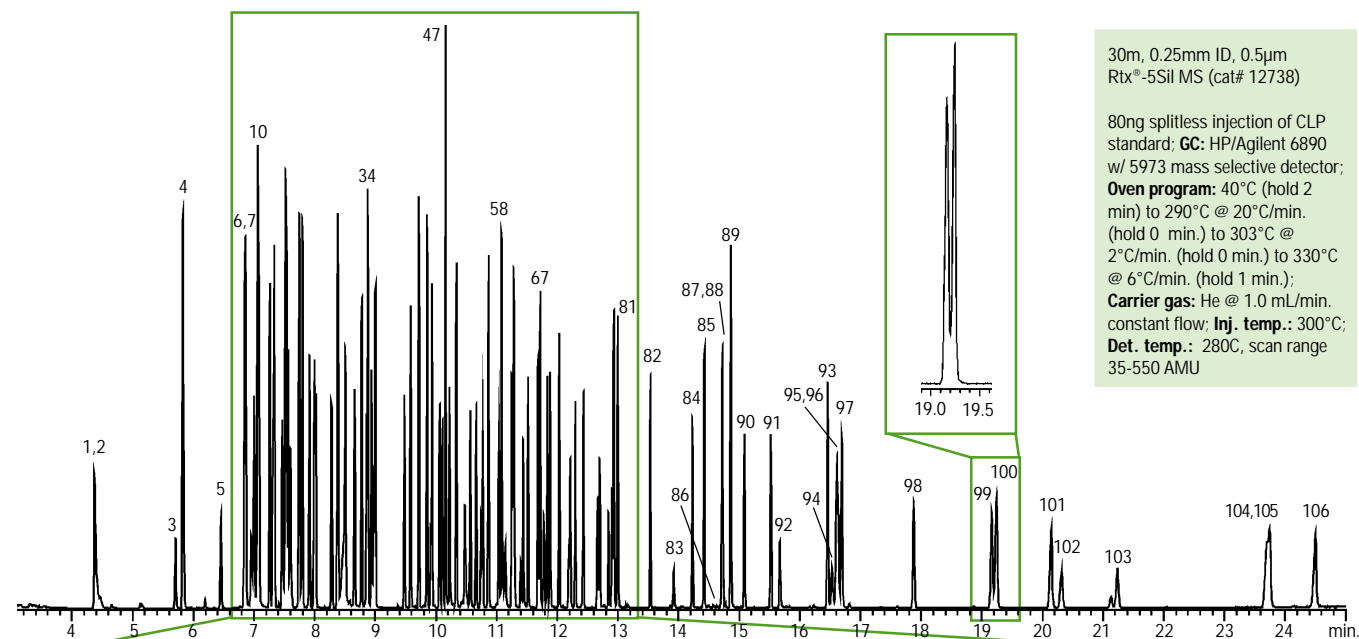
30m, 0.25mm ID, 0.25µm Rtx®-5Sil MS (cat.# 12723)
unknown concentration (>160ng) splitless injection of CLP standard; **GC:** HP/Agilent 6890 w/ 5973 mass selective detector, scan range 35-550 AMU; **Oven program:** 40°C (hold 2 min.) to 245°C @ 25°C/min. (hold 0 min.) to 330°C @ 6°C/min. (hold 5 min.); **Carrier gas:** He @ 1.0 mL/min. constant flow; **Inj. temp.:** 300°C; **Det. temp.:** 280°C

to-separate analytes, sharing common mass spectra and quantitation ions. Figure 5 shows a 80ng per component injection of the compounds listed in Table I with an analysis time under 30 minutes. The expanded sections of the chromatogram show the excellent resolution that can be achieved with the Rtx®-5Sil MS column.

In the past, the GC/MS systems used for semivolatile analysis did not have the sensitivity for split injections, so laboratories were limited to splitless injection. Newer systems such as the Agilent 5973 and ion trap GC/MS have greatly improved sensitivity, which allow the use of **split injection** and still meet the detection limits required by most semivolatile methods. Figure 6 shows the 20ng per component standard injected in split mode using a 20:1 split ratio on a 30m, 0.25mm ID, 0.25µm Rtx®-5Sil MS column. The low bleed exhibited by this column is critical when working with these more sensitive GC/MS systems. A benefit of split injection is narrower peak widths for improved separations between closely eluting compounds. Also, split injections usually result in less reactive compound breakdown because the residence time in the injection port is much shorter than in splitless injection.

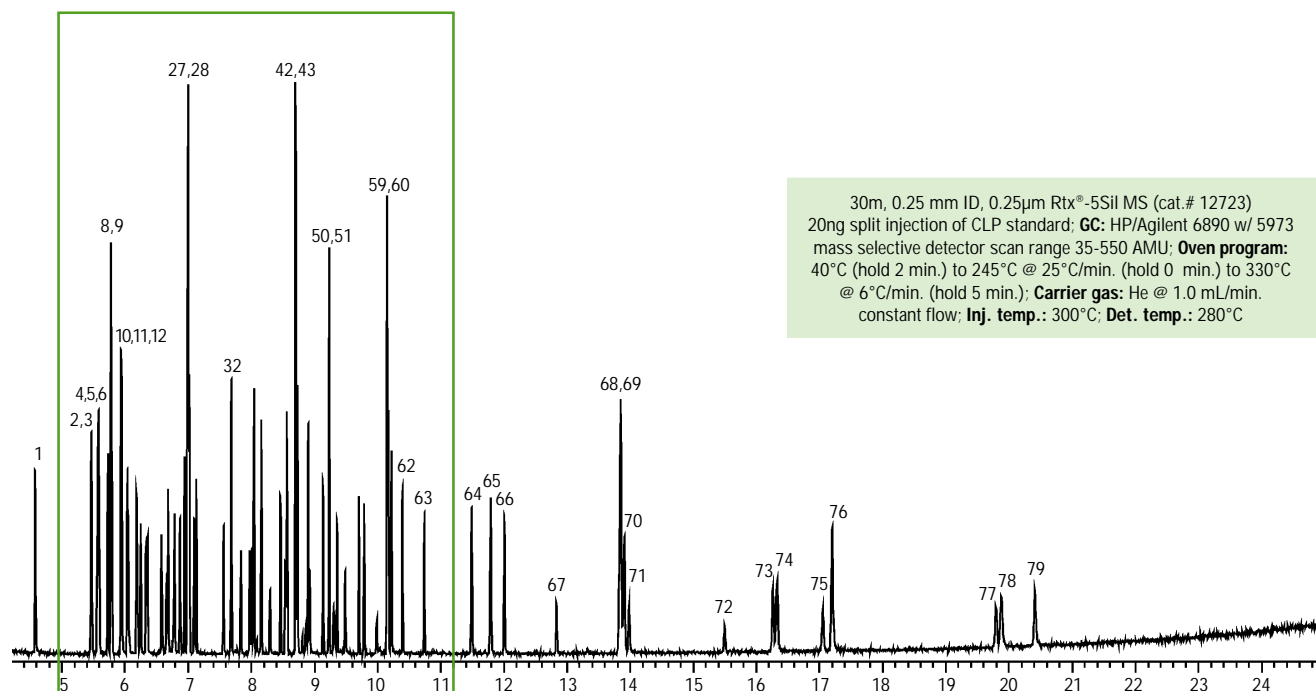
If the sensitivity of an instrument allows for split injection, then column capacity is not nearly the issue it is for splitless injection. Figure 7 shows a 160ng-per-component standard injected under the same conditions as shown in Figure 6. A column with a thinner film can be used because the concentration reaching the column is reduced by 20-fold. The analytical system using split injection will be able to handle higher concentrations of contaminants and possibly stay calibrated longer, but there will be a sacrifice in method detection limits (MDLs). Therefore, it is important to ensure that the MDLs specified in a particular method still can be met if split injection is used.

Figure 5. A 30m, 0.25mm ID, 0.5µm Rtx®-5Sil MS column offers excellent resolution of 106 compounds listed in less than 25 minutes.

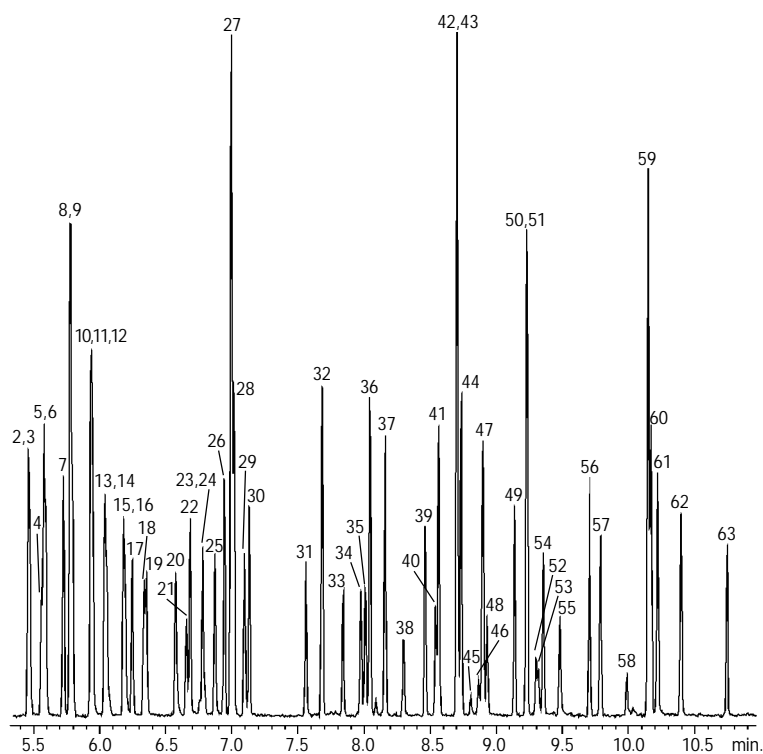


- | | | | | |
|-----------------------------------|--------------------------------|--------------------------------|---------------------------------|--------------------------------|
| 1. N-nitrosodimethylamine | 22. acetophenone | 43. hexachlorocyclopentadiene | 64. 2,3,4,6-tetrachlorophenol | 85. fluoranthene |
| 2. pyridine | 23. hexachloroethane | 44. 1,2,4,5-tetrachlorobenzene | 65. diethyl phthalate | 86. benzidine |
| 3. methyl methanesulfonate | 24. nitrobenzene-d5 | 45. 2,4,6-trichlorophenol | 66. 4-chlorophenyl phenyl ether | 87. pyrene |
| 4. 2-fluorophenol | 25. nitrobenzene | 46. 2,4,5-trichlorophenol | 67. fluorene | 88. aromite |
| 5. ethyl methanesulfonate | 26. isophorone | 47. 2-fluorobiphenyl | 68. 4-nitroaniline | 89. p-terphenyl-d14 |
| 6. phenol-d6 | 27. 2,4-dimethylphenol | 48. safrrole | 69. 4,6-dinitro-2-methylphenol | 90. chlorbenzilate |
| 7. phenol | 28. 2-nitrophenol | 49. 2-chloronaphthalene | 70. diphenylamine | 91. butyl benzyl phthalate |
| 8. aniline | 29. benzoic acid | 50. 2-nitroaniline | 71. azobenzene | 92. kepone |
| 9. bis(2-chloroethyl)ether | 30. bis(2-chloroethoxy)methane | 51. 1,4-naphthoquinone | 72. 2,4,6-tribromophenol | 93. bis(2-ethylhexyl)phthalate |
| 10. 2-chlorophenol | 31. 2,4-dichlorophenol | 52. dimethylphthalate | 73. phenacetin | 94. 3,3'-dichlorobenzidine |
| 11. 2-chlorophenol | 32. 1,2,4-trichlorobenzene | 53. 1,3-dinitrobenzene | 74. 4-bromophenyl-phenyl ether | 95. benzo(a)anthracene |
| 12. 1,3-dichlorobenzene | 33. naphthalene-d8 | 54. 2,6-dinitrotoluene | 75. hexachlorobenzene | 96. chrysene-d12 |
| 13. 1,4-dichlorobenzene-d4 | 34. naphthalene | 55. acenaphthylene | 76. pentachlorophenol | 97. chrysene |
| 14. 1,4-dichlorobenzene | 35. 2,6-dichlorophenol | 56. 3-nitroaniline | 77. pentachloronitrobenzene | 98. di-n-octyl phthalate |
| 15. benzyl alcohol | 36. 4-chloroaniline | 57. acenaphthene-d10 | 78. dinoseb | 99. benzo(b)fluoranthene |
| 16. 1,2-dichlorobenzene-d4 | 37. hexachloropropene | 58. acenaphthene | 79. phenanthrene-d10 | 100. benzo(k)fluoranthene |
| 17. 1,2-dichlorobenzene | 38. hexachlorobutadiene | 59. 2,4-dinitrophenol | 80. phenanthrene | 101. benzo(a)pyrene |
| 18. 2-methylphenol | 39. 4-chloro-3-methylphenol | 60. 4-nitrophenol | 81. anthracene | 102. perylene-d12 |
| 19. bis(2-chloroisopropyl)ether | 40. isosafrole | 61. pentachlorobenzene | 82. di-n-butylphthalate | 103. 3-methylcholanthrene |
| 20. 4-methylphenol/3-methylphenol | 41. 2-methylnaphthalene | 62. 2,4-dinitrotoluene | 83. 4-nitroquinoline-1-oxide | 104. indeno(1,2,3-cd)pyrene |
| 21. N-nitrosodimethylamine | 42. 1-methylnaphthalene | 63. dibenzofuran | 84. isodrin | 105. dibenz(a,h)anthracene |

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



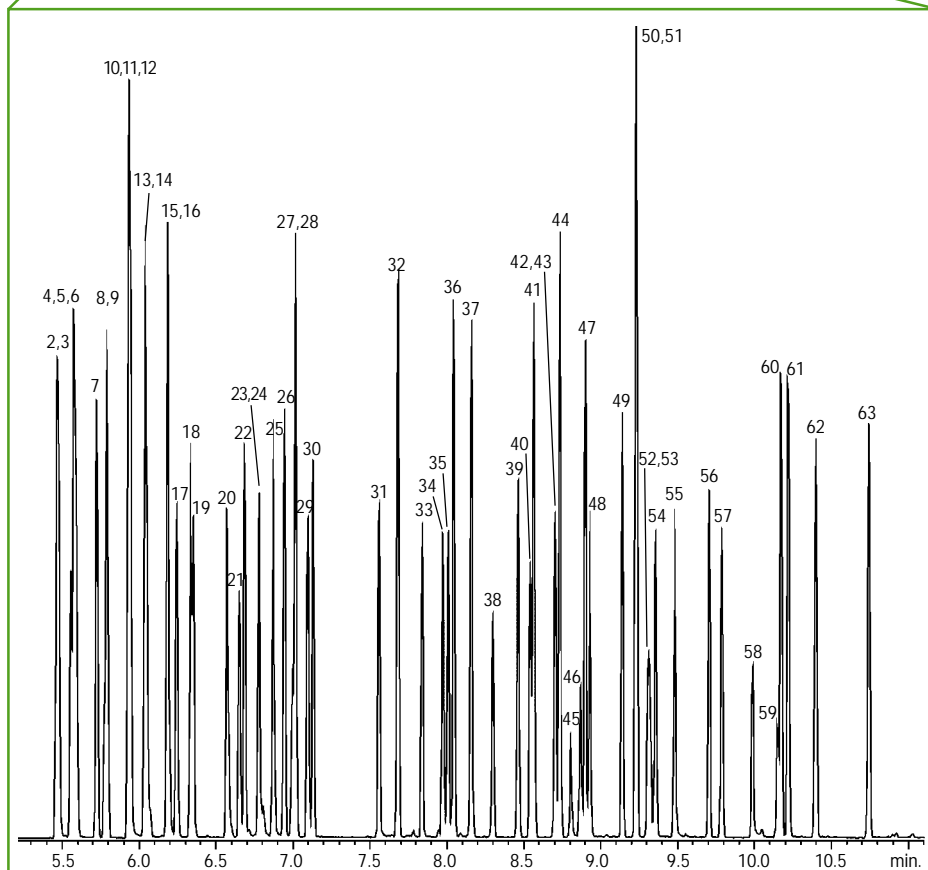
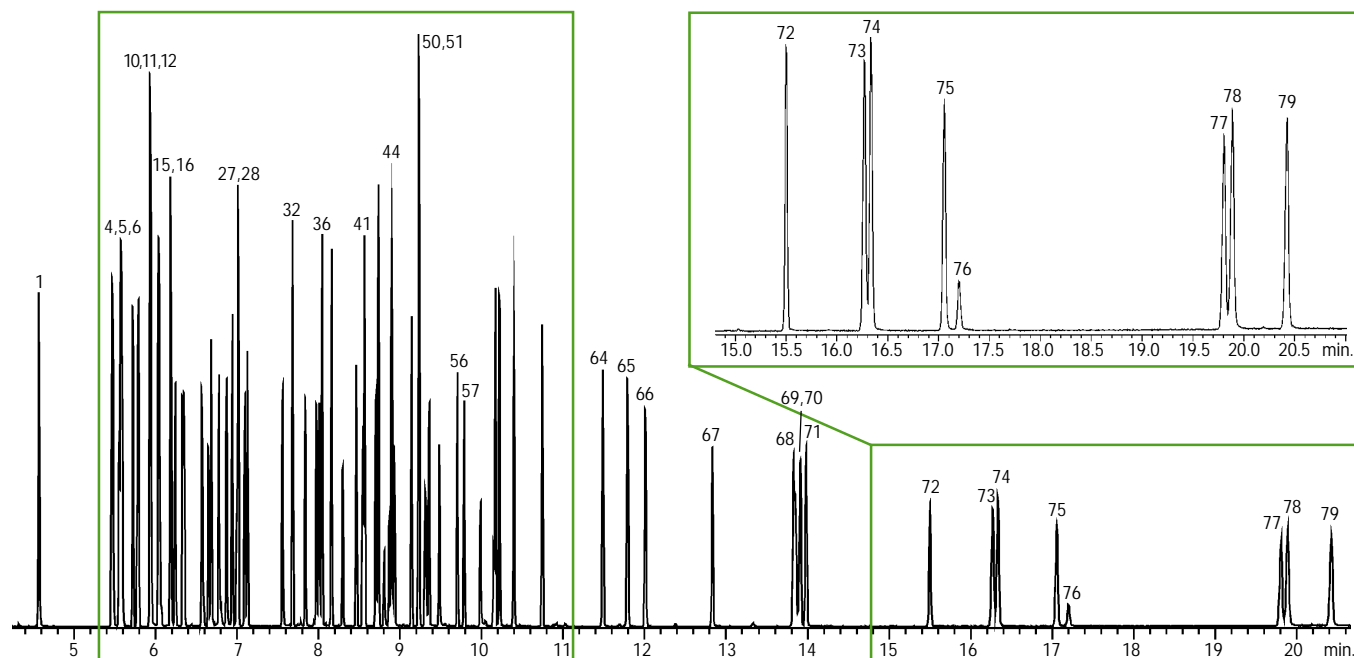
30m, 0.25 mm ID, 0.25µm Rtx®-5Sil MS (cat.# 12723)
20ng split injection of CLP standard; GC: HP/Agilent 6890 w/ 5973
mass selective detector scan range 35-550 AMU; **Oven program:**
40°C (hold 2 min.) to 245°C @ 25°C/min. (hold 0 min.) to 330°C
@ 6°C/min. (hold 5 min.); **Carrier gas:** He @ 1.0 mL/min.
constant flow; **Inj. temp.:** 300°C; **Det. temp.:** 280°C



Peak List for Figures 6 & 7

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

Figure 7. Analyzing semivolatile compounds in the split injection mode can improve peak shape and eliminate column overload.



30m, 0.25mm ID, 0.25µm
Rtx®-5Sil MS (cat.# 12723)

Sample: 160ng split injection of CLP standard;
GC: HP/Agilent 6890 w/ 5973 mass selective
detector, scan range 35-550 AMU; **Oven program:**
40°C(hold 2 min.) to 245°C @ 25°C/min. (hold 0
min.) to 330°C @ 6°C/min. (hold 5 min.); **Carrier**
gas: He @ 1.0mL/min. constant flow; **Inj. temp.:**
300°C; **Det. temp.:** 280°C

Peak list on page 13

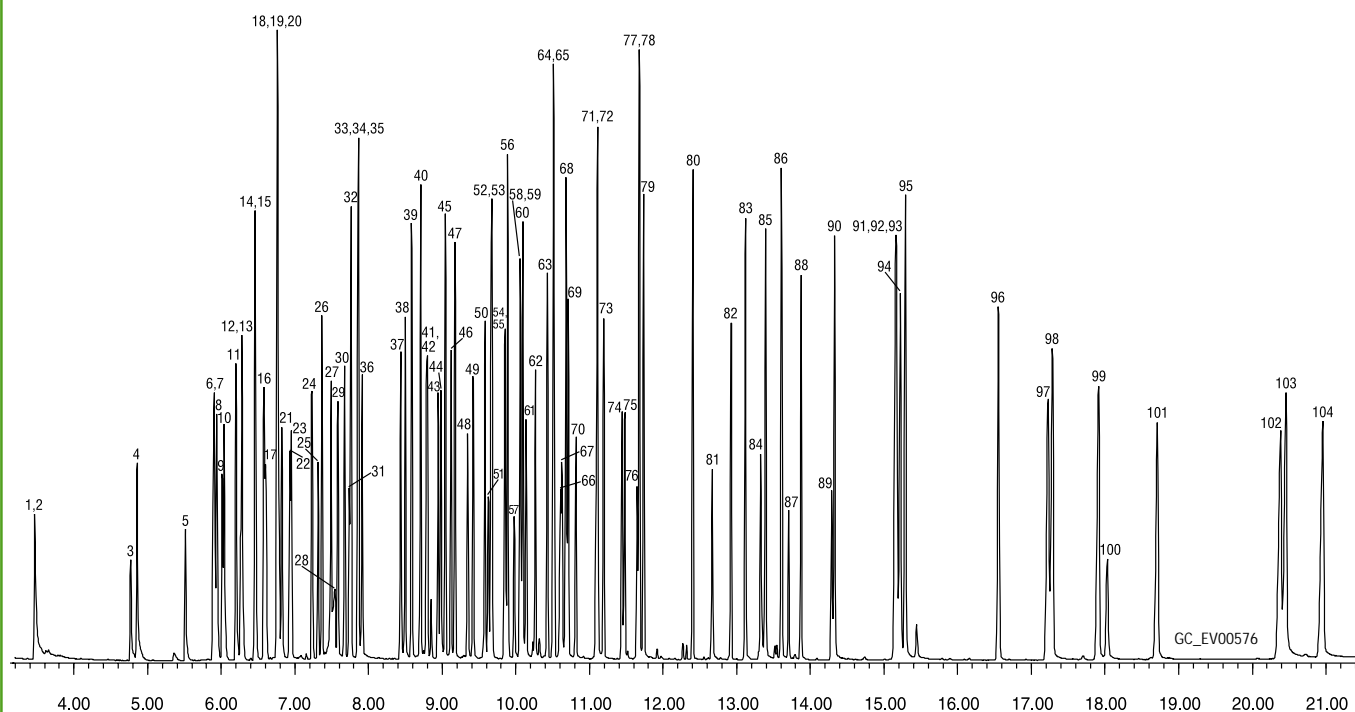
Questions?

Restek's Technical Service
Team is always here to help.
Call us at 800-356-1688 or
814-353-1300, ext. 4,
or email us at
support@restekcorp.com

Reducing Discrimination

Reduced response of the higher molecular weight semivolatile compounds can be caused by discrimination in the injection port. In extreme cases response for the last three PAH compounds may be lost completely at lower calibration levels. To reduce the effects of discrimination in the injection port, we recommend using a drilled Uniliner® inlet liner. Because the column seals into the taper at the bottom of the liner, there is reduced loss of high molecular weight compounds and improved response. The drilled Uniliner® inlet liner has a small hole drilled at the entrance that allows it to work with small diameter columns and Electronic Pressure Control (EPC) injection systems. When using the drilled Uniliner® liner (Figure 8), the response of the last three PAHs is significantly higher compared to the same analysis done with a normal splitless sleeve (see Figure 5). The drilled Uniliner® is available for Agilent 5890 and 6890 GCs (see page 19).

Figure 8. The Rtx®-5Sil MS combined with the Siltek™ drilled Uniliner® liner exhibits excellent peak shape and response for the semivolatile compounds listed in US EPA Method 8270.



1. N-nitrosodimethylamine	27. bis(2-chloroethoxy)methane	53. acenaphthylene	79. anthracene
2. pyridine	28. benzoic acid	54. acenaphthene-d10	80. di-n-butylphthalate
3. methyl methanesulfonate	29. 2,4-dichlorophenol	55. 3-nitroaniline	81. 4-nitroquinoline-1-oxide
4. 2-fluorophenol	30. 1,2,4-trichlorobenzene	56. acenaphthene	82. isodrin
5. ethyl methanesulfonate	31. naphthalene-d8	57. 2,4-dinitrophenol	83. fluoranthene
6. phenol-d6	32. naphthalene	58. pentachlorobenzene	84. benzidine
7. phenol	33. 2,6-dichlorophenol	59. 4-nitrophenol	85. pyrene
8. aniline	34. 4-chloroaniline	60. dibenzofuran	86. p-terphenyl-d14
9. bis(2-chloroethyl)ether	35. hexachloropropene	61. 2,4-dinitrotoluene	87. aramite
10. 2-chlorophenol	36. hexachlorobutadiene	62. 2,3,4,6-tetrachlorophenol	88. chlorbenzilate
11. 1,3-dichlorobenzene	37. 4-chloro-3-methylphenol	63. diethyl phthalate	89. kepone
12. 1,4-dichlorobenzene-d4	38. isosafrole	64. fluorene	90. butyl benzyl phthalate
13. 1,4-dichlorobenzene	39. 2-methylnaphthalene	65. 4-chlorophenyl phenyl ether	91. benzo(a)anthracene
14. 1,2-dichlorobenzene	40. 1-methylnaphthalene	66. 4-nitroaniline	92. 3,3'-dichlorobenzidine
15. benzyl alcohol	41. hexachlorocyclopentadiene	67. 4,6-dinitro-2-methylphenol	93. chrysene-d12
16. 2-methylphenol	42. 1,2,4,5-tetrachlorobenzene	68. diphenylamine	94. chrysene
17. bis(2-chloroisopropyl)ether	43. 2,4,6-trichlorophenol	69. azobenzene	95. bis(2-ethylhexyl)phthalate
18. acetophenone	44. 2,4,5-trichlorophenol	70. 2,4,6-tribromophenol	96. di-n-octyl phthalate
19. 4-methylphenol/3-methylphenol	45. 2-fluorobiphenyl	71. phenacetin	97. benzo(b)fluoranthene
20. N-nitroso-di-n-propylamine	46. safrole	72. 4-bromophenyl phenyl ether	98. benzo(k)fluoranthene
21. hexachloroethane	47. 2-chloronaphthalene	73. hexachlorobenzene	99. benzo(a)pyrene
22. nitrobenzene-d5	48. 2-nitroaniline	74. pentachlorophenol	100. perylene-d12
23. nitrobenzene	49. 1-naphthoquinone	75. pentachloronitrobenzene	101. 3-methylcholanthrene
24. isophorone	50. dimethylphthalate	76. phenanthrene-d10	102. indeno(1,2,3-cd)pyrene
25. 2-nitrophenol	51. 1,3-dinitrobenzene	77. dinoseb	103. dibenzo(a,h)anthracene
26. 2,4-dimethylphenol	52. 2,6-dinitrotoluene	78. phenanthrene	104. benzo(g,h,i)perylene

30m, 0.25mm ID, 0.25µm Rtx®-5Sil MS (cat.# 12723)
Conc.: 24µg/mL in methylene chloride
(cat.#s: 31618, 31619, 31620, 31621, 31622, 31206, 31062, 31063)
Note: Internal standards at 8 ppm

Inj. vol.: 1µL
Inj type: splitless
Hold time: 0.4 min.
Inlet liner: drilled Uniliner® liner, Siltek™ deactivation (cat# 21054-214.1)
Inj. temp.: 300°C

Carrier gas: helium (1mL/min. constant flow)
Linear velocity: 34cm/sec.
Oven temp.: 35°C (hold 2 min.) to 260°C @ 20°C/min. (hold 0 min.), to 330° @ 6°C/min. (hold 1 min.)

Det. type: MS
Transfer line temp.: 280°C
Scan range: 35 to 550amu
Ionization: EI
Mode: full scan

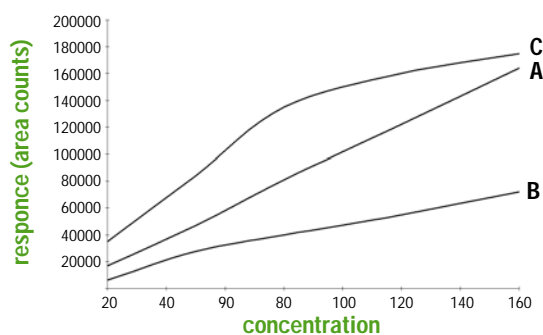
Quantitation

Because splitless injections suffer from irreproducibility, quantitation for semivolatile compounds is by internal standard, using a single ion for each analyte. Internal standards at known concentrations are used to correct for variances in the amount of material transferred to the column with different injections, and also to track MS sensitivity.

All sample analyte concentrations are calculated using response factors obtained from the calibration curve. This method uses single ions (i.e., extracted ions) for each compound so that chromatographic resolution of each compound is not necessary. It is acceptable for compounds to co-elute, as long as they do not have any common ions used for quantitation. Typically, only isomers have similar spectra, so chromatographic resolution is required only for these compounds.

Figure 9 shows the possibilities for calibration curves for this analysis. Curve A is the desired result, indicating a proportional response with increasing concentration. This implies that there is no detector saturation or reactivity for this compound. Curve B is indicative of a compound that undergoes a reaction, usually in the injection port or on the head of the column. If a calibration curve like Curve B is observed, injection port or column maintenance is required. Finally, Curve C shows high-end roll-off, indicating saturation. If a calibration

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



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

Summary

Although the analysis of semivolatile organic compounds is one of the more difficult tests performed by environmental laboratories, using Restek's Rtx®-5Sil MS or Rtx®-5MS columns and following the tips presented in this guide can make it easier to perform. Correct sample preparation, extract cleanup, injection technique, analytical columns, standards, and quantitation can help minimize problems normally associated with semivolatile organic analyses. When problems occur, use the most appropriate troubleshooting and maintenance procedures to quickly optimize your analytical system. When faced with difficulties in your semivolatile analysis, remember that the majority of problems occur in the sample preparation and cleanup step, or in the GC injection port. If you still are having difficulties after reading through this guide, contact Restek's Technical Service Team via email at support@restekcorp.com or via phone at 800-356-1688 or 814-353-1300, ext. 4.

Rtx®-5MS and Rtx®-5Sil MS Columns

Conventional capillary gas chromatography (GC) columns use liquid stationary phases, many of which are crossbonded to yield a higher working temperature. Even with crossbonding, however, the liquid stationary phase will slowly elute. This elution of the stationary phase, also termed column bleed, is more detectable at higher temperatures and is typically observed as an increasing baseline that follows the oven temperature program. Depending on the method of detection, column bleed may not be an issue for certain separations. If the capillary column is connected to a sensitive detector like a mass spectrometer (MS), then column bleed can cause a number of problems—specifically misidentification of analytes, loss of sensitivity, and inaccurate quantitation.

The level of column bleed will affect the sensitivity of any MS, especially ion trap instruments, which use automatic gain control. As the level of column bleed increases, so does the signal from bleed ions in the mass spectra of analytes and unknowns. Also, sensitivity (or detection limit) severely degrades. The contribution of bleed ions to the mass spectra can result in misidentification of compounds, requiring laboratory personnel to subtract these ions before performing library searches. Doing so can add considerable time to their analyses. Finally, if bleed ions contribute to the signal of the quantitation mass, quantitation of analytes and unknowns will be miscalculated. For these reasons, it is critical that analysts choose the lowest-bleed column designed for GC/MS applications.

Many manufacturers offer “MS” phases for applications requiring low bleed. In many cases, these represent nothing more than the reporting of the bleed signal when the column was tested for a single analysis at the manufacturer. Restek has developed true low-bleed MS phases. These columns exhibit a much lower column bleed than was previously available. The Rtx®-5MS column is a low-bleed, dimethyl/diphenyl polysiloxane phase. The Rtx®-5Sil MS is a low-bleed silarylene methyl/phenyl phase. The combination of Restek’s polymer chemistry and rigorous QA testing ensures that each MS column exceeds requirements of the most sensitive mass spectrometers.

online
ordering
now available!
for U.S. customers only
www.restekcorp.com

ID	df (µm)	temp. limits	15-Meter	30-Meter	60-Meter
0.25mm	0.10	-60 to 330/350°C	12605	12608	12611
	0.25	-60 to 330/350°C	12620	12623	12626
	0.50	-60 to 330/350°C	12635	12638	12641
	1.00	-60 to 325/350°C	12650	12653	
0.32mm	0.10	-60 to 330/350°C	12606	12609	12612
	0.25	-60 to 330/350°C	12621	12624	12627
	0.50	-60 to 330/350°C	12636	12639	12642
	1.00	-60 to 325/350°C	12651	12654	
0.53mm	0.50	-60 to 320/340°C	12637	12640	
	1.00	-60 to 320/340°C	12652	12655	
	1.50	-60 to 310/330°C	12667	12670	

Rtx®-5MS

Fused Silica (Crossbond® 5% diphenyl - 95% dimethyl polysiloxane) Stable to 360°C

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.10	-60 to 330/350°C	12705	12708
	0.25	-60 to 330/350°C	12720	12723
	0.50	-60 to 330/350°C	12735	12738
	1.00	-60 to 325/350°C	12750	12753
0.28mm	0.25	-60 to 330/350°C	12790	12793
	0.50	-60 to 330/350°C	12791	12794
	1.00	-60 to 325/350°C	12792	12795
0.32mm	0.10	-60 to 330/350°C	12706	12709
	0.25	-60 to 330/350°C	12721	12724
	0.50	-60 to 330/350°C	12736	12739
	1.00	-60 to 325/350°C	12751	12754
0.53mm	0.50	-60 to 320/340°C	12737	12740
	1.00	-60 to 320/340°C	12752	12755
	1.50	-60 to 310/330°C	12767	12770

Rtx®-5Sil MS

Fused Silica (equivalent selectivity of Crossbond® 5% diphenyl/95% dimethyl polysiloxane) Stable to 360°C



**Phases currently available as
Integra-Guard™ columns:**

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

Innovative Integra-Guard™ Columns

Some people swear by press-fit connectors, and others swear at them. For many analysts, the art of attaching a guard column to an analytical column is a mystery. Restek's chemists have discovered the solution to this mystery—the easiest, most reliable connection is no connection at all! No guard column system is more permanent than one continuous length of tubing containing both the guard column and the analytical column.

Restek offers a wide variety of Integra-Guard™ capillary columns with a guaranteed leak-free connection! The guard column is tied separately from the analytical column, using high-temperature string. The transition area between the columns is the point at which the guard column ends and the analytical column begins. The entire setup is suspended in our unique “crush-free” cage, which prevents the column from coming in contact with anything that could damage it.

Ordering is simple. Just add the appropriate suffix number and price to the analytical column's catalog number and price. For example, a 30m, 0.25mm ID, 0.25µm Rtx®-5MS with a 5m Integra-Guard™ column is cat.# 12623-124.

ID (mm)	Length (m)	Suffix #
0.25	5	-124
	10	-127
0.28	5	-243
	10	-244

ID (mm)	Length (m)	Suffix #
0.32	5	-125
	10	-128
0.53	5	-126
	10	-129

For analysts who prefer to attach a guard column to the analytical column themselves, Restek offers deactivated guard columns and Press-Tight® connectors.

formoreinfo

For detailed information on types of guard columns, their uses, and a complete product listing, request Restek's **Guard Column Fast Facts flyer** (lit. cat.# 59319)

Intermediate-Polarity Deactivated Guard Columns

Nominal ID (mm)	Nominal OD (mm)	1-Meter	5-Meter
0.15	0.363 ± 0.012	10101	10042
0.18	0.37 ± 0.04	10102	10046
0.25	0.37 ± 0.04	—	10043
0.28	0.37 ± 0.04	—	10003
0.32	0.45 ± 0.04	—	10044
0.45	0.69 ± 0.05	—	10005
0.53	0.69 ± 0.05	—	10045

Press-Tight® Connectors

- Seals all common sizes (0.18 to 0.53mm ID, outside diameters from 0.3 to 0.75mm) of fused silica tubing.
- Connect guard columns to analytical columns, repair broken columns, or connect column outlets to transfer lines.
- Angled connectors are designed to approximate the curvature of a capillary column and reduce strain on column-end connections.
- Made from inert fused silica.

Universal Angled Press-Tight® Connectors:

cat.# 20446 (5-pk.); cat.# 20447 (25-pk.); cat.# 20448 (100-pk.)

Universal Press-Tight® Connectors:

cat.# 20400 (5-pk.); cat.# 20401 (25-pk.); cat.# 20402 (100-pk.)

Deactivated Universal Press-Tight® Connectors:

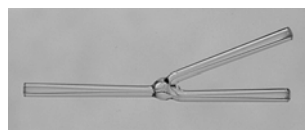
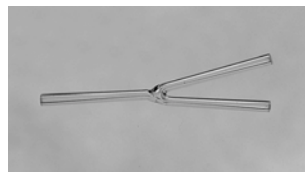
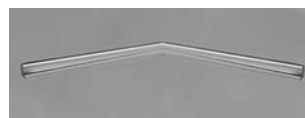
cat.# 20429 (5-pk.); cat.# 20430 (25-pk.); cat.# 20431 (100-pk.)

Universal “Y” Press-Tight® Connectors:

cat.# 20405 (ea.); cat.# 20406 (3-pk.)



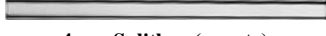
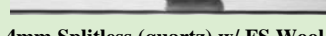


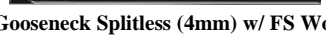


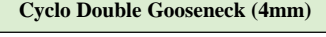
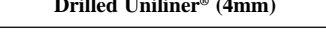
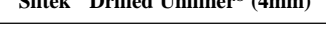
Universal Angled “Y” Press-Tight® Connectors:

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


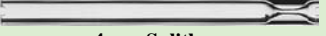
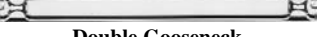
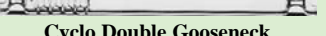



Inlet Liners

For Agilent/HP & Finnigan GCs

COLUMN INSTALLS THIS END	Splitless Liners for Agilent/HP & Finnigan GCs	Benefits/Uses:	ID*/OD & Length (mm)	ea.	cat.# 5-pk.	25-pk.	Similar to Agilent part #
	 4mm Splitless	trace samples >2µL	4.0 ID 6.5 OD x 78.5	20772	20773	20774	—
	 4mm Splitless w/ FS Wool	trace samples >2µL	4.0 ID 6.5 OD x 78.5	22400	22401	22402	19251-60540
	 4mm Splitless (quartz)	trace samples >2µL	4.0 ID 6.5 OD x 78.5	20912	20913	—	—
	 4mm Splitless (quartz) w/ FS Wool	trace samples >2µL	4.0 ID 6.5 OD x 78.5	22403	22404	—	18740-80220 5181-8818
	 Gooseneck Splitless (2mm)	trace samples <2µL	2.0 ID 6.5 OD x 78.5	20795	20796	20797	—
	 Gooseneck Splitless (4mm)	trace samples >2µL	4.0 ID 6.5 OD x 78.5	20798	20799	20800	5181-3316
	 Gooseneck Splitless (4mm) w/ FS Wool	trace samples >2µL	4.0 ID 6.5 OD x 78.5	22405	22406	22407	5062-3587
	 Double Gooseneck Splitless (4mm)	trace, active samples >2µL	4.0 ID 6.5 OD x 78.5	20784	20785	20786	5181-3315
	 Cyclo Double Gooseneck (2mm)	trace, active, dirty samples <2µL	2.0 ID 6.5 OD x 78.5	20907	20908	—	—
	 Cyclo Double Gooseneck (4mm)	trace, active, dirty samples >2µL	4.0 ID 6.5 OD x 78.5	20895	20896	20997	—
	 Drilled Uniliner® (4mm)	allows direct injection when using EPC-equipped GC	4.0 ID 6.3 OD x 78.5	21054	21055	—	—
	 Siltek™ Drilled Uniliner® (4mm)	allows direct injection when using EPC-equipped GC	4.0 ID 6.3 OD x 78.5	21054-214.1	21055-214.5	—	—




For Varian GCs

COLUMN INSTALLS THIS END	Splitless Liners for Varian 1075/1077GCs	Benefits/Uses:	ID*/OD & Length (mm)	ea.	cat.# 5-pk.	25-pk.	Similar to Varian Part #
	 2mm Splitless	trace samples <2µL	2.0 ID 6.3 OD x 74	20721	20722	20723	01-900109-05
	 4mm Splitless	trace samples >2µL	4.0 ID 6.3 OD x 74	20904	20905	20906	01-900109-05
	 Double Gooseneck	trace, active samples up to 4µL	4.0 ID 6.3 OD x 74	20847	20848	20849	—
	 Cyclo Double Gooseneck	trace, dirty, active samples up to 4µL	4.0 ID 6.3 OD x 74	20897	20898	—	—
COLUMN INSTALLS THIS END	1078/1079 Liners for Varian GCs	Benefits/Uses:	ID*/OD & length (mm)	ea.	cat.# 5-pk.	25-pk.	Similar to Varian Part #
	 1078/1079 Splitless	trace samples <2µL	2.0 ID 5.0 OD x 54	21711	21712	—	03-918466-00


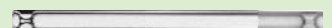


*No1

Inlet Liners



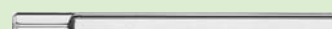

For Shimadzu GCs

Splitless Liners for Shimadzu GCs	Benefits/Uses:	ID**/OD & Length (mm)	ea.	cat.# 5-pk. 25-pk.	Similar to Shimadzu Part #
 94mm Splitless with Wool*	trace samples	3.5 ID 5.0 OD x 94	20955	20956 20957	221-09145
 94mm Double Gooseneck	reduces backflash and catalytic decomposition	3.5 ID 5.0 OD x 94	20958	20959 20960	—
 94mm Single Gooseneck	reduces backflash, also operates in DI mode	3.5 ID 5.0 OD x 94	20961	20962 20963	221-41599-00

For PerkinElmer GCs

Splitless Liners for PerkinElmer GCs	Benefits/Uses:	ID**/OD & Length (mm)	ea.	cat.# 5-pk. 25-pk.	Similar to PE Part #
 Auto SYS Splitless w/Wool (2mm ID)*	trace samples	2.0 ID 6.2 OD x 92.1	20829	20830 20831	N6101372
 Auto SYS Double Gooseneck	trace, active samples up to 4µL	3.5 ID 6.2 OD x 92.1	20853	20854 —	—
 Auto SYS Cyclo Double Gooseneck	trace, dirty, active samples up to 4µL	3.5 ID 6.2 OD x 92.1	20899	20900 —	—
 Auto SYS XL Split/Splitless	most common analyses	2.0 ID 4.0 OD x 86.2	21717	21718 —	N612-1004

For Thermo Orion GCs

Splitless Liners for 5000-6000 Series GCs	Benefits/Uses:	ID**/OD & Length (mm)	ea.	cat.# 5-pk. 25-pk.	Similar to TO Part #
 Splitless (4mm ID)	trace samples	4.0 ID 5.5 OD x 79.5	20814	20815 20816	—
Splitless Liners for 8000 & TRACE™ Series GCs	Benefits/Uses:	ID**/OD & Length (mm)	ea.	cat.# 5-pk. 25-pk.	Similar to TO Part #
 Splitless (3mm ID)	trace samples	3.0 ID 8.0 OD x 105	20942	20943 20944	453 20032
 Splitless (5mm ID)	trace samples	5.0 ID 8.0 OD x 105	20945	20946 20947	453 20033
 Double Gooseneck	trace, active samples up to 4µL	4.0 ID 8.0 OD x 105	20952	20953 —	—

O-Rings

Viton®

Viton® o-rings are universal. One size fits both split (6.3mm ID) and splitless (6.5mm ID) sleeves.

	Max. temp.	Similar to Agilent Part #	cat.#	Qty.
Viton® (fluorocarbon)	350°C	5180-4182	20377	25-pk.

Graphite

Graphite o-rings have excellent thermal stability and can be used at injection port temperatures up to 450°C!

	Similar to Agilent Part #	10-pk.	50-pk.
6.35mm ID for split liners	5180-4168	20296	20297
6.5mm ID for splitless liners	5180-4173	20298	20299



*Liner is
**Nomini

Deactivated Fused Silica Wool

- Ensure uniform vaporization in split or splitless liners.
- Prolong column life by trapping septum particles.
- Recommended for autosamplers with fast injection rates.
- Inertness tested for endrin breakdown.

cat.# 20790, (10 grams)

free
guide

Request the handy,
pocket-sized, *Inlet
Supplies Guide*
(lit. cat.# 59893A).



Replacement Inlet Seals

- Special grade of stainless steel deforms easily, ensuring a completely leak-free seal.
- Available in stainless steel, gold-plated, and Silcosteel®-treated.
- Cross-Disk ideal for high-flow split applications on EPC-equipped GCs.
- Shipped with washers.

For Agilent 5890/6890/6850 Split/Splitless Injection Ports

Single-Column Installation, Opening Size 0.8mm ID	
Stainless Steel Inlet Seal*	
21315, 2-pk.	21316, 10-pk.
Gold-Plated Inlet Seal**	
21317, 2-pk.	21318, 10-pk.
Silcosteel® Inlet Seal	
21319, 2-pk.	21320, 10-pk.

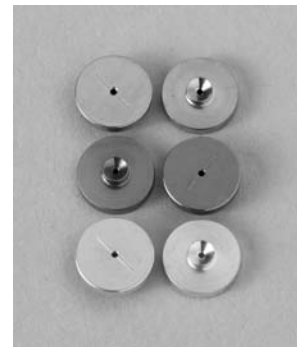
*0.8mm ID stainless steel inlet seal is equivalent to Agilent part #18740-20880.

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

Cross-Disk for Agilent GCs†

Cross-Disk, Opening Size 0.8mm ID	
Gold-Plated Inlet Seal	
20477, 2-pk.	20476, 10-pk.
Silcosteel® Inlet Seal	
20475, 2-pk.	20474, 10-pk.
Cross-Disk, Opening Size 1.2mm ID	
Gold-Plated Inlet Seal	
21009, 2-pk.	21010, 10-pk.
Silcosteel® Inlet Seal	
21011, 2-pk.	21012, 10-pk.

†Similar to Agilent part #5182-9652.



Thermolite® Septa

- Each batch tested on FIDs, ECDs, & MSDs to ensure lowest bleed.
- Excellent puncturability.
- Preconditioned and ready to use.
- Do not adhere to hot metal surfaces.
- Usable to 340°C inlet temperatures.
- Packaged in non-contaminating tins.

Septum Diameter	25-pk.	50-pk.	100-pk.
5mm (3/16")	20351	20352	20353
6mm (1/4")	20355	20356	20357
7mm	20381	20382	20383
8mm	20370	20371	—
9.5mm (3/8")	20359	20360	20361
10mm	20378	20379	20380
11mm (7/16")	20363	20364	20365
11.5mm	22385	22386	22387
12.5mm (1/2")	20367	20368	20369
17mm	20384	20385	20386
Shimadzu Blue	20372	20373	20374



Call our literature hotline
at 800-356-1688
or 814-353-1300, ext. 5,
or your local Restek
representative for
Restek's 20-page bulletin,
*A Guide to Minimizing
Septa Problems*
(lit. cat.# 59886).

free
guide

HRoMalytic +61(0)3 9762 2034
ECHnology Pty Ltd

Australian Distributors; Importers & Manufacturers

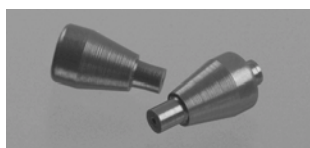
Website : www.chromtech.net.au E-mail : info@chromtech.net.au TelNo : 03 9762 2034 . . . in AUSTRALIA



EZ-Vent™ 2000 for Agilent GCs



EZ-Vent™ 2000 for Varian GCs



EZ-Vent™ 2000 ferrules

EZ-Vent™ 2000

- Change GC/MS columns in minutes without venting.
- Silcosteel® treated for greater inertness.
- Deactivated transfer line minimizes bleed into the source.
- Stainless steel body and high-temperature polyimide ferrules minimize leaks at the problematic transfer line fitting.
- Less expensive than other “no-vent” fittings.
- 100µm transfer line throttles vacuum and prevents column pump down.
- Available for Agilent GCs with 5971/5972 or 5973 MS and Varian 3400, 3600, or 3800 GCs with Saturn 2000 MS.
- Precision-machined orifice.



Kits

EZ-Vent™ 2000 for Agilent GCs with 5971/5972 or 5973 MS

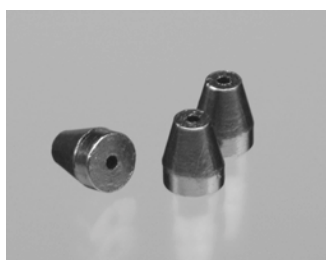
Includes EZ-Vent™ 2000, 1/16" SS nut, 0.4mm ID ferrules for connecting capillary column, 0.4mm ID ferrules for connecting transfer line, 100µm deactivated transfer line (3 ft.), and EZ-Vent™ column plug; cat.# 21013, (kit)

EZ-Vent™ 2000 for Varian Saturn 2000 MS systems with 3400, 3600, or 3800 GCs

Includes EZ-Vent™ 2000, 1/16" SS nut, 0.4mm ferrules for connecting capillary column, 0.4mm ID ferrules for connecting transfer line, 100µm deactivated transfer line (3 ft.), and EZ-Vent™ column plug; cat.# 21014, (kit)

Ferrules

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



Ferrule ID (mm)	Fits Column ID (mm)	Qty.	Graphite	Vespe®/ Graphite
0.4	0.25	50-pk.	20227	20229
0.4	0.25	10-pk.	20200	20211
0.5	0.32	10-pk.	20201	20212
0.5	0.32	50-pk.	20228	20231
0.6	0.28	10-pk.	—	20232
0.8	0.53	10-pk.	20202	20213
0.8	0.53	50-pk.	20224	20230

Compact Ferrules for Agilent GCs

Ferrule ID (mm)	Fits Column ID (mm)	Qty.	Graphite	Vespe®/ Graphite
0.4	0.25	10-pk.	20250	20238
0.4	0.25	50-pk.	20251	20239
0.5	0.32	10-pk.	21007	20248
0.5	0.32	50-pk.	21008	20249
0.8	0.53	10-pk.	20252	20263
0.8	0.53	50-pk.	20253	20264

Encapsulated Ferrules

- Will not deform and stick in fittings.
- Reusable.
- For 1/16" compression fittings.

Ferrule ID	Fits column ID	cat.#/10-pk.
0.4mm	0.25mm	21036
0.5mm	0.32mm	21037
0.8mm	0.53mm	21038

Direct Replacement Split/Splitless Injection Ports for Agilent GCs

Would you like better performance from your injector? Restek's Silcosteel®-coated split/splitless injector is a **direct replacement** for Agilent 5890 and 6890/6850 GCs. The injector is manufactured from high-quality stainless steel and meets or exceeds Agilent original equipment specifications. Silcosteel® passivates the metal surface to ensure an inert pathway for the sample, delivering increased performance.

SILCOSTEEL®
version available

For Agilent 5890 GCs

Description	cat.#, (ea.)
Replacement Weldment (Similar to Agilent part# 19251-60575)	20265
Replacement Shell Weldment (Similar to Agilent part# 19251-80570)	20266
Silcosteel® Weldment	20267
Silcosteel® Shell Weldment	20268

For Agilent 6890/6850 GCs

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



Weldment for Agilent 5890 GCs



Shell weldment for Agilent 5890 GCs



Weldment and shell weldment for Agilent 6890/6850 GCs

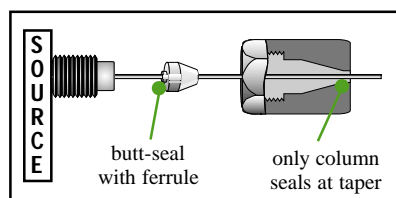
MSD Conversion Fitting—Improved

- Uses a flat, soft aluminum sealing ring to deform and butt-seal against the MSD interface (see figure below).
- A standard Vespel® ferrule seals the column and 1/16-inch stainless steel nut.
- Fitting is constructed of nickel-plated brass for longevity and softness.
- Can use any standard Vespel® or Vespel®/graphite 1/16-inch ferrule.
- Includes a 1/16-inch stainless steel nut and two replacement sealing rings. Order ferrules separately.

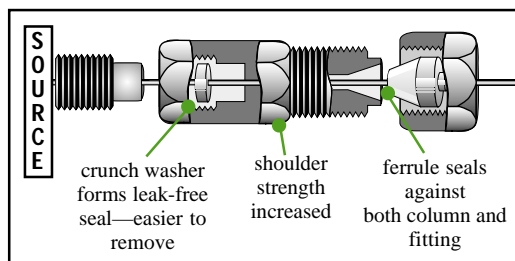
MSD Conversion Fitting: cat.# 21314, (ea.)

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

Original Agilent Design



Restek MSD Conversion Fitting

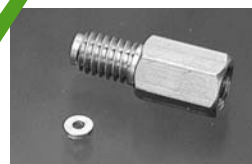


MSD Source Nut

The nut bore has been changed from 0.8mm to 1.2mm to permit easy removal of ferrules with a standard tapered-needle file (cat.# 20106). The nuts still match the manufacturer's original part specifications and are made of brass to prevent thread-stripping on the transfer line. (Similar to Agilent part #05988-20066.)

(Detector) MSD Source Nut: cat.# 20643, (2-pk.)

 **check it out**



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





US EPA Method 8270D outlines the analysis of semivolatile organic pollutants in solid waste, soil, water, and air matrices, using GC/MS. Update IVA of the third edition of SW-846—*Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*—includes EPA Method 8270D, in which there were no major revisions from EPA Method 8270C.

Method 8270 Calibration Kits

8270 Calibration Kit

31618: 8270 Calibration Mix #1
31619: 8270 Calibration Mix #2
31620: 8270 Calibration Mix #3
31621: 8270 Calibration Mix #4
31622: 8270 Calibration Mix #5



Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31626	31626-500

8270/Appendix IX Calibration Kit

31618: 8270 Calibration Mix #1
31619: 8270 Calibration Mix #2
31620: 8270 Calibration Mix #3
31621: 8270 Calibration Mix #4
31622: 8270 Calibration Mix #5
31623: 8270 Calibration Mix #6
31625: Appendix IX Mix #1



Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31627	31627-500

Aramite Mix

2,000ppm each in hexane, 1mL/ampul

	Each	5-pk.	10-pk.
	31624	31624-510	
w/data pack	31624-500	31624-520	31724

US EPA Methods 8270C & 8270D Analytical Reference Materials

Internal Standard Mixtures

SV Internal Standard Mix

acenaphthene-d10 naphthalene-d8
crysene-d12 perylene-d12
1,4-dichlorobenzene-d4 phenanthrene-d10
4,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31006	31006-510	
w/data pack	31006-500	31006-520	31106

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31206	31206-510	
w/data pack	31206-500	31206-520	31306

Surrogate Mixtures

B/N Surrogate Mix (4/89 SOW)

2-fluorobiphenyl p-terphenyl-d14
nitrobenzene-d5
1,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31024	31024-510	
w/data pack	31024-500	31024-520	31124

5,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31062	31062-510	
w/data pack	31062-500	31062-520	31162

5,000µg/mL each in methylene chloride, 5mL/ampul

	Each	5-pk.	10-pk.
	31086	31086-510	
w/data pack	31086-500	31086-520	31186

Acid Surrogate Mix (4/89 SOW)

2-fluorophenol 2,4,6-tribromophenol
phenol-d6
2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31025	31025-510	
w/data pack	31025-500	31025-520	31125

10,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31063	31063-510	
w/data pack	31063-500	31063-520	31163

10,000µg/mL each in methylene chloride, 5mL/ampul

	Each	5-pk.	10-pk.
	31087	31087-510	
w/data pack	31087-500	31087-520	31187

Matrix Spiking Mixtures

B/N Matrix Spike Mix

acenaphthene N-nitroso-di-n-propylamine
1,4-dichlorobenzene pyrene
2,4-dinitrotoluene 1,2,4-trichlorobenzene
1,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31004	31004-510	
w/data pack	31004-500	31004-520	31104

5,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31074	31074-510	
w/data pack	31074-500	31074-520	31174

5,000µg/mL each in methylene chloride, 5mL/ampul

	Each	5-pk.	10-pk.
	31084	31084-510	
w/data pack	31084-500	31084-520	31184

Acid Matrix Spike Mix

4-chloro-3-methylphenol pentachlorophenol
2-chlorophenol phenol
4-nitrophenol

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31014	31014-510	
w/data pack	31014-500	31014-520	31114

10,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31061	31061-510	
w/data pack	31061-500	31061-520	31161

10,000µg/mL each in methylene chloride, 5mL/ampul

	Each	5-pk.	10-pk.
	31071	31071-510	
w/data pack	31071-500	31071-520	31171

GC/MS Tuning Mixture

GC/MS Tuning Mixture

benzidine DFTPP
4,4'-DDT pentachlorophenol

1,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31615	31615-510	
w/data pack	31615-500	31615-520	31715

US EPA Methods 8270C & 8270D Analytical Reference Materials

Calibration Check Compound Mixtures

8270 B/N Calibration Check Mix

acenaphthene
benzo(a)pyrene
1,4-dichlorobenzene
di-*n*-octyl phthalate

diphenylamine
fluoranthene
hexachlorobutadiene

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31616	31616-510	
w/data pack	31616-500	31616-520	31716

8270 Acid Calibration Check Mix

4-chloro-3-methylphenol
2,4-dichlorophenol
2-nitrophenol

pentachlorophenol
phenol
2,4,6-trichlorophenol

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31617	31617-510	
w/data pack	31617-500	31617-520	31717

Calibration Mixtures

8270 Calibration Mix #1

benzoic acid
4-chloro-3-methylphenol
2-chlorophenol
2,4-dichlorophenol
2,6-dichlorophenol
2,4-dimethylphenol
4,6-dinitro-2-methylphenol
2,4-dinitrophenol
dinoseb
2-methylphenol

3-methylphenol
4-methylphenol
2-nitrophenol
4-nitrophenol
pentachlorophenol
phenol
2,3,4,6-tetrachlorophenol
2,4,5-trichlorophenol
2,4,6-trichlorophenol

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31618	31618-510	
w/data pack	31618-500	31618-520	31718

8270 Calibration Mix #2

aniline
benzidine
4-chloroaniline
3,3'-dichlorobenzidine
diphenylamine
2-nitroaniline

3-nitroaniline
4-nitroaniline
N-nitrosodimethylamine
N-nitrosodi-*n*-propylamine
pyridine

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31619	31619-510	
w/data pack	31619-500	31619-520	31719

Calibration Mixtures

8270 Calibration Mix #3

aramite
bis (2-chloroethyl) ether
bis (2-chloroethoxy) methane

bis (2-chloroisopropyl) ether
4-bromophenyl phenyl ether
chlorobenzilate
2-chloronaphthalene
4-chlorophenyl phenyl ether
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
1,3-dinitrobenzene

hexachlorobenzene
hexachlorobutadiene
hexachloro-cyclopentadiene
hexachloroethane
hexachloropropene
isodrin
kepone
pentachlorobenzene
pentachloronitrobenzene
1,2,4,5-tetrachlorobenzene
1,2,4-trichlorobenzene

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31620	31620-510	
w/data pack	31620-500	31620-520	31720

8270 Calibration Mix #4

acetophenone
azobenzene
benzyl alcohol
bis (2-ethylhexyl) phthalate
butyl benzyl phthalate
dibenzofuran
diethyl phthalate
dimethyl phthalate
di-*n*-butyl phthalate
di-*n*-octyl phthalate
2,4-dinitrotoluene

2,6-dinitrotoluene
ethyl methanesulfonate
isophorone
isosafrole (*cis* & *trans*)
methyl methanesulfonate
1,4-naphthoquinone
nitrobenzene
4-nitroquinoline-1-oxide
phenacetin
safrole

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31621	31621-510	
w/data pack	31621-500	31621-520	31721

8270 Calibration Mix #5

acenaphthene
acenaphthylene
anthracene
benzo(a)anthracene
benzo(a)pyrene
benzo(b)fluoranthene
benzo(ghi)perylene
benzo(k)fluoranthene
chrysene
dibenz(a,h)anthracene

fluoranthene
fluorene
ideno(1,2,3-cd)pyrene
3-methylcholanthrene
1-methylnaphthalene
2-methylnaphthalene
naphthalene
phenanthrene
pyrene

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31622	31622-510	
w/data pack	31622-500	31622-520	31722

Calibration Mixtures

8270 Calibration Mix #6

diallate (*cis* & *trans*)
dimethoate
disulfoton
famphur
methyl parathion
phosphorothioate

parathion
phorate
pronamide
thionazine
0,0,0-triethyl

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31623	31623-510	
w/data pack	31623-500	31623-520	31723

Organochlorine Pesticide Mix AB #1

aldrin
α-BHC
α-chlordane
β-BHC
4,4'-DDD
4,4'-DDE
4,4'-DDT
δ-BHC
dieldrin
endosulfan I

endosulfan II
endosulfan sulfate
endrin
endrin aldehyde
endrin ketone
γ-BHC (lindane)
γ-chlordane
heptachlor
heptachlor epoxide (B)
methoxychlor

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

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

Appendix IX Mix #1

2-acetylaminofluorene
4-aminobiphenyl
p-dimethylaminoazobenzene
3,3'-dimethylbenzidine
α,α'-dimethylphenethylamine (free base)
methapyrilene (free base)
1-naphthylamine
2-naphthylamine
5-nitro-*o*-toluidine
N-nitrosodibutylamine

N-nitrosodiethylamine
N-nitrosomethylethylamine
N-nitrosomorpholine
N-nitrosopiperidine
N-nitrosopyrrolidine
1,4-phenylenediamine
2-picoline
o-toluidine

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31625	31625-510	
w/data pack	31625-500	31625-520	31725

CLP GPC Calibration Mix

Qualitative mixture useful for determining GPC dump/collect times. Data packs are not available. The compounds are dissolved in methylene chloride at the concentrations listed.

CLP GPC Calibration Mix

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

In methylene chloride, 1mL/ampul

1mL/ampul	Each	5-pk.	10-pk.
	32019	32019-510	32119
5mL/ampul	Each	5-pk.	10-pk.
	32023	32023-510	32123

Revised GPC Calibration Mix

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

In methylene chloride, 1mL/ampul

1mL/ampul	Each	5-pk.	10-pk.
	32041	32041-510	32141
5mL/ampul	Each	5-pk.	10-pk.
	32042	32042-510	32142

**Restek
Tip**

CLP OLM 04.1, 04.2 Semivolatile Dilution

Benzaldehyde and atrazine will react quickly and directly with the methanol stabilizer used in most brands and grades of methylene chloride. This reaction will prevent you from obtaining stable, working-level calibration standards. Therefore, Restek has prepared the CLP OLM 04.1 Semivolatile B/N Mega-Mix™ from methylene chloride that is stabilized with amylene and is completely free of methanol. Restek strongly recommends screening the methylene chloride you use to dilute these mixtures to confirm that it is free of methanol.

EPA CLP—Semivolatiles Calibration Mixtures

04.1 SOW, 04.2 OSW

Restek chemists carefully reviewed the 04.2 Statement of Work and determined that the identical products listed in 04.1 will also be required for the 04.2 revision. The products listed here are a result of this work.

CLP 04.1 Phenols Calibration Mix

4-chloro-3-methylphenol	2-methylphenol
2-chlorophenol	4-methylphenol
4-nitrophenol	2-nitrophenol
2,4-dichlorophenol	pentachlorophenol
2,4-dimethylphenol	phenol
2,4-dinitrophenol	2,4,5-trichlorophenol
2-methyl-4,6-dinitrophenol	2,4,6-trichlorophenol

2,000µg/mL each in methylene chloride, 1mL/ampul

	Each	5-pk.	10-pk.
	31494	31494-510	—
w/data pack	31494-500	31494-520	31594

CLP 04.1 B/N MegaMix™

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

CLP 04.1 B/N MegaMix™ Mix A

acenaphthene	di-n-octyl phthalate
acenaphthylene	dibenz(a,h)anthracene
acetophenone	dibenzofuran
anthracene	3,3'-dichlorobenzidine
atrazine	diethyl phthalate
benzo(a)anthracene	dimethyl phthalate
benzaldehyde	2,4-dinitrotoluene
benzo(a)pyrene	2,6-dinitrotoluene
benzo(b)fluoranthene	fluoranthene
benzo(k)fluoranthene	fluorene
benzo(ghi)perylene	hexachlorobenzene
1,1'-biphenyl	hexachlorobutadiene
bis(2-chloroethoxy)methane	
hexachlorocyclopentadiene	hexachloroethane
bis(2-chloroethyl)ether	ideno(1,2,3-cd)pyrene
bis-(2-chloroisopropyl)ether	isophorone
bis(2-ethylhexyl)phthalate	2-methylnaphthalene
4-bromophenyl phenyl ether	naphthalene
butyl benzyl phthalate	nitrobenzene
caprolactam	n-nitrosodi-n-propylamine
carbazole	n-nitrosodiphenylamine
2-chloronaphthalene	phenanthrene
4-chlorophenyl phenyl ether	pyrene
chrysene	
di-n-butyl phthalate	

1,000µg/mL each in methylene chloride/benzene (75:25)

CLP 04.1 B/N MegaMix™ Mix B

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

1,000µg/mL each in methylene chloride

	Each	5-pk.	10-pk.
	31495	31495-510	—
w/data pack	31495-500	31495-520	31595

CLP OLM 04.1 SV Kit #1

31000: SV Screening Mix
31001: SV Tuning Mix
31493: CLP 04.1 BNA Surrogate Mix
31492: CLP 04.1 B/N Matrix Spike Mix
31005: Acid Matrix Spike Mix
31006: SV Internal Standard Mix
31494: CLP 04.1 Phenols Calibration Mix
31495: CLP 04.1 B/N MegaMix™
31012: SV Calibration Mix #6 (pesticides)

kit

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31603	31603-500

CLP OLM 04.1 SV Kit #2

31494: CLP 04.1 Phenols Calibration Mix
31495: CLP 04.1 B/N MegaMix™
31012: SV Calibration Mix #6 (pesticides)

kit

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31604	31604-500

CLP OLM 04.1 SV Kit #3

31494: CLP 04.1 Phenols Calibration Mix
31495: CLP 04.1 B/N MegaMix™

kit

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31605	31605-500

Questions?

Restek's Technical Service Team is always here to help. Call us at 800-356-1688 or 814-353-1300, ext. 4, or email us at support@restekcorp.com

EPA CLP—Semivolatiles Calibration Mixtures and Kits

3/90 and 4/89 SOW

SV Calibration Mix #1

benzyl alcohol 3-nitroaniline
4-chloroaniline 4-nitroaniline
2-nitroaniline

2,000µg/mL each in CH₂Cl₂, 1mL/ampul

	Each	5-pk.	10-pk.
	31007	31007-510	
w/data pack	31007-500	31007-520	31107

SV Calibration Mix #2

benzoic acid 4-methylphenol
4-chloro-3-methylphenol 2-nitrophenol
2-chlorophenol 4-nitrophenol
2,4-dichlorophenol pentachlorophenol
2,4-dimethylphenol phenol
2,4-dinitrophenol 2,4,5-trichlorophenol
2-methyl-4,6-dinitrophenol 2,4,6-trichlorophenol
2-methylphenol

2,000µg/mL each in CH₂Cl₂, 1mL/ampul

	Each	5-pk.	10-pk.
	31008	31008-510	
w/data pack	31008-500	31008-520	31108

SV Calibration Mix #3

bis(2-chloroethoxy)methane 4-chlorophenyl phenyl ether
bis(2-chloroethyl)ether dimethyl phthalate
bis(2-chloroisopropyl)ether di-*n*-butyl phthalate
bis(2-ethylhexyl)phthalate di-*n*-octyl phthalate
4-bromophenyl phenyl ether N-nitrosodimethylamine
butyl benzyl phthalate N-nitroso-di-*n*-propylamine
2-chloronaphthalene N-nitrosodiphenylamine

2,000µg/mL each in CH₂Cl₂, 1mL/ampul

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

SV Calibration Mix #4

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

2,000µg/mL each in CH₂Cl₂, 1mL/ampul

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

SV Calibration Mix #5

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

2,000µg/mL each in CH₂Cl₂, 1mL/ampul

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

SV Calibration Mix #6

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

2,000µg/mL each in toluene/hexane (1:1), 1mL/ampul

	Each	5-pk.	10-pk.
	31012	31012-510	
w/data pack	31012-500	31012-520	31112

SV Calibration Mix #7

1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene

2,000µg/mL each in CH₂Cl₂, 1mL/ampul

	Each	5-pk.	10-pk.
	31013	31013-510	
w/data pack	31013-500	31013-520	31113

3,3'-Dichlorobenzidine

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

	Each	5-pk.	10-pk.
	31026	31026-510	
w/data pack	31026-500	31026-520	31126

CLP Semivolatile Calibration Kit #1

(with pesticides)

kit

31007: SV Calibration Mix #1 (anilines)
31008: SV Calibration Mix #2 (phenols)
31009: SV Calibration Mix #3 (base neutrals)
31010: SV Calibration Mix #4 (base neutrals)
31011: SV Calibration Mix #5 (PAHs)
31012: SV Calibration Mix #6 (pesticides)
31013: SV Calibration Mix #7 (dichlorobenzenes)
31026: 3,3'-dichlorobenzidine

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31461	31461-500

CLP Semivolatile Calibration Kit #2

(without pesticides)

kit

31007: SV Calibration Mix #1 (anilines)
31008: SV Calibration Mix #2 (phenols)
31009: SV Calibration Mix #3 (base neutrals)
31010: SV Calibration Mix #4 (base neutrals)
31011: SV Calibration Mix #5 (PAHs)
31013: SV Calibration Mix #7 (dichlorobenzenes)
31026: 3,3'-dichlorobenzidine

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31462	31462-500

Semivolatile Calibration Kit #3

(with benzidine)

kit

31007: SV Calibration Mix #1 (anilines)
31008: SV Calibration Mix #2 (phenols)
31009: SV Calibration Mix #3 (base neutrals)
31010: SV Calibration Mix #4 (base neutrals)
31011: SV Calibration Mix #5 (PAHs)
31013: SV Calibration Mix #7 (dichlorobenzenes)
31030: 605 Benzidines Calibration Mix
(benzidine & 3,3'-dichlorobenzidine)

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31463	31463-500

Semivolatile Organics Kit (3/90 SOW)

kit

31000: SV Screening Mix
31001: SV Tuning Compound
31002: B/N Surrogate Std. Mix (3/90 SOW)
31003: Acid Surrogate Std. Mix (3/90 SOW)
31004: B/N Matrix Spike Mix
31005: Acid Matrix Spike Mix
31006: SV Internal Standard Mix
31007: SV Calibration Mix #1
31008: SV Calibration Mix #2
31009: SV Calibration Mix #3
31010: SV Calibration Mix #4
31011: SV Calibration Mix #5
31012: SV Calibration Mix #6
31013: SV Calibration Mix #7
31026: 3,3'-dichlorobenzidine

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
31051	31051-500

Reach for Restek!



Restek's **Chromatography Information Services (CIS)** team focuses its resources on innovative ways to teach the art and science of chromatography. This knowledge management group is dedicated to exploring and implementing ways of capturing, organizing, and disseminating chromatographic knowledge, experience, and wisdom to our internal and external customers worldwide.



Our **Technical Service Department** is staffed with over 35 experienced chemists from various departments within Restek. Whether your chromatography problem is simple or complex, reach for Restek's Technical Service Team and we will do everything we can to help you find a solution.



Restek's highly-trained **Customer Service Team** looks forward to providing you Plus 1™ service. Plus 1™ service means we will surpass your expectations every time you contact us. We are here when you place an order, track a package, check the status of an open order, or request a price quote. That's what having the best customer service in the business is all about—Plus 1™ service!

online
ordering
now available!
for U.S. customers only

new products • tech support • online ordering
literature archive • chromatogram search wizard
trade show presentations • company information
job opportunities • seminar dates • much more!

www.restekcorp.com

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

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



Restek trademarks: Crossbond®, EZ-Vent™, MegaMix™, Plus 1™, Press-Tight®, Rtx®, Silcosteel®, Stabilwax®, Thermolite®, XTT®. **Other trademarks:** Agilent (Agilent Technologies, Inc.); Auto SYS (Perkin-Elmer); Channeltron (Galileo); Florisil (U.S. Silica Co.); Mininert (Valco Instruments Co., Inc.); Rapid Vap (Labconco Corporation); Teflon, Vespel, and Viton (E.I. duPont de Nemours & Co., Inc.); TRACE (ThermoQuest Corp.); Whatman (Whatman Paper Limited Company). For permission to reproduce any portion of this technical guide, please contact Restek's publications/graphics department by phone (ext. 2128) or FAX (814) 353-9278. ©Copyright 2002, Restek Corp.

Lit. Cat. #59411A



110 Benner Circle
Bellefonte, PA 16823

Restek France: 01 60 78 32 10

Restek GmbH: 06172 2797 0

Restek Ireland: 28 9081 4576

Thames Restek U.K. Ltd.: 01753 624111

Or contact:

European Sales Manager: 28 9081 4576

Middle East Sales Manager: 01753 624111

PRESORTED STANDARD
U.S. POSTAGE
PAID
RESTEK



Australian Distributors; Importers & Manufacturers

Website : www.chromtech.net.au E-mail : info@chromtech.net.au TelNo : 03 9762 2034 . . . in AUSTRALIA