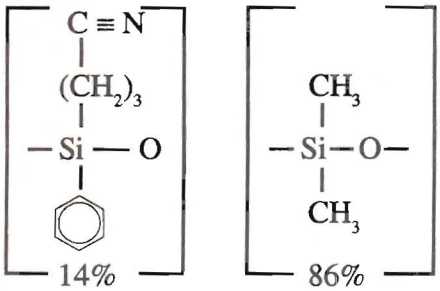
THE RESTEK ADVANTAGE

Rtx®-1701

The Highest Quality 1701 Columns Available

- Polymer synthesized and purified by Restek in our own laboratory.
- Ideal for pesticide, solvent, and drug analysis.
- Maximum operating temperature 280°C.
- Available in a wide variety of lengths, IDs, and film thicknesses.

Figure 1 - Rtx®-1701 phase composition.
14% cyanopropylphenyl
86% dimethyl polysiloxane



One of the most popular stationary phases used in capillary gas chromatography is the Rtx®-1701 (14% cyanopropyl phenyl/86% dimethyl polysiloxane). The unique polarity of this phase makes it ideal for a wide variety of applications including the analysis of chlorinated pesticides, solvents, and drugs. Rtx®-1701 columns are fully bonded, exhibit low bleed, and can be used with even the most sensitive detectors. Each Rtx®-1701 column is tested for inertness, efficiency, and bleed.

Thermal stability

The chemists at Restek have coupled innovative polymer synthesis with advanced deactivation techniques to produce an intermediate polar stationary phase with high thermal stability. The Rtx®-1701 polymer is thermally stable to 280°C. However, the usable operating temperature will decrease with increased film thickness. While a 1.5µm film of Rtx®-1701 is thermally stable to 280°C, the bleed would not be acceptable with most detectors. Therefore, the recommended operating temperature was reduced to 250°C as a practical usable operating temperature. Table I shows the film thicknesses and the recommended operating temperatures for Restek's Rtx®-1701 columns.

Table I: Rtx®-1701's Film Thicknesses and Recommended Operating Temperatures

Film Thickness	Recommended Operating Temperature
0.10 & 0.25µm	280°C
0.50µm	270°C
1.00µm	260°C
1.50µm	250°C

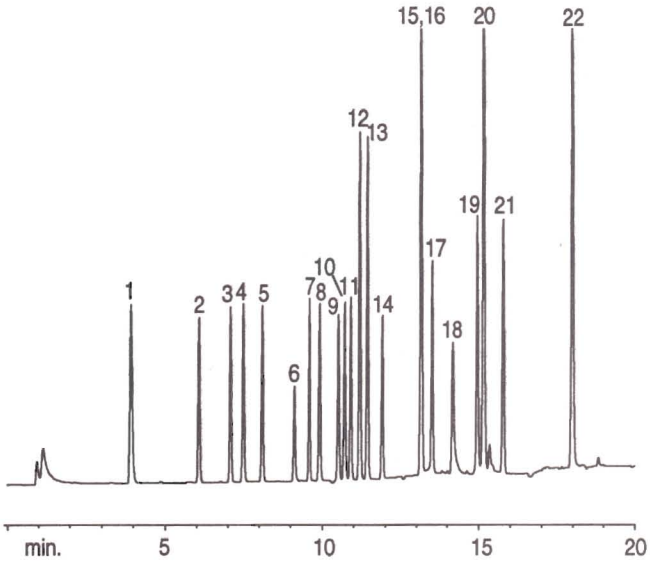
in this issue...

Applications

Rtx®-1701 is recommended for the analysis of CLP chlorinated pesticides

The analytical protocol for the EPA's Contract Lab Program (CLP) requires running samples on two different polarity columns for positive identification and improved quantitative reliability. One of the columns commonly used for CLP pesticide analysis is the Rtx®-1701. It is used in conjunction with either an Rtx®-5, Rtx®-35, or Rtx®-50 column. Most CLP labs use the 30 meter, 0.53mm ID, 0.50µm or the 30 meter, 0.53mm ID, 1.0µm Rtx®-1701 column. However, by using a thin film column, resolution can be improved, analysis time shortened, and bleed levels decreased. Figure 2 shows the analysis of CLP pesticides on a 30 meter, 0.53mm ID, 0.25µm Rtx®-1701 column. Analysis time is reduced to 18 minutes and resolution is improved compared with thicker film columns. Also, the thinner film produces slightly less bleed which is very important with sensitive Electron Capture Detectors (ECDs) used for this analysis.

Figure 2 - A thin film Rtx®-1701 column provides fast analyses and lower ECD bleed for CLP pesticides.



1. tetrachloro-*m*-xylene
2. α-BHC
3. γ-BHC
4. heptachlor
5. aldrin
6. β-BHC
7. δ-BHC
8. heptachlor epoxide
9. endosulfan I
10. γ-chlordane
11. α-chlordane
12. p,p'-DDE
13. dieldrin
14. endrin
15. p,p'-DDD
16. endosulfan II
17. p,p'-DDT
18. endrin aldehyde
19. endosulfan sulfate
20. methoxychlor
21. endrin ketone
22. decachlorobiphenyl

30m, 0.53mm ID, 0.25µm Rtx®-1701 (cat.# 12025)
1.0µl direct injection of pesticides
concentration: 80-800ppb (ng/ml)

Oven temp.:	150°C (hold 2 min.) to 275°C @ 7°C/min. (hold 1 min.)		
Inj./det. temp.:	200°C/275°C	**Detector:**	ECD
Carrier gas:	helium	**Linear velocity:**	40cm/sec. set @ 150°C
ECD sensitivity:	5.12 x 10^{-10} AFS	**Flow rate:**	5cc/min.

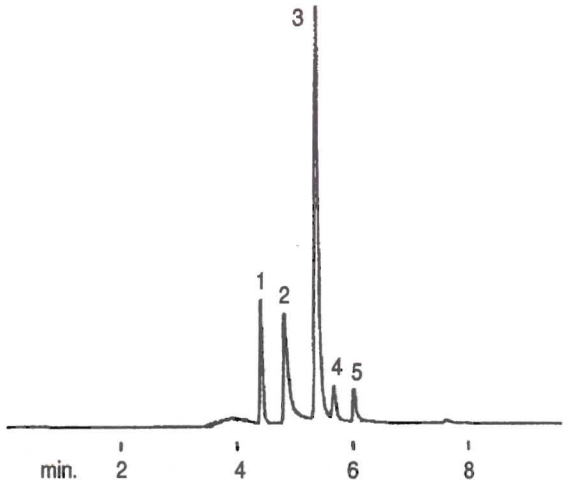
Separate formaldehyde, water, and methanol with an Rtx®-1701 column

Formaldehyde is the 24th highest volume chemical produced in the United States. It is used in the manufacture of phenolic resins and insulating foams. It is most commonly available as a 37-50% aqueous solution that can contain as much as 15% methanol. Therefore, the separation of formaldehyde from both water and methanol is critical for an accurate analysis of this common raw material. Figure 3 shows this important separation on a 60 meter, 0.25mm ID, 1.0µm Rtx®-1701 column. These three components, plus acetaldehyde, a common impurity in formaldehyde, are all separated in 6 minutes.

Analyze acidic/neutral drugs with an Rtx®-1701 column

Barbiturates, hypnotics, sedatives and anti-convulsants are acidic or neutral drugs that are considered to be polar in nature. Intermediate polarity columns produce better peak shapes for acidic and neutral drugs. Traditionally, the analysis

Figure 3 - The Rtx®-1701 resolves water and methanol from formaldehyde.

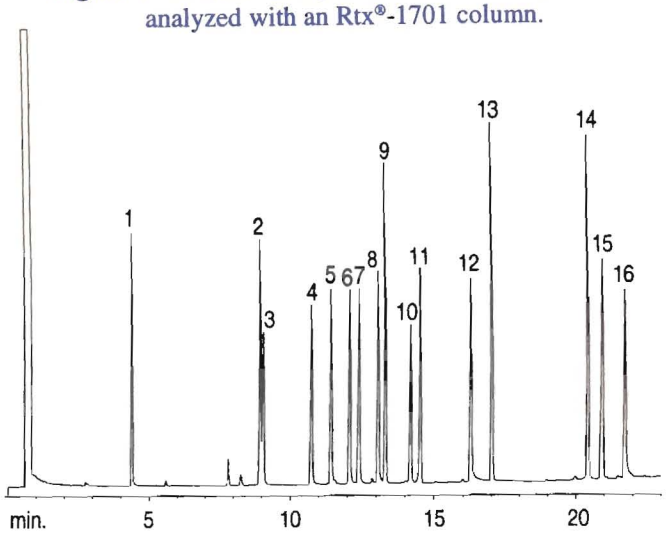


1. air
2. water
3. formaldehyde
4. acetaldehyde
5. methanol

60m, 0.25mm ID, 1.0µm Rtx®-1701 (cat.# 12056)
0.5µl split injection of a formaldehyde sample

Oven temp.:	40°C isothermal
Inj./det. temp.:	150°C/175°C
Detector:	TCD
Carrier gas:	helium
Linear velocity:	20cm/sec. (flow rate: 0.8cc/min.)
TCD sensitivity:	8 mV
Split vent:	30cc/min.

Figure 4 - A wide variety of acid/neutral drugs can be analyzed with an Rtx®-1701 column.



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

15m, 0.53mm ID, 0.50µm Rtx®-1701 (cat.# 12037)
1.0µl splitless injection of acidic/neutral drugs
concentration: 50µg/ml

Oven temp.:	100°C to 280°C @ 7°C/min.		
Inj./det. temp.:	250°C/175°C	**Detector:**	TCD
Carrier gas:	helium	**Linear velocity:**	40cm/sec. set @ 100°C
FID sensitivity:	5.12 x 10^{-10} AFS	**Splitless hold time:**	0.5 min.

of acidic and neutral drugs is performed on phenyl containing stationary phases like the Rtx®-20 or the Rtx®-35. The unique polarity of the Rtx®-1701 makes it ideal for use as either a primary column or as a confirmational column for these compounds. Figure 4 shows the analysis of a group of acidic and neutral drugs on a 15 meter, 0.53mm ID, 0.50µm Rtx®-1701 column. Good peak shapes and resolution can be maintained while providing elution order switching when compared to phenyl phases.

The low bleed and inertness of the Rtx®-1701 make it ideal for many types of analyses. It is available in a wide variety of lengths, IDs, and film thicknesses for a broad range of applications. Whether you are analyzing pesticides, solvents, or drugs the Rtx®-1701 will yield consistent and accurate results.

Rtx-1701 polymer is synthesized & purified in our own laboratory.

Product Listing

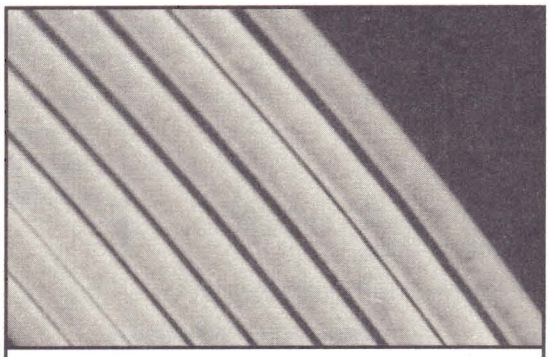
Rtx®-1701 (Fused Silica)		(Crossbond® 14% cyanopropylphenyl-86% methyl polysiloxane) Polymer stable to 280°C **Applications:** pesticides, PCBs, oxygenates, pharmaceuticals, solvents				
	df (µm)	**temp. range**	**15-meter**	**30-meter**	**60-meter**	**105-meter**
0.25mm ID	0.10	-20 280°C	12005	12008	12011	12014
	0.25	-20 280°C	12020	12023	12026	12029
	0.50	-20 270°C	12035	12038	12041	12044
	1.00	-20 260°C	12050	12053	12056	12059
0.32mm ID	0.10	-20 280°C	12006	12009	12012	12015
	0.25	-20 280°C	12021	12024	12027	12030
	0.50	-20 270°C	12036	12039	12042	12045
	1.00	-20 260°C	12051	12054	12057	12060
	1.50	-20 250°C	12066	12069	12072	12075
0.53mm ID	0.10	-20 280°C	12007	12010	12013	Custom
	0.25	-20 280°C	12022	12025	12028	
	0.50	-20 270°C	12037	12040	12043	
	1.00	-20 260°C	12052	12055	12058	
	1.50	-20 250°C	12067	12070	12073	
0.18mm ID	**df (µm)**	**temp. range**	**10-meter**	**20-meter**	**40-meter**	
	0.20	-20 280°C	42001	42002	42003	
	0.40	-20 270°C	42010	42011	42012	

MXT®-1701 metal columns are also available. Please see our 1994-95 Chromatography Products Catalog or call your local distributor for more information.

Analyze Fixed Gases Using the New Rt-Msieve™ 13X PLOT Columns

- Unique selectivity of Rt-Msieve 13X improves overall analysis.
- Immobilized to eliminate particle generation.
- Columns can be reactivated after water contamination.
- Guaranteed column-to-column reproducibility.
- Available in 0.53mm and 0.32mm IDs.

Until recently, the only way to achieve rapid separations of fixed gases was the use of molecular sieve packed and micropacked columns. Traditional Molecular Sieve5Å Porous Layer Open Tubular (PLOT) columns have been useful, but the extended retention and broadened peak width of carbon monoxide has been unavoidable. The Restek wizards have developed the Rt-Msieve™ 13X PLOT column to improve the analysis of fixed gases.



After ten minutes of aggressive sonication, the molecular sieve particles remain intact.

Fast and efficient analysis of fixed gases

The Rt-Msieve™ 13X combines the efficiency of traditional molecular sieve PLOT columns with the unique selectivity of 13X molecular sieve. Figure 1 shows the rapid and efficient analysis of the permanent gases on the 30m, 0.32mm ID Rt-Msieve™ 13X. Baseline separation of all compounds is achieved in just over 2 minutes. Figure 2 shows the same analysis using the 15m, 0.32mm ID Rt-Msieve™ 13X PLOT column with complete resolution in 1.5 minutes.

Unique selectivity of Molecular Sieve 13X material

Until now, only Molecular Sieve 5Å PLOT columns have been available. With the Rt-Msieve 13X PLOT columns, the separation of nitrogen and methane is increased while overall analysis time is decreased by reducing the retention of carbon monoxide.[1] The 13X molecular sieve also produces a narrower peak shape for carbon monoxide allowing for lower levels of detection. Figure 3 shows the analysis of the permanent gases on a Molecular Sieve 5Å PLOT column. While the 5Å PLOT column provides good resolution, the peak shapes are broadened, thus decreasing the minimum detection limit approximately 10-fold.

Resists particle generation

PLOT columns are prepared by coating a thick film of very small particles on the inside column wall. A major drawback of PLOT columns is particle generation caused by vibration or pressure surges. The material in the Rt-Msieve™ 13X has been immobilized by a process unique to Restek to minimize any particle generation. This immobilization is stable for applications where column flow rates are disrupted during valve switching or backflushing operations. Non-immobilized PLOT columns will damage or clog valves, causing expensive repairs and down time.

Available in 0.53mm ID and 0.32mm ID

The Rt-Msieve™ 13X is available in two configurations to satisfy a wide variety of applications. Use the 30m, 0.53mm ID Rt-Msieve™ 13X for most applications and when using on-line analyzers. The 0.53mm ID Rt-Msieve™ 13X PLOT columns offer the flexibility and increased capacity many analysts require. For increased efficiency and low flow applications, Restek offers the 30m, 0.32mm ID Rt-Msieve™ 13X. The 0.32mm ID Rt-Msieve™ 13X PLOT is ideal for portable analyzers having limited gas supplies where low carrier gas flow is essential. For decreased analysis times, 15-meter versions are available for both IDs.

Columns can be reactivated

Molecular sieves are very hydrophyllic and will adsorb any water present in the sample. Water contamination will have detrimental affects on separations causing, 1) the carbon monoxide peak shape to deteriorate and, 2) a reduction in overall resolution. Rt-Msieve™ 13X PLOT columns can be reactivated after water contamination by conditioning at 300°C under dry carrier gas flow, thus extending column lifetime.

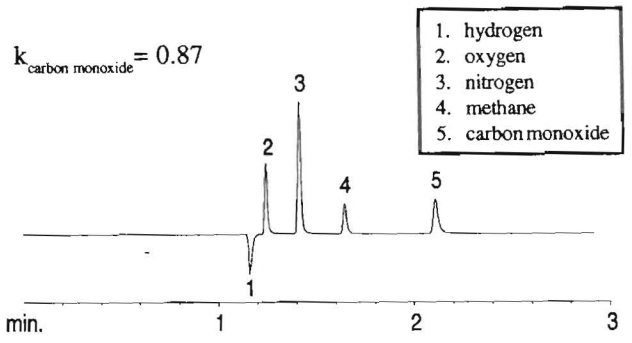
Column-to-column reproducibility guaranteed

All Rt-Msieve™ 13X PLOT columns are tested with a mixture of permanent gases. Columns must pass rigorous specifications for efficiency and strict retention time criteria. This stringent testing insures analysts of column-to-column and run-to-run reproducibility.

The resolution of permanent gases can be improved and the overall analysis time can be reduced using the new Rt-Msieve™ 13X PLOT columns. The immobilized particles minimize potential damage to valves and reduce detector noise. These columns are available in 0.53mm ID for increased capacity or in 0.32mm ID for reduced carrier gas consumption. Rigorous testing guarantees the performance of all Rt-Msieve™ 13X columns.

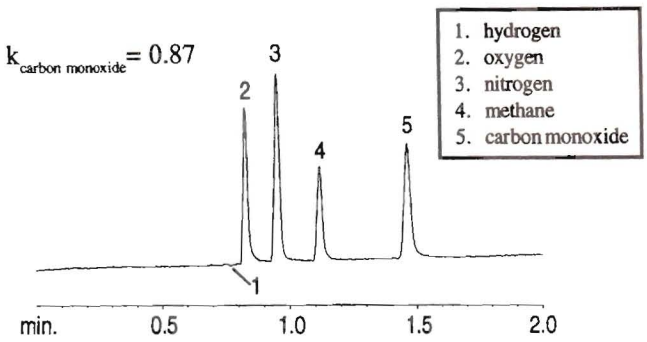
1. Cowper, C.J., DeRose, A.J., *The Analysis of Gases by Chromatography,* Pergamon Press, 1983.

Figure 1 - Resolve permanent gases in just over 2 minutes with a 30-meter Rt-Msieve™ 13X PLOT column.



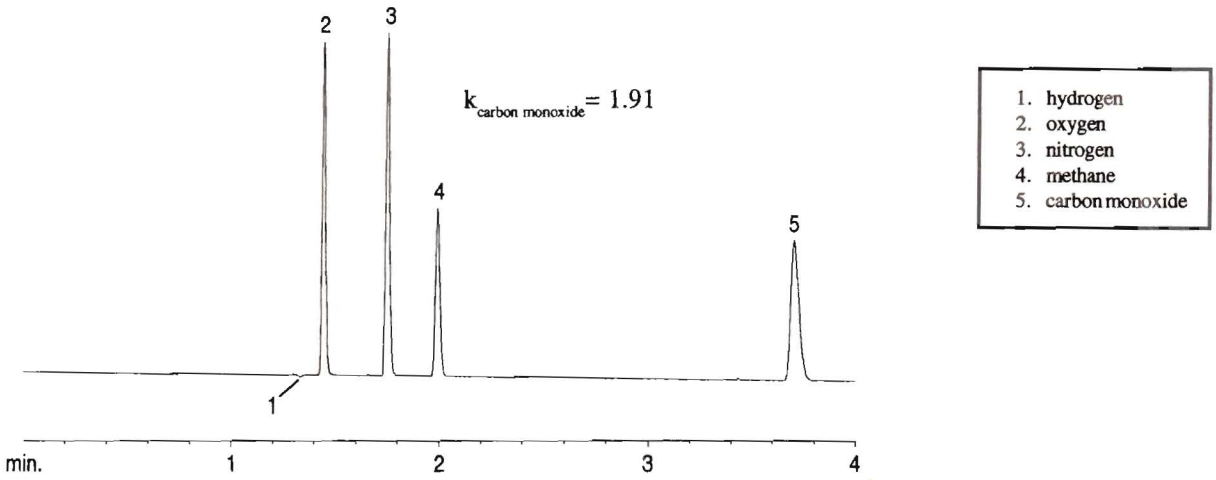
30m, 0.32mm ID Rt-Msieve™ 13X PLOT column (cat.# 19705)
15µl split injection of permanent gases (hydrogen spiked)
Oven temp.: 40°C isothermal
Inj./det. temp.: 200°C/200°C **Detector:** microcell TCD
Carrier gas: helium
Linear velocity: 44cm/sec. set @ 40°C (2cc/min.)
Detector sensitivity: 50mV full scale **Split ratio:** 15:1

Figure 2 - Resolve permanent gases in 1½ minutes with a 15-meter Rt-Msieve™ 13X PLOT column.



15m, 0.32mm ID Rt-Msieve™ 13X PLOT column (cat.# 19707)
20µl split injection of permanent gases
Oven temp.: 40°C isothermal
Inj./det. temp.: 200°C/200°C **Detector:** microcell TCD
Carrier gas: helium
Linear velocity: 32cm/sec. set @ 40°C (1.5cc/min.)
Detector sensitivity: 50mV full scale **Split ratio:** 15:1

Figure 3 - The Molecular Sieve 5Å column produces broader peak shapes and longer retention times for carbon monoxide than the Rt-Msieve™ 13X.



30m, 32mm ID Molecular Sieve 5A PLOT column
20µl split injection of permanent gases
Oven temp.: 40°C isothermal
Inj./det. temp.: 200°C/200°C **Detector:** microcell TCD
Carrier gas: helium **Linear velocity:** 39cm/sec. set @ 40°C (1.85cc/min.)
Detector sensitivity: 50mV full scale **Split ratio:** 15:1

Immobilized to eliminate particle generation.

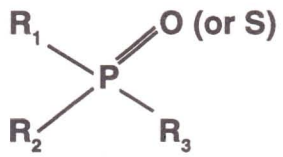
Rt-Msieve™ 13X Columns

15m, 0.32mm ID	cat. # 19707
15m, 0.53mm ID	cat. # 19708
30m, 0.32mm ID	cat. # 19705
30m, 0.53mm ID	cat. # 19706

Analyzing Organophosphorus Pesticides

Pesticides are comprised of many different classes of compounds such as phenoxy-acids and carbamates, as well as molecules containing chlorinated, nitrogen, and organophosphorus functionalities. Organophosphorus pesticides commonly exist as neutral phosphoryl or thiophosphoryl compounds (Figure 1). Their pentavalent character gives them literally hundreds of phosphorous moieties that result in a variety of chemical, physical, and biological properties. They are applied as fungicides, herbicides, insect chemosterilants, and contact or systemic insecticides. Organophosphorus pesticides have a short residence time in the environment due to their instability. Most readily hydrolyze, some photodegrade, and others decompose in alkaline conditions. These characteristics greatly contrast to chlorinated pesticides which show long-term persistence in the environment. The diversity of organophosphorus compounds and their limited life expectancy in the environment makes their use increasingly popular in common pesticide applications.

Figure 1



Most organophosphorus pesticides exist as phosphoryl or thiophosphoryl compounds.

The Environmental Protection Agency has developed specific methods to determine the presence and concentration of these compounds. EPA Method 8141A is a capillary gas chromatographic method that determines the level of organophosphorus compounds in water and soil matrices. This method recommends several different capillary columns for resolving the compounds of interest. In addition, the method has recommendations on detection systems and calibration procedures.

Restek's deactivation produces an inert column that reduces degradation of organophosphorus compounds.

When selecting a column for organophosphorus pesticide analysis, a chromatographer must consider three important factors: column inertness, efficiency, and retention time reproducibility. Because these pesticides are prone to breakdown, the column must be inert to prevent decomposition or reactivity. When analyzing complex mixtures, the column must exhibit high efficiency for maximum resolution. Some methods suggest the use of retention time windows. Retention time windowing requires that the column must demonstrate reproducible results to insure proper identification. Restek offers capillary columns that meet the requirements for analyzing organophosphorus pesticides. Two calibration mixtures containing 28 EPA Method 8141A compounds were completely resolved using an Rtx®-35, 30m, 0.53mm, 0.50µm column (Figures 2 and 3). The active organophosphorus pesticides exhibit minimal breakdown and were well resolved on the Rtx®-35 stationary phase. Relative standard deviations for retention times were less than 1%.

Figure 2 - Rtx®-35 is an excellent column for resolving organophosphorus pesticides.

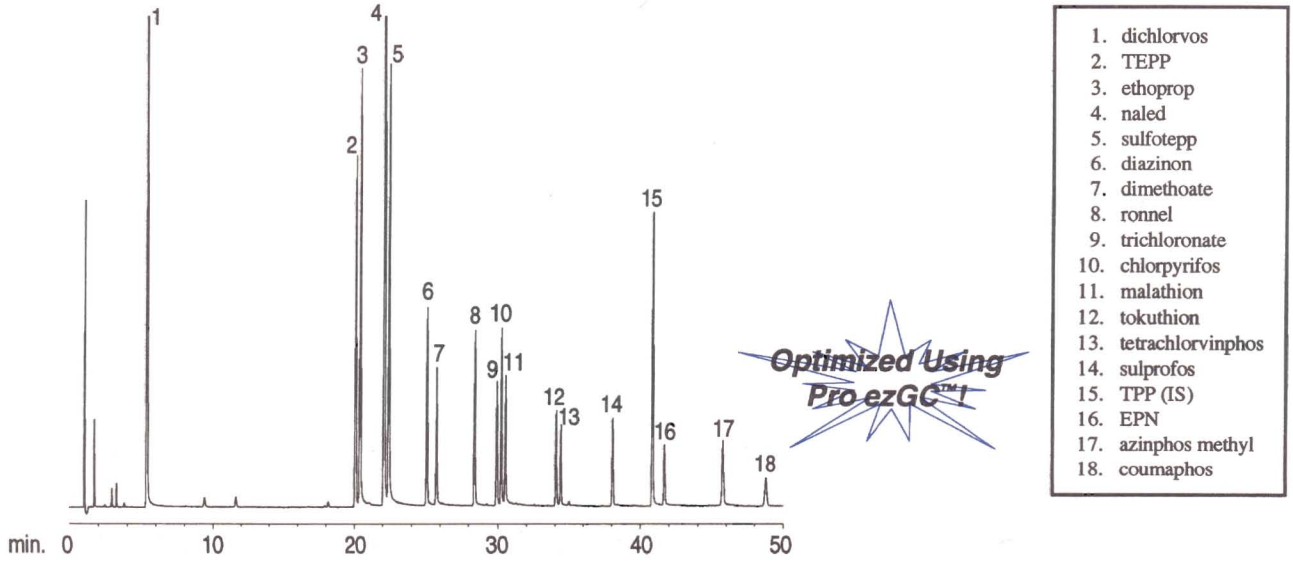
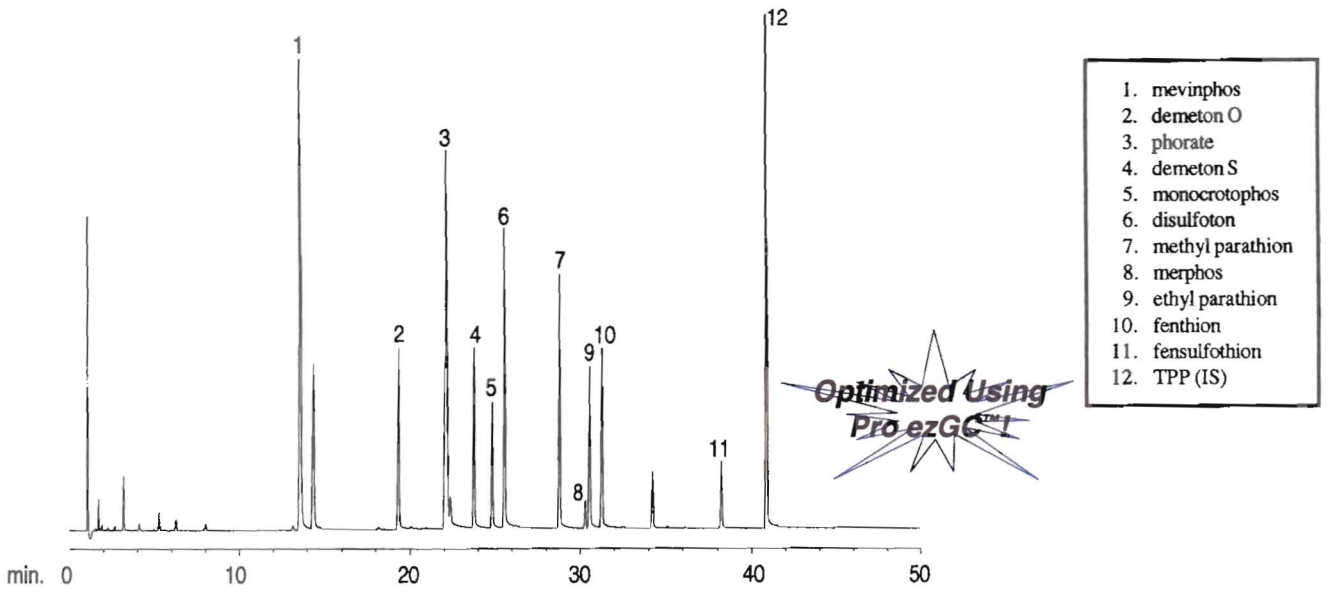


Figure 3 - The Rtx®-35 baseline resolves all organophosphorus pesticides.



In addition to column considerations, frequent maintenance must be performed on the inlet system. Over numerous analyses, the rate of adsorption may increase due to the non-volatile contamination that can build up in the inlet sleeve. Sleeve cleaning or replacement will typically restore chromatographic performance.

Analyzing organophosphorus pesticides can be demanding due to their unstable nature. Also, the wide variety of these pesticides can create difficult separation problems. Highly inert capillary columns, available from Restek, can alleviate the problem of analyte adsorption. These efficient and reproducible columns prevent coelutions and minimize percent errors in relative retention times.

References:
1. Eto, Morifusa. *Organophosphorus Pesticides: Organic and Biological Chemistry.* CRC Press, Inc., Ohio, 1974.
2. EPA SW-846 Module Method 8141A: *Organophosphorus Compounds By Gas Chromatography: Capillary Column Technique.* Non-promulgated, 1990.

Run Conditions for Figures 2 & 3

30m, 0.53mm ID, 0.50µm Rtx®-35 (cat.# 10440)
0.5µl direct injection of Organophosphorus Pesticide
Mix A (Fig. 2) & Mix B (Fig. 3)*
on-column concentration: 25-100ng
Oven temp.: 125°C (hold 10 min.) to 250°C @ 4°C/min. (hold 15 min.)
Inj./det. temp.: 200°C/250°C
Detector: FPD
Carrier gas: helium
Linear velocity: 35cm/sec. set @ 125°C
FPD sensivity: 2 x 10^{-9}AFS

Product Listing

Rtx®-35 column
30m, 0.53mm ID, 0.50µm
cat.# 10440

Vu-Tight® Direct Injection Inlet Sleeves

- Visually observe the press-tight connection between the column end and sleeve.
- Fits 0.32 and 0.53mm ID capillary columns.
- Slotted top prevents obstruction of carrier gas flow.
- Designed for 1/4" injection ports.

Vu-Tight® Direct Injection Sleeve (1/4" OD)

Can be easily packed with wool for dirty samples.

cat.# 20342 each
cat.# 20343, 5-pk.
cat.# 20344, 25-pk.

Cyclo Vu-Tight® Direct Injection Sleeve (1/4" OD)

Is ideal for dirty samples and prevents non-volatile residue from contaminating the column.

cat.# 20787 each
cat.# 20788, 5-pk.

Vu-Tight® Installation Fittings: Includes a 1/4" SS nut & graphite ferrule for attaching the sleeve to the GC inlet, and a 1/4" to 1/16" SS reducer & 1/4" by 0.5mm ID graphite ferrule for attaching the column to the sleeve. **cat.# 20504, kit**

Restek's Rtx®-65

Highest Percent Phenyl Stationary Phase Available

- Selective for aromatic compounds.
- Maximum operating temperature 300°C.
- Available in a variety of lengths, IDs, and film thicknesses.

Many of the most popular stationary phases are based on phenyl methyl polysiloxanes including Rtx®-5, Rtx®-20, Rtx®-35, and Rtx®-50. The popularity of these stationary phases is due to their high thermal stability and their selectivity for aromatic compounds. With increased phenyl substitution, better separation of similar aromatic compounds can be achieved through increased interaction of these compounds with the phase. The Rtx®-65TG, developed specifically for the analysis of triglycerides, resolves triglycerides by degree of unsaturation as well as by carbon number. This same stationary phase, Rtx®-65 (65%diphenyl/35%dimethyl polysiloxane), is now available for a wide variety of applications.

Retention indices

As the concentration of phenyl groups increase, stationary phase polarity also increases. For compounds in a homologous series, as stationary phase polarity increases, absolute retention of the compounds also increases. When analyzing different classes of compounds, higher polarity phenyl/methyl stationary phases will preferentially retain polar, aromatic compounds. Table I lists Kovat's retention indices of the Rtx®-65 compared to the Rtx®-50 and Stabilwax® phases. As the retention indices indicate, the Rtx®-65 shows greater retention than the Rtx®-50, but lower retention than "polar" phases such as the polyethylene glycol (Stabilwax®). The selective characteristics of the Rtx®-65 offer analysts a column at the upper end of the intermediate polarity range and separations previously unobtainable on lower or higher polarity phases.

Table I - Rtx®-65 Kovat's Retention Indices

phase	benzene	butanol	2-pentanone	nitropropane	average
Rtx®-50	777	764	806	911	815
Rtx®-65	794	779	825	937	834
Stabilwax®	956	1142	987	1217	1076

Thermal stability

The chemists at Restek have coupled innovative polymer synthesis with advanced deactivation techniques to produce an intermediate polarity stationary phase with high thermal stability. The maximum operating temperature of the Rtx®-65 is 300°C. However, due to the increased aromatic content, the minimum operating temperature of the phase is restricted to 50°C. Operating Rtx®-65 columns below this temperature will result in broad peak shapes of early eluting components. The film thicknesses and recommended minimum and maximum operating temperatures of the Rtx®-65 columns offered by Restek are shown in the product listing on page 9.

Applications

Rtx®-65 as a confirmational column for EPA Method 604 Phenols

Confirmatory analyses are often required in many EPA methods. By running samples on a second column of different polarity, a more positive confirmation of component identity is achieved. The analysis of priority pollutant phenols is routinely performed on an Rtx®-5 (5%diphenyl/ 95% dimethyl polysiloxane) column. The Rtx®-65 is an excellent confirmational column to the Rtx®-5 for the analysis of EPA Method 604 as shown in Figure 2. The Rtx®-65 produces a different elution order for 4 of the eleven phenols and the analysis time is less than 23 minutes. The inertness of both columns is demonstrated by the excellent response of the compounds such as 2,4-dinitrophenol and pentachlorophenol, even at low concentrations.

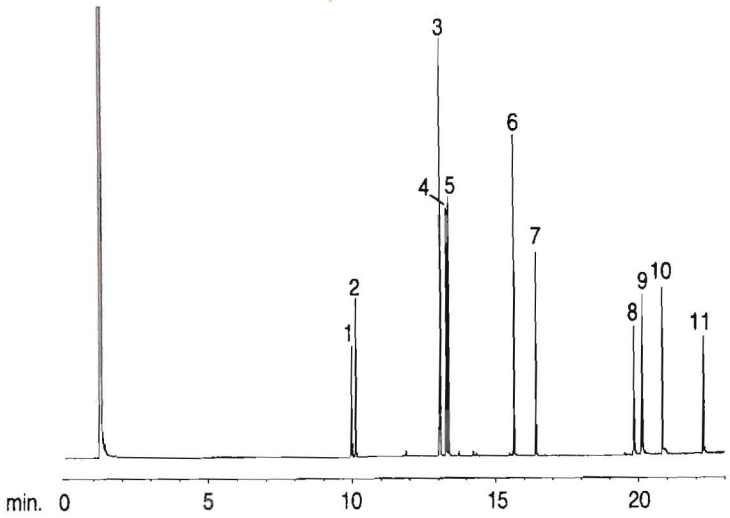
FAMEs

Because the consumption of large amounts of saturated fats has been linked to heart disease and cancer, accurate fatty acid analysis of food products is extremely important. Fatty acids are frequently analyzed in their methylated form to increase sample volatility, improve peak shape, and provide more accurate chromatographic data. Fatty Acid Methyl Esters (FAMEs) are commonly analyzed on polar stationary phases such as the Stabilwax® (polyethylene glycol or PEG) or the Rtx®-2330 (90%biscyanopropyl/ 10% cyanopropylphenyl polysiloxane). However, both of these stationary phases suffer from limited thermal stability and short column lifetimes. The Rtx®-65 is an excellent column choice for FAME analysis as shown in Figure 3. The Rtx®-65 provides resolution of the C14 to C24 fatty acids in canola oil in under 12 minutes and elutes FAMEs according to equivalent chain length similarly to the Stabilwax® column.

Triglycerides

The Rtx®-65TG is the same polymer as the Rtx®-65 but is designed specifically for triglyceride analysis. The Rtx®-65TG is selective in resolving triglycerides according to degree of unsaturation, as well as carbon number and has a maximum operating temperature of 370°C. The Rtx®-65TG is available in both 15 and 30 meter lengths in 0.25, 0.32, and 0.53mm IDs with a 0.10µm film thickness. Rtx®-65TGs are specially tested with a temperature programmed triglyceride test mixture and are guaranteed for low column bleed and high efficiency. Separation by degree of unsaturation as well as carbon number can be achieved in 32 minutes with minimal column bleed at 365°C.

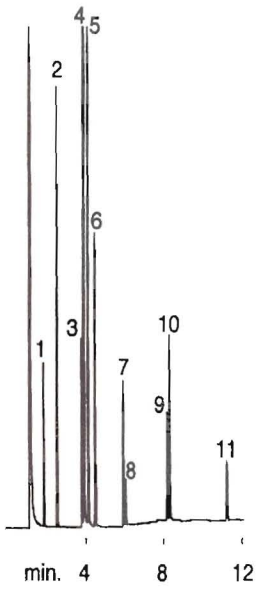
Figure 2 - The Rtx®-65 provides a different elution order than the Rtx®-5 or Rtx®-200 for EPA Method 604 Phenols.



1. 2-chlorophenol
2. phenol
3. 2,4-dimethylphenol
4. 2-nitrophenol
5. 2,4-dichlorophenol
6. 4-chloro-3-methylphenol
7. 2,4,6-trichlorophenol
8. 2,4-dinitrophenol
9. 4-nitrophenol
10. 2-methyl-4,6-dinitrophenol
11. pentachlorophenol

30m, 0.25mm ID, 0.25µm Rtx®-65 (cat.# 17023)
1µl split injection of 604 phenols
on-column concentration=50ng/µl
Oven temp.: 40°C (hold 4 min.) to 250°C @ 10°C/min.
Inj./det. temp.: 300°C/300°C **Detector:** FID
Carrier gas: hydrogen **Linear velocity:** 40cm/sec. set @ 40°C
FID sensitivity: 1.28 x 10^{-10} AFS **Split ratio:** 40:1

Figure 3 - Resolve saturated fatty acid methyl esters according to equivalent chain length on the Rtx®-65.



1. C14:0
2. C16:0
3. C18:0
4. C18:1
5. C18:2
6. C18:3
7. C20:0
8. C20:1
9. C22:0
10. C22:1
11. C24:0

30m, 0.25mm ID, 0.25µm Rtx®-65 (cat.# 17023)
0.5µl split injection of FAMEs (Canola Oil)
Oven temp.: 225°C (hold 5 min.) to 250°C @ 10°C/min. (hold 10 min.)
Inj. & det. temp.: 250°C **Detector:** FID
Carrier gas: hydrogen **Linear velocity:** 40cm/sec. set @ 225°C
FID sensitivity: 4 x 10^{-11} AFS

The Rtx®-65 column line is made with an intermediate polarity phenyl methyl stationary phase that is ideal for many types of analyses. Because of the high thermal stability, Rtx®-65 columns offer an excellent alternative to the more polar stationary phases such as the polyethylene glycols and bis-cyanopropyl phenyls. The Rtx®-65TG is ideal for triglyceride analysis providing separation of fatty acids by degree of unsaturation as well as carbon number. Low column bleed and excellent column efficiency are also traits of this new stationary phase.

Product Listing

Use Rtx®-65 columns for general purpose separations like phenols and fatty acids.

Rtx®-65 (Fused Silica)		(Crossbond® 65% diphenyl-35% dimethyl polysiloxane)	
	df (µm)	15-meter	30-meter
0.25mm ID	0.25	17020	17023
	0.50	17035	17038
	1.00	17050	17053
0.32mm ID	0.25	17021	17024
	0.50	17036	17039
	1.00	17051	17054
0.53mm ID	0.25	17022	17025
	0.50	17037	17040
	1.00	17052	17055

Use Rtx®-65TG columns for separating triglycerides requiring high column temperatures for elution.

Rtx®-65TG (Fused Silica)		(Crossbond® 65% diphenyl-35% dimethyl polysiloxane)	
ID	df (µm)	15-meter	30-meter
0.25mm	0.10	17005	17008
0.32mm	0.10	17006	17009
0.53mm	0.10	17007	17010

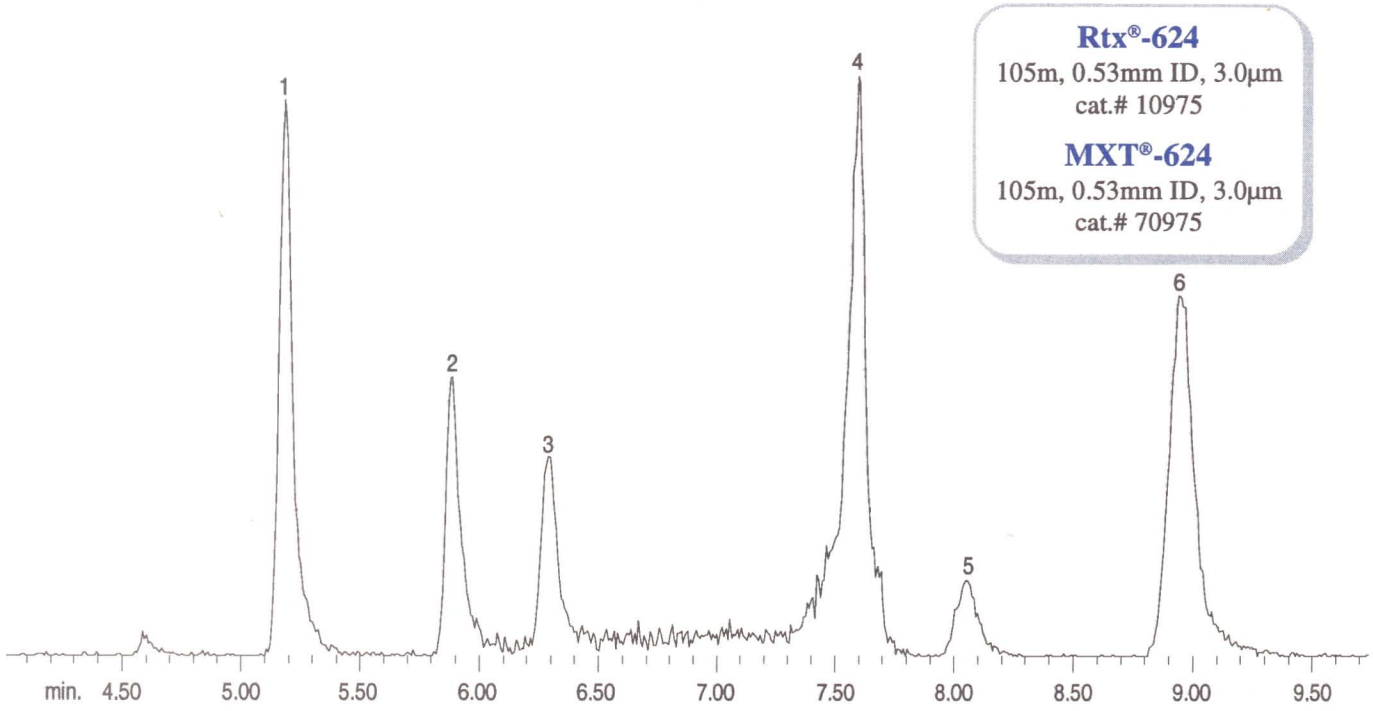
Rtx®-624 Capillary Column Meets New EPA CLP Resolution Requirements for VOA Gases

The February 1994 revision of the Contract Lab Program (CLP) Statement of Work for Volatile Organic Analysis includes a change in requirements for the separation of volatile gases. The new criteria reads as follows: *"For capillary columns, if the gaseous compounds chloromethane, bromomethane, vinyl chloride, and chloroethane fail to exhibit narrow, symmetrical peak shapes, are not separated from the solvent front, or are not resolved greater than 90% from each other, as evidenced by the RIC, then a sub-ambient oven controller must be used, and the initial temperature must be less than or equal to 10°C."*

Restek's 105m, 0.53mm ID, 3.0µm Rtx®-624 fused silica and MXT®-624 metal capillary columns meet this resolution requirement without the need for sub-ambient cooling. The Rtx®-624 stationary phase was specially designed for the separation of volatile organic compounds. Figure 1 shows the typical separation of the first six gases obtained with the Rtx®-624 column. All six compounds are baseline resolved using an initial starting temperature of 35°C.

The resolution of these volatile gases can be affected by the desorb flow rate, the trap type, and the dead volume between the purge & trap system and the column. A desorb flow of 8 to 10ml/min. produces a narrow sample band which results in improved separation. The VOCARB™ 3000 trap produced the best resolution when compared to other commonly used traps. The dead volume between the purge & trap system must be minimized to ensure good peak symmetry. The use of a low volume injector or a narrow bore inlet sleeve installed into a standard injector will result in better resolution of the volatile gases. If these three recommendations are followed, the new CLP resolution criteria can be easily met using the Rtx®-624 column.

Figure 1 - The 105-meter Rtx®-624 column completely resolves the VOA gases to meet the latest revision of the CLP SOW 2/94.



105m, 0.53mm ID, 3.0µm Rtx®-624 (cat.# 10975)

Oven temp.:	35°C (hold 8 min.) to 220°C @ 8°C/min.
Inj./Det. temp.:	100°C/280°C
Detector:	HP 5971MSD
Scan range:	35-260 AMU
Purge & Trap:	Tekmar 3000 interfaced to the GC system using a Low Volume Injector
Trap:	VOCARB™ 3000
Purge:	11 min.
Trap pressure control:	4psi
Desorb preheat:	245°C
Desorb temp.:	250°C
Desorb time:	2 min.
Desorb flow rate:	10cc/min.

Peak Identifications

1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane

Pro ezGC™ Retention Index Libraries

Three New Libraries are Available

Restek's retention index libraries and *Pro ezGC™* software can be used to select the most appropriate column and optimize chromatographic methods without making a single injection. Restek has added three new libraries and updated two existing libraries to increase the versatility of this method development tool. Analysts can choose from a wide range of environmental, pharmaceutical, clinical/forensic, solvent & chemical, and food & flavor components on common stationary phases. The libraries have been generated using Restek stationary phases, but for modeling purposes, the data closely simulates the performance of other commercially available phases (DB™-1, SPB-1, DB™-5, SPB-5, etc.). Call your local distributor for a list of components or more information on using *Pro ezGC™* and retention index libraries to optimize your chromatographic analyses.

Environmental - Base, Neutral and Acid Extractables: The analysis of BNAs is one of the most common GC/MS applications performed by environmental laboratories. This library contains 96 semi-volatile retention indices for EPA methods 525, 625, and 8270. These indices were determined using the Restek Rtx®-5/XTI®-5 stationary phase.
cat.# 21457

Environmental - Pesticides/Herbicides Part 2: This library contains a collection of 30 organophosphorous and 53 nitrogen containing pesticides from EPA Methods 507, 614, 619, 1618, and 8141A. Thermodynamic retention indices are provided using the Rtx®-5, -35, and -1701 phases. Part 2 is designed to compliment the chlorinated pesticides and phenoxy acid herbicides offered in Restek's Pesticides/Herbicides Part 1.
cat.# 21458

Solvents and Chemicals Part 2: This new library contains over 120 aromatics, esters, and ethers and is designed to compliment Solvents and Chemicals, Part 1 containing alcohols, aldehydes, and ketones. Retention indices are provided for the Rtx®-1, Rtx®-624, and Stabilwax® stationary phases.
cat.# 21459

Food and Flavor Volatiles Rev. 2.0: This updated library contains 150 new indices for alcohols, esters and ketones in addition to terpenes and other compounds originally offered in Rev. 1.0. In total there are now 300 retention indices for compounds found in food and flavor analyses, calculated on the Rtx®-1 and Stabilwax® stationary phases.
cat.# 21451

Drugs and Pharmaceuticals Rev. 2.0: This new update adds Rtx®-1 and Rtx®-35 stationary phases to the existing Rtx®-5, -50, and -200 stationary phases. The expanded library now contains retention indices for over 100 drugs and pharmaceuticals frequently analyzed by forensic, clinical, and drug testing laboratories.
cat.# 21453

Other Retention Index Libraries Available:

Fatty Acid Methyl Ester (FAME): cat.# 21455

Environmental - Pesticides/Herbicides (Part 1): cat.# 21456

Environmental - PCBs: cat.# 21454

Environmental - Volatiles: cat.# 21452

Solvents and Chemicals (Part 1): cat.# 21450

Software:

***Pro ezGC™* Software ver. 1.5: cat.# 21481**

***Pro ezGC™* ver. 1.0 to *Pro ezGC™* ver. 1.5: cat.# 21485**

***ezGC™* ver. 1.0 or 1.5 upgrade to *Pro ezGC™* ver. 1.5: cat.# 21482**

***ezGC™* software ver. 1.5: cat.# 21480**

***ezGC™* ver. 1.0 to *ezGC™* ver. 1.5: cat.# 21483**

ezGC™ and Pro ezGC™ Method Development Software

Hints for the Capillary Chromatographer

Using Electrolytic Conductivity Detectors

The electrolytic conductivity detector (ELCD) was developed in the early 1960's for the detection of organics in aqueous solutions. The ELCD is a highly selective, destructive detector for organic molecules containing fluorine, chlorine, bromine, nitrogen, and sulfur. It can be operated in three different modes: halogen, nitrogen, or sulfur. The ELCD is extensively used in the environmental field for the analysis of halogenated organics compounds such as those monitored in EPA Methods 502.1, 502.2, 601, 602, 8010, and 8021. It can also be used for various other environmental applications where compounds containing nitrogen, sulfur, or chlorine are of interest, such as organochlorine pesticides, PCBs, organophosphorus pesticides, and nitrosamines. It is also commonly used for pharmaceutical samples.

Detector Design and Operation

The ELCD operates as an electrical conductivity measuring device. However, it is the chemical aspect of the ELCD that provides the basis for its selectivity. The ELCD system consists of four main areas: reactor, conductivity cell, solvent, and electronic detection system.

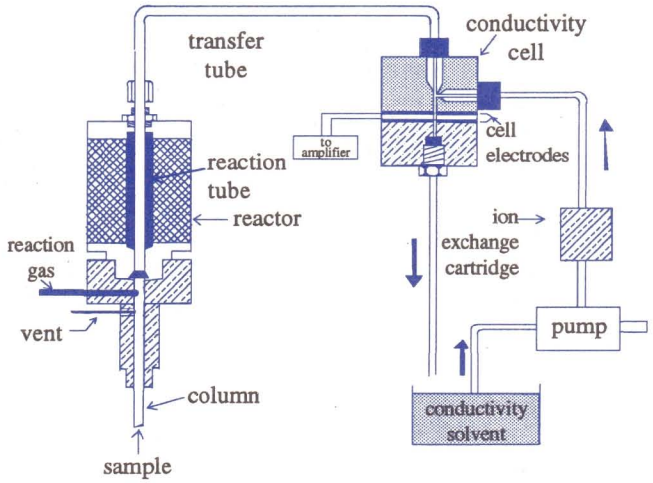
Reactor tube

The reactor is the bridge between the capillary column and the conductivity cell. It consists of the detector base, reaction tube, and heating element. Organic compounds eluting from the capillary column, enter the detector base, combine with reaction gas, and proceed through a high temperature reaction tube usually made of nickel or fused silica. In the reaction tube, most of the compounds are pyrolyzed and, with the presence of an active gas such as hydrogen or oxygen, chemical reactions will occur. In the sulfur mode, the sample components are oxidized using O_2 reaction gas to form SO_2. In the halogen and nitrogen modes, the sample components are reduced using hydrogen as a reaction gas to form HCl, HBr, HF, or NH_3. The reaction tube acts as a catalyst to help speed up the reaction.

Conductivity cell

The reacted sample is then swept through a Teflon® transfer line into the conductivity cell. In the conductivity cell, species formed in the reaction tube are dissolved and ionized in a deionized conductivity solvent flowing through the conductivity cell. Different solvents are used depending on the reaction mode. The change in the conductivity, caused by the reacted sample, is measured in the conductivity cell. Any species which is ionized during dissolution gives increased conductivity to the cell.

Figure 1 - Understanding the basic parts and operation of an ELCD enhances an analyst's ability to properly use the detector.



Solvent/ Resin Beds

In order to obtain a good response from the conductivity cell, the solvent flow and pH must be optimized. The sensitivity of the detector is inversely proportional to flow rate. Therefore, higher flow rates can be used where sensitivity is of little concern. The pH of the solvent is controlled by passing it through an ion exchange resin located in the solvent reservoir bottle. The proper resin mixture will provide the correct pH for the solvent. The halogen and sulfur modes are acidic and require an acidic solvent, whereas the nitrogen mode is basic and requires a basic solvent.

Detection Modes

Halogen compounds

The most common use of the ELCD is for the detection of volatile halocarbons in water. The ELCD reduces halogenated compounds in a nickel reaction tube (850-1000°C) to haloacids by mixing them with hydrogen reaction gas. Non-halogenated hydrocarbons are reduced to methane which is non-ionic. The haloacids are dissolved in n-propanol and the change in solvent conductivity is measured at the cell.

Sulfur compounds

In the sulfur mode, air is used as a reaction gas. Sulfur compounds are oxidized in a nickel reaction tube (800°C) producing SO_2 and/or SO_3. Hydrocarbons in the sample are

oxidized to form CO or CO_2. Pure methanol or a methanol/water mixture is the preferred solvent for this mode because it allows ionization of the sulfur compounds but minimizes the formation of CO_2. To eliminate the interference of haloacids from halogenated compounds in the sample, a scrubber is positioned after the reactor, but prior to the conductivity cell. The scrubber is made of stainless steel or copper tubing with several strands of silver wire positioned inside the tubing. The silver complexes with the haloacids and removes them from the sample stream.

Nitrogen compounds

The nitrogen mode reduces nitrogen containing compounds using hydrogen reaction gas in a nickel reaction tube (800°C) to form NH_3. The NH_3 will not completely ionize unless it is dissolved in an aqueous solution. Therefore, water containing a small amount of an organic solvent is recommended for greater sensitivity and better performance. A scrubber containing quartz thread is used for the nitrogen mode to remove other components from the sample.

Operating Hints

ELCD performance depends on the reactor, conductivity cell, and the solvent system. Because the reaction tube is the major part of the reactor, the tube should be replaced routinely. Hydrocarbons and column bleed can coat the inside of the reaction tube and decrease its catalytic activity. A drop in sensitivity, especially of brominated compounds, is a good indication the reaction tube needs to be replaced. Other factors, like baseline instability and ghost peaks, are also an indication the reaction tube may be fouled and requires replacement. A solvent vent, controlled by a solenoid valve, is located between the GC column and the reaction tube. This valve allows venting of the solvent to prevent premature fouling of the reaction tube. It may be necessary to condition a new reaction tube after replacement. Conditioning the tube with the reaction gas (H_2) flowing for 24 hours is recommended. Cooling the reactor before removing the capillary column from the detector is critical. If room air is allowed into the detector while the reactor is still hot, the nickel reaction tube will oxidize. An oxidized nickel reaction tube can result in sensitivity loss and tailing peaks.

The internal volume of the electrolytic conductivity cell is also important in determining ELCD performance. Older ELCDs have large cell volumes that were developed for use with packed columns operated at high flow rates. If these older detectors are used with capillary columns at low flow rates, excessive peak tailing will occur. Newer ELCDs have much smaller conductivity cells which significantly reduce peak tailing, even when operated at lower flow rates.

The Teflon transfer line, located between the reactor and the conductivity cell, requires cleaning and replacement depending on its usage. Clean the transfer line by rinsing with methanol or, for a more thorough cleaning, rinse with a 10% solution of HCl, followed by a methanol rinse. Dry the tubing before reinstalling it. With the time and effort required to clean the transfer line, it may be more cost effective to simply replace it.

Table I - Troubleshooting Hints for ELCDs

Symptom	Remedy
Noisy baseline	Clean transfer line from reactor to cell. Replace reaction tube. Use pure carrier gas and filters. Replace quartz insert.
Peak tailing	Clean conductivity cell/backflush. Increase reactor temperature. Replace reaction tube. Clean transfer line. Replace or cut 10cm off the detector end of the column.
Low response	Replace reaction tube. Clean or replace transfer line. Use correct solvent and replace if necessary. Optimize detector parameters.
High background	Incompatible column phase (F,N) Contaminated gases. Incorrect column installation. Condition column. Condition reaction tube.

Always use high purity solvents (HPLC grade for halogen mode) in the ELCD. The pH and background conductivity of the solvent are maintained by circulation through an ion-exchange resin. It is recommended to change the resin every six months. Problems often associated with the solvent reservoir may be sudden low or negative response, or baseline instability. It is important to maintain the proper solvent pH in the nitrogen mode to avoid the presence of negative peaks. The solvent may be slightly acidic due to trace amounts of CO_2. This can cause neutralization of low levels of NH_3, resulting in negative peaks. One way to avoid this problem is to totally exclude CO_2 from the solvent system by using nonpermeable tubing.

Table I lists some of the common problems experienced with ELCDs and troubleshooting hints.

Electrolytic conductivity detectors are excellent for environmental and pharmaceutical analyses due to their highly selective nature. Because of the selectivity of the ELCD, sample cleanup procedures do not have to be as stringent as with other detectors where interference can be a problem. However, because the ELCD is a more complex detector, frequent maintenance and optimization of detector flow rates is required. Attention to the basic operating hints, as outlined above, will result in a highly sensitive, reliable detection system.

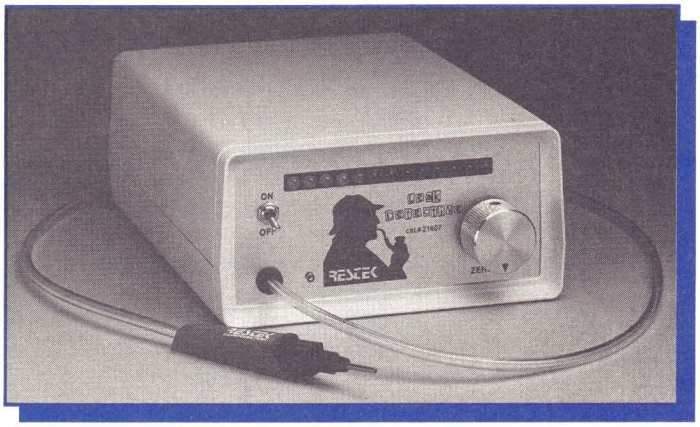
References:

1. Hill, Herbert and Dennis McMinn, ed., *Detectors for Capillary Chromatography*, John Wiley & Sons, New York, 1992.
2. Buffington, Rosemary and Michael K. Wilson, *Detectors for Gas Chromatography - A Practical Primer*, Hewlett-Packard Co., Avondale, PA, 1991.

Peak Performers

The affordable solution for GC leak detection...

Restek's Leak Detective™



- Detects minute leaks not possible with liquid leak detectors.
- Compact, lightweight, hand-held design.
- Lowest cost thermal conductivity leak detector available.
- Contamination-free leak detection.
- Battery or line operated.
- Detects leaks of helium or hydrogen at ≥20µl/min. or ≥200ppm

Restek's new Leak Detective™ is the affordable solution for GC leak detection. Leaks can increase detector noise, cause baseline instability, waste carrier gas, and shorten column lifetimes. The Leak Detective™ allows detection of minute gas leaks which may go undetected by liquid leak detectors.

The compact design of the Leak Detective™ ensures comfortable hand-held operation. Trace leaks of both helium and hydrogen* can be detected. Sensitivity is similar to other models on the market with detectability of helium or hydrogen and leak rates of 20µl/min. or an absolute concentration less than 200ppm Leaks are indicated by an audible alarm, as well as an LED readout. Two 9-volt batteries (included) provide 10-12 hours of continuous operation, or the unit can be used with an AC adaptor (included).

Restek Leak Detective™
cat.# 21607 each

**not designed for use in explosive atmospheres*

Channeltron® 5778 Electron Multiplier

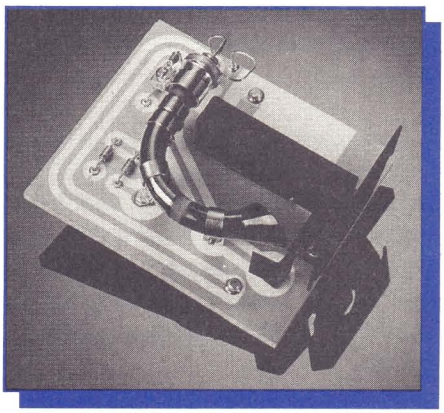
for the HP 5971A and 5972A MSD

The new 5778 provides these benefits over the multipliers originally supplied with HP 5871A and 5972A MSDs:

- 25-50% increase in sensitivity
- 2-5X increase in dynamic range
- Double the lifetime
- Easy, self-aligning installation

The new Channeltron® 5778 Electron Multiplier from Galileo offers increased performance for HP 5971A and 5972A MSDs and the new HP GCD. If your applications demand high sensitivity, extended linear dynamic range, and longer multiplier lifetime, the 5778 Electron Multiplier will meet these requirements.

The 5778 design increases sensitivity through superior signal collection and a reduction of unwanted noise. This sensitivity allows detection of sub picogram and femtogram levels from complex mixtures. The separate input and booster stages of the 5778 provide a linear response and dynamic range which



exceeds the limits of the instrument and of other available multipliers. The patented plug-in, ceramic board-mounted design allows easy installation and assures alignment of the critical ion optics.

Channeltron® Electron Multipliers are available for other mass selective detectors upon request. Please call your local distributor for more information.

Channeltron® 5778 Electron Multiplier: cat.# 21608 each

Inlet Seals with Both Large and Small Holes for HP 5890 GCs

Now Available in Two Different Sized Openings

The inlet seal at the base of the HP 5890 GC injection port comes into contact with the sample and must be changed frequently to prevent adsorption and/or breakdown of active compounds. Restek now offers inlet seals with two different sized openings: 1.2mm and 0.8mm. The 1.2mm inlet seal is recommended for use with Vespel®/Graphite ferrules or when installing two columns using a 2-hole ferrule. The 0.8mm inlet seal is recommended for use with graphite ferrules and single capillary column installations.

Inlet seals with the 0.8mm or 1.2mm opening are available in three styles: stainless steel, Silcosteel®, or gold plated. The gold surface offers better inertness and easier sealing than standard stainless steel. Restek's unique Silcosteel® process places a micron thin layer of fused silica and a deactivation layer over the stainless steel to provide inertness similar to a fused silica capillary column. Both the Silcosteel® and gold plated inlet seals reduce breakdown and adsorption of active compounds.

1.2mm Replacement Inlet Seals
cat# 20390, 2-pk.
cat.#20391, 10-pk.

1.2mm Gold Plated Inlet Seals
cat.#21305, 2-pk.
cat.#21306, 10-pk.

1.2mm Silcosteel® Treated Inlet Seals
cat.#21307, 2-pk.
cat.#21308, 10-pk.

0.8mm Replacement Inlet Seals
cat# 21315, 2-pk.
cat.# 21316, 10-pk.

0.8mm Gold Plated Inlet Seals
cat.# 21317, 2-pk.
cat.# 21318, 10-pk.

0.8mm Silcosteel® Treated Inlet Seals
cat.# 21319, 2-pk.
cat.# 21320, 10-pk.

PRICES SLASHED on XTI®-5 Columns

- Low bleed for GC/MS.
- Thermal stability to 360°C.
- Displays high response factors for active compounds.
- Guaranteed low bleed at maximum temperature.

The technology used to produce XTI®-5 columns increases the polymer stability by 20°C over our standard Rtx®-5 column, giving them the highest operating temperature of any 5% diphenyl/95% dimethyl polysiloxane capillary column available.

The XTI®-5 is inert to the most reactive environmental compounds, and it has the efficiency to resolve closely eluting isomers. Every XTI®-5 column is tested with a specially designed, temperature programmed environmental test mix to ensure that it meets strict performance requirements. In addition, each column is programmed to its maximum temperature and monitored for bleed.

100% Satisfaction Guarantee
We are confident that the XTI®-5 out-performs any competitor's column for environmental analyses or columns specially marketed for use with mass spectrometers. If the XTI®-5 does not consistently provide the highest response factors, lowest bleed, and best thermal stability for priority pollutant analyses, just contact our technical service department. We'll replace the column or give you a complete refund.

Call your local distributor for new price information.

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

The Restek Advantage is printed on recycled paper.