THE RESTEK ADVANTAGE

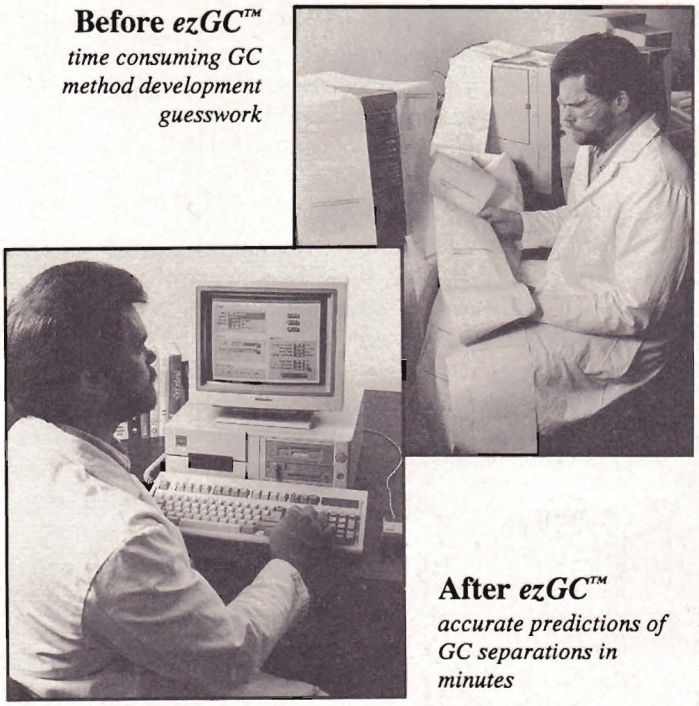
Sneak Preview! *ezGC™* Software

Simplifies GC Method Development

- Saves time and money by reducing analysis times and improving sample resolution.
- Automatically determines optimum temperature program rates and column flow rates.
- Works with constant flow, constant pressure, or electronic pressure/ flow programming.
- Visually demonstrates changes in resolution when the column parameters and operating conditions are changed.
- Easy to use, mouse driven software with built in help menus.
- Takes the guesswork out of capillary column selection.
- Easy to install and works on all DOS operating systems with 512K of free RAM.
- Costs about the same as a 30-meter column.

Did you ever work with a chromatographer who seems to know how to pick the best temperature program and flow conditions? After years and years of experience they seem to inherently know which GC parameters work best. They have learned how parameters such as temperature, flow, and distribution coefficients affect a separation. Why wait years? Use *ezGC™* and quickly become a master at capillary column selection and optimization.

Before *ezGC™*
time consuming GC method development guesswork



After *ezGC™*
accurate predictions of GC separations in minutes

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Even experienced chromatographers will benefit by using *ezGC™*. Restek's applications department was hard at work trying to optimize the temperature program rate for the 60 compounds in EPA Method 502.2. They tried 4, 10, 12, and 16°C/min., but there were so many compounds that new coelutions occurred at each temperature program ramp. The separations were so complex that they couldn't figure out whether faster or slower program rates were better. After several frustrating days of working on the project, they tried *ezGC™*. They entered the retention times into the *ezGC™* program and let the software do the optimization. *ezGC™* predicted 7.5°C/min. as the optimum temperature program rate and printed a simulated chromatogram illustrating the expected separations. They were impressed but still not convinced. Actual chromatograms were then generated at 7 and 8°C/min., but only 7.5°C gave the best separation, just as the program predicted. Now our applications department is so convinced of the power of *ezGC™* that they use it for all optimization work.

You can save time and money in your laboratory by using *ezGC™* to optimize all your analyses. If you have a simple analysis with no coelutions, you can use the software to predict the fastest temperature program and flow conditions while

maintaining baseline resolution (R≥1.5). And, if your sample contains compounds which may switch elution orders at the new optimized conditions, *ezGC™* will list the new elution order.

Did you ever wonder how your sample would look on a different film thickness? If you are using a 0.25µm film and you suspect that a 0.5µm film would improve resolution, use *ezGC™* to print a simulated chromatogram with the 0.5µm film. In fact, you can try any other film thickness and *ezGC™* will provide simulated chromatograms at optimized run conditions. How about a longer length or different inside diameter? Enter the desired column dimensions into the *ezGC™* program and it will provide a simulated chromatogram for visual examination. Now you don't have to waste your time or money buying experimental columns to optimize your analysis, *ezGC™* can do it for you.

How does *ezGC™* work?

In the past 20 years, several attempts have been made to predict retention and elution in gas chromatography. Initially, elution order was predicted by Kováts indices (1). However, Kováts indices are restricted to isothermal conditions. With the increasing use of temperature programming, Kováts indices were not applicable in many situations. A modified retention index equation was developed by Van den Dool and Kratz[2] that incorporated Kováts indices into temperature programming. This modified retention index works relatively well, as was demonstrated in *The Restek Advantage* (January 1992). However, neither the Kováts or Van den Dool and Kratz methods account for changes in carrier gas viscosity, linear velocity, film thickness, etc. Recently, advances have been made in developing a more sophisticated method to predict GC behavior. Several researchers, Dose[3]; Curvers and Rijks[4]; and Snow and McNair[5] have contributed to a method for calculating temperature programmed or isothermal retention from thermodynamic parameters. The distribution coefficient K_D is related to the Gibbs free energy of gases in solution by the following equation:

$$\Delta G = RT \ln K_D \text{ and since } \Delta G = \Delta H - T\Delta S$$

substituting $K_D = k * \beta$, the following equation can be derived:

$$\ln k = \left(\frac{\Delta H}{R}\right) * \left(\frac{1}{T}\right) + \ln\left(\frac{a}{\beta}\right)$$

where

$$a = \left(\frac{\Delta S}{R}\right)$$

This new equation is in the form of $y = mx + b$ where $\frac{\Delta H}{R}$ is the slope of the line and the quantity $\ln(a/\beta)$ is the y intercept. The *ezGC™* software incorporates these fundamental concepts into a computer algorithm that makes it possible to accurately predict GC retention times routinely to within 2%.

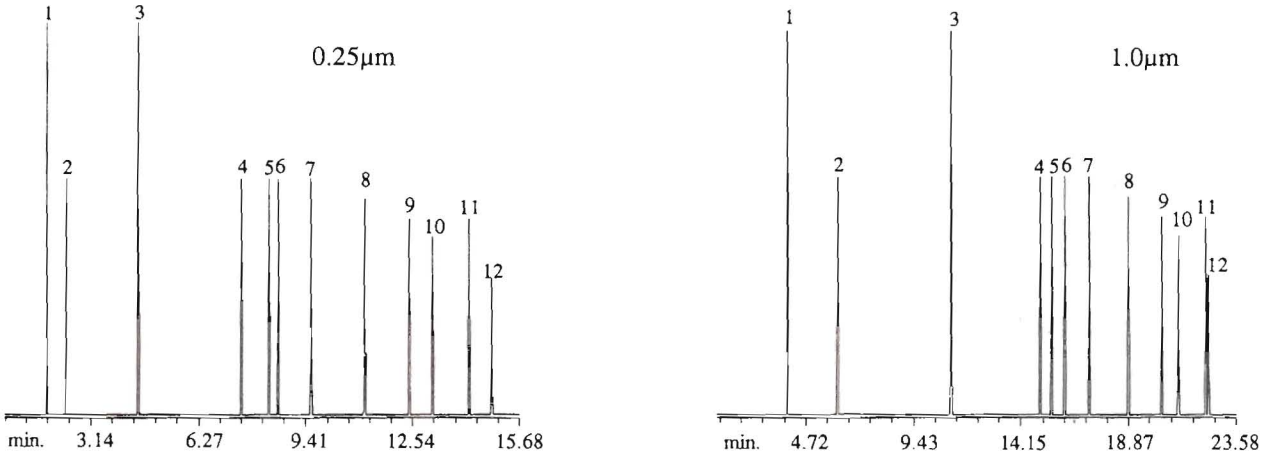
How hard is it to use *ezGC™*?

By following a few simple steps, optimum operating conditions can easily be predicted for any analysis. To utilize *ezGC™*, simply obtain an accurate dead time and run your sample at fast and slow temperature program ramps. Enter the retention times for both runs in the program and you are ready to try new temperature program rates, flow rates, column IDs, film thicknesses, or column lengths. An on-line help manual is available at any time to answer questions, and in those rare cases when you need extra help, experienced Restek technical service chemists will be available to assist you with your more detailed questions.

Ways to generate optimum conditions

Optimum temperature programmed run conditions can be generated two ways. In one case, a specific set of GC conditions is entered and under those conditions, the *ezGC™* program will predict the retention times of the components.

Figure 1 - *ezGC™* quickly predicts actual peak resolution when increasing the film thickness from 0.25 to 1.0µm when using the same temperature program.



30m, 0.25mm ID Rtx®-5

Oven temp.: 35°C (hold 5 min.) @4°C/min. to 280°C **Linear velocity:** 43.0cm/sec. @ 35°C

Table I - Comparison of Experimental vs. Calculated Retention Times

#	Component Name	Exp. tR (min.)	Calc. tR (min.)	Exp. Calc. Error (min.)	(Exp.-Calc.) /Exp. % Error (min.)
1	hexane	3.891	3.900	-0.009	-0.2
2	benzene	6.032	6.117	-0.085	-1.4
3	toluene	11.001	11.076	-0.075	-0.7
4	chlorobenzene	15.002	14.991	0.011	0.1
5	ethylbenzene	15.500	15.495	0.005	0.0
6	*m*-xylene	16.184	16.059	0.125	0.8
7	styrene	17.395	17.129	0.266	1.5
8	isopropylbenzene	19.082	18.861	0.221	1.2
9	n-propylbenzene	20.517	20.345	0.172	0.8
10	1,3,5-trimethylbenzene	21.202	21.071	0.131	0.6
11	tert-butylbenzene	22.385	22.259	0.126	0.6
12	decane	22.501	22.364	0.137	0.6
				Average error	0.7

Predicted results can be viewed in either a table format or a computer simulated chromatogram. Figure 1 shows simulated chromatograms demonstrating how the analysis would look if the stationary phase film thickness was increased from 0.25 to 1.0µm with the same program conditions. The 30m, 1.0µm film thickness increases the analysis times from approximately 14 to 22 minutes. Figure 2 shows the predicted optimum temperature program ramp for the 5m, 1.0µm column to maximize resolution and minimize analysis times. Baseline resolution is obtained in under 6 minutes with the 5m column.

Another way to generate the optimum conditions is by entering a range of desirable temperature program conditions into the program. The optimum conditions, yielding the shortest analysis time with the best resolution, will be listed first with other possibilities listed sequentially. Computing time varies with the number of permutations requested.*

Quickly compare differences in analysis and resolution changes when varying linear velocity, ID, film thickness, length, or theoretical plates

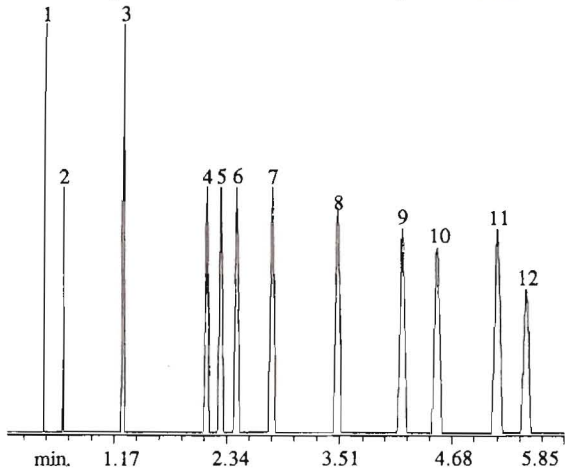
ezGC™ permits a visual comparison of analysis times and resolution when column parameters such as linear velocity (including electronic pressure or flow programming), column diameter, theoretical plates, film thickness, and/or the column length are varied. Table I shows the predicted vs. actual retention times for a 1.0µm Rtx®-5 using data generated on a 0.25µm capillary column. The absolute error is approximately 2%.

ezGC™ simplifies method development

ezGC™ greatly reduces the workload of GC method development. It also insures the best resolution and analysis time conditions for existing methods. This versatile program allows any parameter or combination of parameters to be changed and quickly viewed in either a table format or simulated chromatogram. *ezGC™* can be installed on any IBM PC or compatible system with a hard drive and 512K of free memory.

** A 386SX-25 without a coprocessor was able to evaluate 350 temperature programs for 12 components in under 1 minute.*

Figure 2 - *ezGC™* predicts the optimum resolution and fastest analysis times with a 5.0m, 1.0µm column.

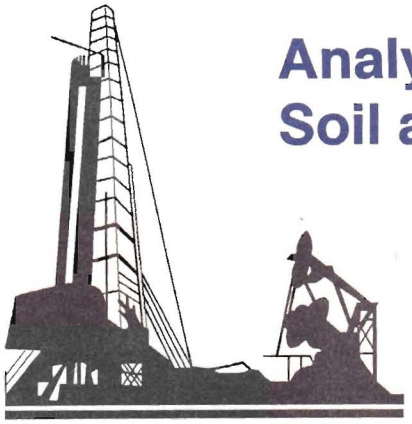


5.0m, 0.25mm ID, 1.0µm Rtx®-5
Oven temp.: 50°C @4°C/min. to 80°C
Linear velocity: 40.9cm/sec. @ 60°C

After reading about *ezGC™*, you may ask, "How could method development be easier?" The answer is, by having Restek generate thousands of thermodynamic retention index libraries on volatile organics, industrial solvents, pharmaceutical compounds, and flavors/fragrances using a wide variety of bonded phases. Restek has dedicated a large portion of our application chemists' time towards generating extensive libraries that interface to *ezGC™*. See the July 1993 issue of *The Restek Advantage* for information on Restek's thermodynamic retention indice libraries. ■

References

(1) Kováts, E., Giddings, J.C., and Keller, R.A., *Advances in Chromatography*, Volume 1, Chapter 7. New York: Marcel Dekker (1965).
(2) Van den Dool, H. and Kratz, P.D., *Journal of Chromatography*, Volume 11, pp.463-471, (1963).
(3) Dose, E.V., *Anal. Chem.*, 1987, 59, 2414-2419.
(4) Curvers, J., Rijks, J., Cramers, C., Knauss, K., Larson, P., *HRC & CC*, Vol. 8, Sept. 1985.
(5) Snow, N.H. and McNair, H.M., *J. of Chrom. Sci.*, Vol. 30, July 1992.



Analysis of Gasoline Range Organics in Soil and Water

Individual states have adopted analytical methods for measuring hydrocarbon contamination in soil and water (1) resulting from leaking underground storage tanks (LUST). The following article addresses some of the more common questions regarding the determination of benzene, toluene, ethylbenzene and total xylenes (BTEX), and total petroleum hydrocarbons (TPH) from gasoline range organics (GRO). Future articles will address the analysis of diesel range organics (DRO) and heavy petroleum products such as lubrication oil.

In general, GRO methods for analysis of TPH and BTEX use a purge and trap sampler, a wide bore capillary column, and both photo ionization (PID) and flame ionization (FID) detectors. The purge and trap sampler (2) is used to extract and concentrate the more volatile gasoline components from water and soil (methanol extract) prior to introduction into the gas chromatograph. The sampling procedure for water is as specified in EPA Method 602 (3). For soils, a methanol extract is added to the purge vessel containing a volume of water as specified in EPA Method 8020 (4). The FID responds to all hydrocarbon species in the complex gasoline sample and is used to detect the total volatile hydrocarbons. A PID, when operated with a 10.0 eV lamp, yields more specific response to aromatic and other unsaturated hydrocarbons present in gasoline and is used to quantitate BTEX. A wide range of columns can be used for GRO analysis, depending upon the requirements specified in each state's analytical procedure. In general, the column, operating under the conditions of the method, must meet some minimum requirements for retention and resolution.

Gasoline is a complex mixture, containing in excess of 400 individual hydrocarbon compounds; so if BTEX is to be determined, the column must resolve these aromatics. Since xylenes are reported as a total, it is not necessary to separate the ortho, meta and para isomers. The resolution between ethyl benzene and *m*-, *p*-xylene is typically the most difficult separation to obtain. Since the same chromatographic method is normally used for both water and soils, the column must also resolve gasoline from the methanol solvent peak. States may differ on which hydrocarbon is used to define the beginning and end of the gasoline compounds to be measured, so the requirements of the column will vary. The 105-meter Rtx®-502.2 column is a good choice for most methods because it resolves 3-methyl pentane from methanol without subambient oven temperatures and provides baseline resolution of ethyl benzene from *m*-, *p*-xylene.

Determining gasoline retention range and calibrating response

Hydrocarbon calibration standards serve two purposes in TPH/BTEX analysis. Since the reporting of TPH requires the summation of the total gasoline area, the standard must contain the first and last components defining the retention time range. Individual states differ on the compounds defining the retention time range for gasoline. Figure 1 shows a chromatogram

Figure 1 - The GRO Mix can be used to establish the start/stop times of the gasoline range and to calibrate FID/PID detector response.

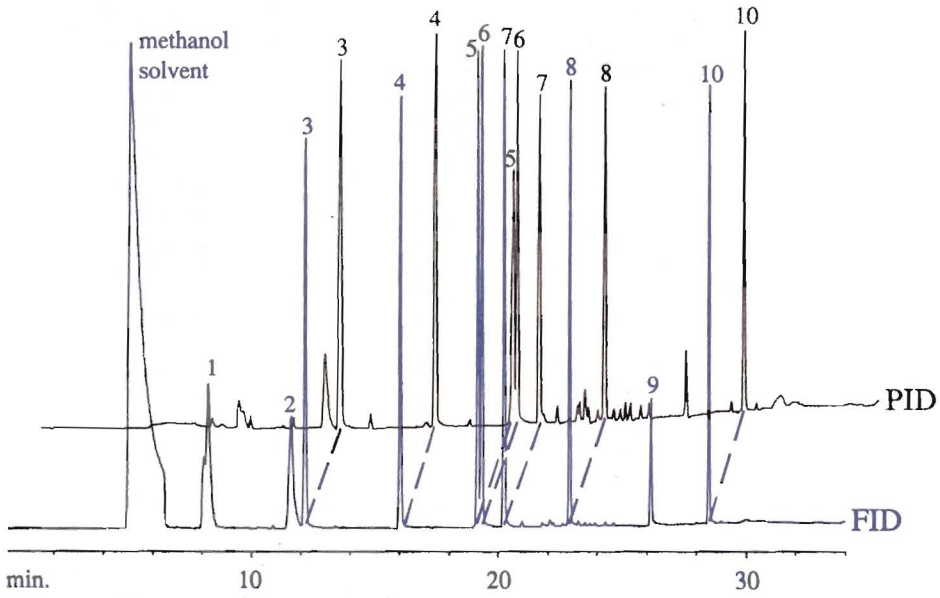
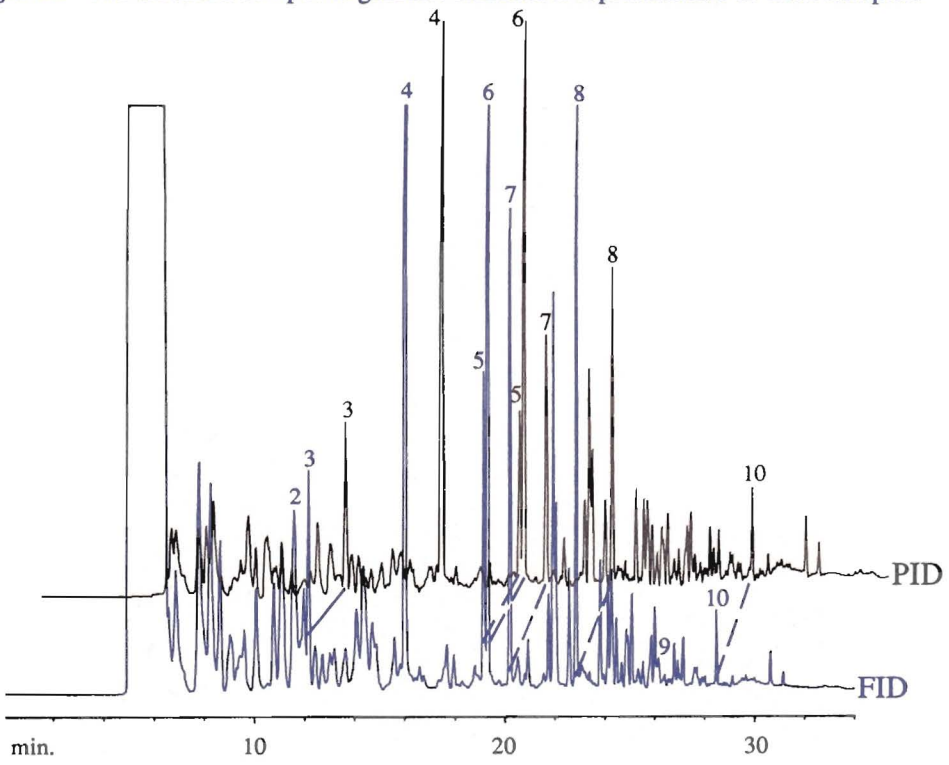


Figure 2 - The unleaded composite gasoline standard is representative of GRO samples.



Sample analysis and evaluating method performance

Once the retention time range and response factors are determined, it is good practice to perform an analysis of a spiked soil or water to determine the analyte recovery and method repeatability. For TPH, a typical gasoline such as the Restek composite gasoline can be used. To calculate BTEX recovery, an individual aromatic standard should be used. This is because the exact concentration of BTEX in the gasoline standard is not easily determined. The addition of an internal standard and surrogate to the samples prior to analysis will usually increase the precision of the results, especially for BTEX. Internal and surrogate standards that have been used successfully include α,α,α-triflourotoluene, 1-chloro-4-fluorobenzene, and 4-bromoflourobenzene.

of Restek's GRO Mix plus dodecane.* The second step is to calibrate the detector for the aromatic hydrocarbons (BTEX) and for the entire gasoline range (TPH). For BTEX, the calibration is straightforward, but for TPH there are two possible procedures. One procedure is to analyze a mixture of individual hydrocarbons covering the gasoline range (Figure 1) and calculate an average response factor from the response factors of each individual component. This calibration standard should be representative of the different types of hydrocarbons in gasoline. States recommending this method of calibration will specify the hydrocarbon components to be used. The other procedure for calibrating TPH response is to analyze a quantitative standard containing one or more gasolines. In theory, a composite should be more representative of the gasoline present in a wide range of samples to be analyzed. An example of a chromatogram generated from Restek's composite gasoline standard appears in Figure 2.

Avoiding some of the common pitfalls

The most common problem encountered in TPH/BTEX analysis is the presence of interfering compounds in the chromatographic analysis. Interferences can be caused by organic solvents present in the samples, background organic

Peak List and Run Conditions for Figures 1 - 2

	COMPOUNDS
1	3-methylpentane
2	2,2,4-trimethylpentane (isooctane)
3	benzene
4	toluene
5	ethylbenzene
6	*m*-xylene
7	*o*-xylene
8	1,2,4-trimethylbenzene
9	dodecane
10	naphtalene

Fig. 1) 105m, 0.53mm ID, 3.0µm Rtx®-502.2 (cat.# 10910)
Sample: GRO Mix (WISC) + dodecane
Concentration: 200ppb each in 5ml of H_2O

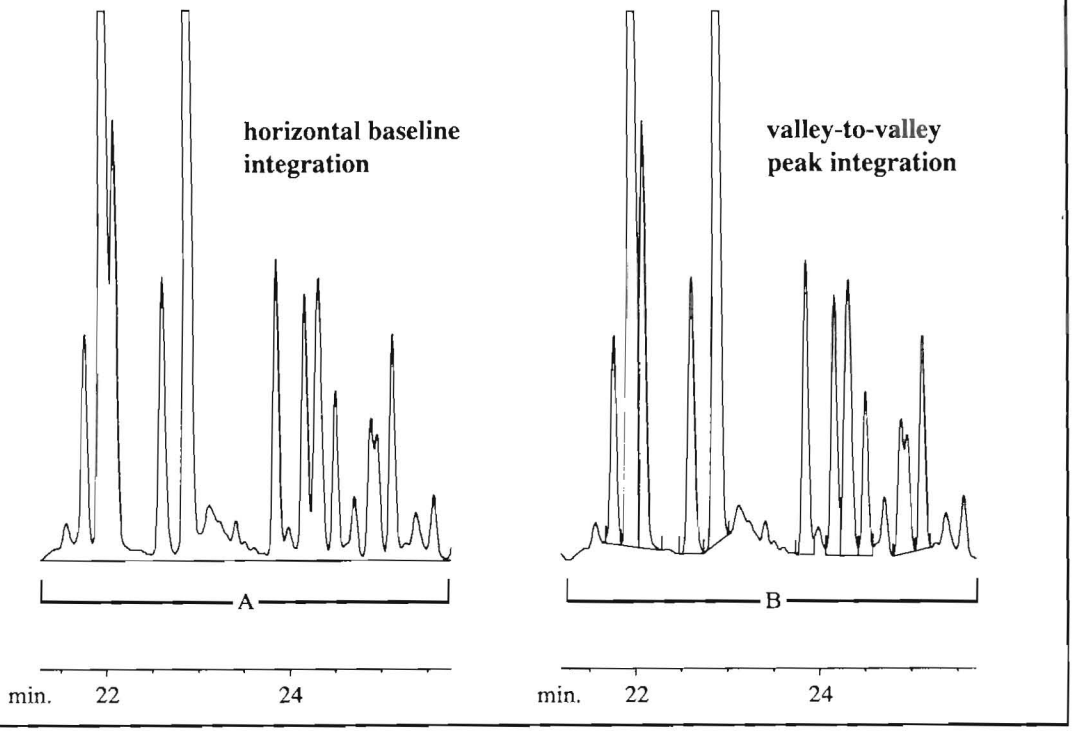
Fig. 2) 105m, 0.53mm ID, 3.0µm Rtx®-502.2 (cat.# 10910)
Sample: Unleaded Gasoline Composite Standard
Concentration: 5ppm in 5ml of H_2O

Oven temp.:	40°C (hold 1 min.) to 100°C @ 5°C/min., then to 240°C @ 8°C/min. (hold 8 min.)		
Inj. / det. temp.:	200°C/250°C		
Carrier gas:	helium (10cc/min.)		
FID sensitivity:	16 x 10^{-11} AFS		
Trap:	Tenax, Silica Gel, Charcoal		
Purge:	12 min. @ 40cc/min.		
Desorb preheat:	175°C	**Desorb temp.:**	180°C
Desorb time:	2 min.	**Desorb flow:**	10cc/min.

* *Some states specify dodecane as the end of gasoline.*

contamination, or carryover of hydrocarbons from previous chromatographic analyses. Each of these problems can result in reporting higher concentrations especially for TPH. To avoid contamination, prescreening the samples on a separate GC, prior to sample preparation is recommended. Overloading the instrument with hydrocarbon contaminants can be minimized by adjusting the sample amount, keeping it within the linear range of the method.

Figure 3 - Horizontal baseline integration provides best results for TPH analysis.



Another column problem encountered with this analysis is low TPH recoveries when response factor calibration is based upon a hydrocarbon component standard as opposed to a composite gasoline standard. A likely explanation for this is that the start and stop integration for gasoline is often well inside the gasoline range, depending upon the hydrocarbons used to set the range. Furthermore, low recoveries are often obtained due to errors in integrating the gasoline area. Figure 3 shows the difference between baselines obtained using horizontal baseline integration (A) and valley-to-valley peak integration (B) modes. The area resulting from the peak integration will give low recoveries because part of the gasoline area is excluded from the calculation. For best results with GRO samples, the baseline obtained should be determined at the beginning and end of the analysis, and a horizontal hold applied between these two points.

Although GRO methods differ between states, the basic procedures are similar. The capillary column frequently recommended for TPH and BTEX analysis is a 105m, 0.53mm ID, 3.0µm Rtx®-502.2. System calibration can be accomplished with either mixtures of individual hydrocarbons or composite gasoline standards. Analysts should refer to their specific methods for analytical and calibration procedures. ■

References

1) Tamlyn Oliver and Paul Kostecki, *Soils Magazine*, December 1992.
2) USEPA, *SW-846 Test Methods for Evaluating Solid Waste*, 3rd Edition; Method 5030, "Purge and Trap".
3) Federal Register 1984 Vol. 49, No. 209; USEPA Method 602 (Purgeable Aromatics).
4) USEPA, *SW-846 Test Methods for Evaluating Solid Waste*, 3rd Edition; Method 8020, "Aromatic Volatile Organics by Gas Chromatography".

Product Listing

Rtx®-502.2 105m, 0.53mm ID, 3.0µm cat.# 10910, $1200

Unleaded Gasoline Composite Standard
cat.# 30081, $25 each
cat.# 30081-500, $35 ea. w/data pack
cat.# 30181, $225 10pk. w/data pack

GRO Mix (WISC) cat# 30069, $25 each
cat.# 30069-500, $55 ea. w/data pack
cat.# 30169, $225 10pk. w/data pack

GRO Mix (EPA) cat.# 30065, $25 ea.
cat.# 30065-500, $55 ea. w/data pack
cat.# 30165, 10pk. w/data pack

1-chloro-4-fluorobenzene Standard
cat.# 30066, $25 each
cat.# 30066-500, $35 ea. w/data pack
cat.# 30166, $225 10pk. w/data pack

4-bromofluorobenzene Standard
cat.# 30067, $25 each
cat.# 30067-500, $35 ea. w/data pack
cat.# 30167, $225 10pk. w/data pack

α,α,α-trifluorotoluene Standard
cat.# 30068, $25 each
cat.# 30068-500, $35 ea. w/data pack
cat.# 30168, $225 10pk. w/data pack

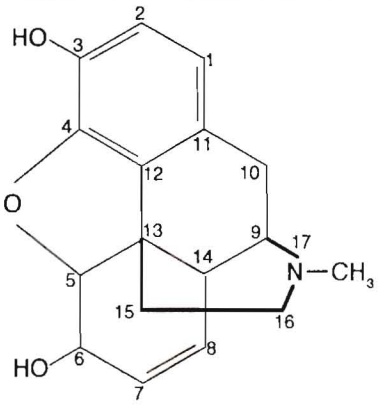
Additional calibration and internal standards/surrogate mixtures are available, including the modified Wisconsin PVOC/GRO Mix. Please call 800-356-1688 for information.

Clinical Corner

Opiate Analysis

Opiates or opioids are terms that classify a group of compounds with morphine-like actions. Their pharmacological properties include analgesia or pain relief, drowsiness and respiratory depression. Figure 1 shows the structure for morphine. Substitutions at the 3, 6, and 17 positions produce compounds with varying degrees of potency and pharmacological activity. The National Institute for Drug Abuse (NIDA) has targeted opiates as a class to be monitored in urine for detection of drug abuse. Testing guidelines have been established with a limit of detection of 0.3µg/ml for morphine. Screening of opiates is commonly done by using enzyme immunoassays. Enzyme immunoassays have the ability to cross react with a number of structurally similar opiates including codeine, hydromorphone, hydrocodone, levorphanol, and oxycodone. In order to differentiate between all of the possible substances being detected by enzyme immunoassay, confirmational analysis by GC/MS should be performed.

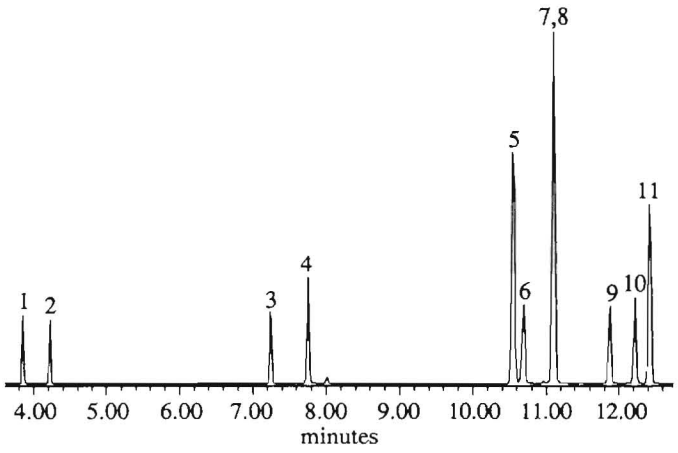
Figure 1 - Morphine structure



Chromatographic performance of the opiates is significantly affected by small changes in their chemical structure. The presence of hydroxyl groups at the 3 and 6 positions produce compounds that are more polar and reactive. Compounds with reactive hydroxyl groups in their chemical structure can suffer from adsorption and peak tailing, leading to diminished response in chromatographic systems that contain active sites. Sample preparation of sensitive compounds, like opiates, should take place in silanized glassware and samples should be stored in deactivated sample vials. Derivatization of reactive hydroxyl groups can improve chromatographic performance and detection limits and prevent sample loss on glassware and sample vials. Both trimethylsilyl and fluoroacyl derivatives of the opiates yield end products that are less polar and/or more volatile than the underivatized compound.

For this analysis, trimethylsilyl derivatives were prepared using BSTFA with 1% TMCS. Derivatizing the reactive hydroxyl group with a less polar trimethylsilyl group eliminates the tailing peaks commonly seen with compounds like morphine. Figure 2 shows the analysis of a selection of opiates on an Rtx®-5 column. Compounds that have been derivatized prior to analysis are designated as TMS in the peak list. The TMS derivatized opiates chromatograph well on a low polarity (Rtx®-5) column with good resolution and peak shape.

Figure 2 - Opiates analysis on an Rtx®-5 column.



30m, 0.25mm ID, 0.25µm Rtx™-5 (cat.# 10223)
2µl split injection of Opiates

Oven temp.: 200°C to 325°C @ 7°C/min.
Inj. temp.: 250°C
Det. type: HP MSD 5971A **Det. temp.:** 300°C
Carrier gas: helium **Linear velocity:** 30cm/sec. set @ 200°C
Split ratio: 50:1 **Ionization:** EI **Mode:** SIM

	COMPOUNDS	IONS MONITORED
1	meperidine	71, 246
2	alphaprodine	172, 187
3	methadone	72
4	levorphanol (TMS)	150, 270, 271, 328
5	codeine (TMS)	178, 196, 234, 371
6	hydrocodone	242, 299
7	morphine (TMS)	234, 429
8	hydromorphone (TMS)	356
9	oxycodone (TMS)	371, 386
10	oxymorphone (TMS)	444, 445
11	nalorphine (TMS)	414, 455

Sensitivity and specificity in confirming the presence of opiates in different samples can be enhanced by selectively choosing certain ions to monitor. Identification based upon the presence of distinctive, high mass ions is preferred, especially when analyzing derivatized compounds. Trimethylsilyl derivatives will add 72 amu for every hydroxyl group derivatized.

(**Clinical Corner** is continued on page 9.)

New! Rt-βDEXm™ Columns

Designed for the Separation of Optical Isomers

- Highly selective for the separation of enantiomers
- Inert and efficient
- Available in both 0.25 and 0.32mm ID
- Equivalent pricing to conventional liquid phase columns
- Individually tested with a chiral mix
- Permethylated β cyclodextrin derivative

Cyclodextrins Provide Unique Selectivity

The importance of chiral molecules and the role which enantiomers play concerning biological activity has escalated efforts in the production of optically pure isomers. High resolution gas chromatography is an exceptional analytical tool in the determination of optical purity of both natural and synthetic molecules.

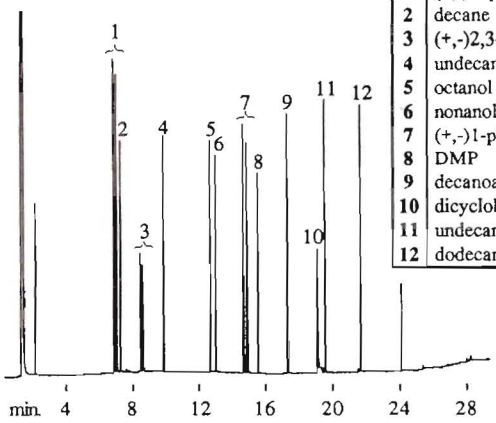
Alkylated cyclodextrin materials can be mixed with common liquid stationary phases to produce capillary columns with the ability to separate volatile enantiomers. The permethylated derivative of beta cyclodextrin is especially selective for a wide variety of chiral separations (1). Optical isomers or enantiomers are non-superimposable mirror images of one another differing only in their interaction with plane polarized light. They have identical physical properties such as boiling point, melting point, and spectroscopic features. Therefore, common liquid phases used in gas chromatography do not possess adequate selectivity for enantiomer separation. The actual mechanism by which cyclodextrin macromolecules (host) and enantiomers (guest) interact is not completely understood (2). Several forces may be involved in relation to "host-guest complexing" but the final result is chiral recognition.

Restek now offers chiral columns to meet the needs of enantiomeric separations. The Rt-βDEXm™ chiral column is a permethylated beta cyclodextrin material doped into the Rtx®-1701 (14% cyanopropyl/86% dimethyl polysiloxane) stationary phase. The Rt-βDEXm™ columns are available in 30-meter lengths with 0.25 and 0.32mm IDs. A 0.25µm stationary phase film thickness provides maximum efficiency and yields optimal resolution of enantiomeric pairs.

Restek's new Rt-βDEXm™ chiral columns are specially tested to ensure reproducibility and selectivity

To assure column-to-column reproducibility, Restek has designed a special test mix for Rt-βDEXm™ columns. The test mix includes three pairs of enantiomers: (+,-)α-pinene, (+,-)2,3-butanediol, and (+,-)1-phenylethanol. The 2,3-butanediol also serves as a test probe for inertness and selectivity. The 2,6-dimethyl phenol and dicyclohexylamine are included to insure acid/base compatibility of the stationary phase. A series of methyl esters is included for total retention and column efficiency measurements. Figure 1 shows the Chiral test mixture analyzed on a 30m, 0.25mmID, 0.25µm Rt-βDEXm™. The symmetrical peak shape and complete resolution of the racemic mixture of the enantiomers indicates both excellent column inertness and selectivity.

Figure 1 - The Rt-βDEXm™ column demonstrates excellent column inertness and resolution of test enantiomers.



Chiral Column Test Mix		
1	(+,-)α-pinene	1.0mg/ml
2	decane	.28
3	(+,-)2,3-butanediol	1.0
4	undecane	.29
5	octanol	.36
6	nonanol	.40
7	(+,-)1-phenylethanol	1.0
8	DMP	.32
9	decanoate	.42
10	dicyclohexylamine	.42
11	undecanoate	.41
12	dodecanoate	.31

30m, 0.25mm ID, 0.25µm Rt-βDEXm™(cat.# 13100)
1µl split injection of Chiral Test Mix (cat.# 35001)
Oven temp.: 40°C to 200°C @ 6°C/min.
Inj. & det. temp.: 200°C **Carrier gas:** hydrogen
Linear velocity: 50cm/sec. set @ 40°C **FID sensitivity:** 8 x 10^{-11} AFS
Split ratio: 42:1

Although the Rtx®-1701 siloxane stationary phase in the Rt-βDEXm™ column is immobilized, the cyclodextrin material can be rinsed out with many common solvents. Therefore, column rinsing is not recommended.

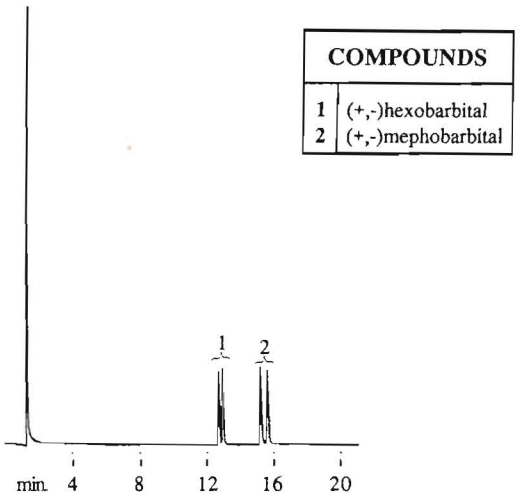
FDA recommends pharmokinetic and toxicity testing for individual enantiomers of new chiral drugs

Stereochemical properties of chiral drugs have been found in many instances to be the controlling factor concerning activity. For example, one enantiomer may be involved in a biological function while its isomeric partner is inactive or exhibits a different functionality. Metabolism of enantiomers may differ significantly, allowing for different rates of reaction for a particular biological process. In some cases one optical isomer may be harmful. Therefore, the Food and Drug Administration (FDA) has recently required drug manufacturers to test individual enantiomers of new chiral drugs for toxicity (3). Figure 2 shows a chromatogram of two common barbiturates analyzed on the Rt-βDEXm™. Resolution of the hexobarbital and mephobarbital enantiomers is obtained in 16 minutes.

Enantiometric separation is highly useful in identification and quality control of many flavors and essential oils

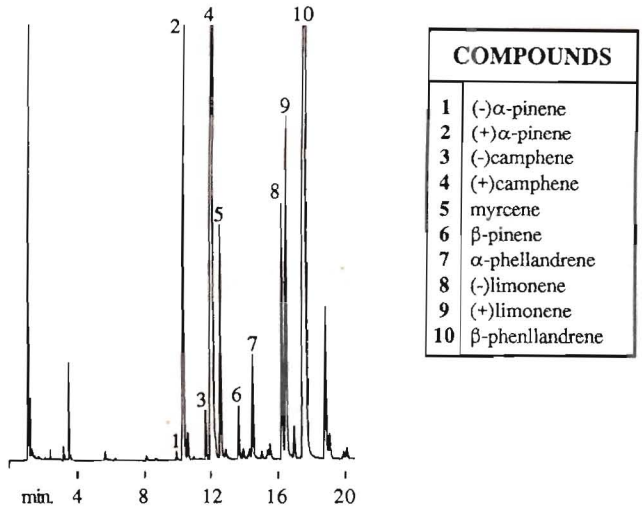
Enantiomeric recognition of compounds contained in natural products has enhanced our level of understanding in many

Figure 2 - The Rt-βDEXm™ column permits enantiomeric separation of common barbiturates.



30m, 0.25mm ID, 0.25µm Rt-βDEXm™(cat.# 13100)
0.5µl split injection of hexobarbital & mephobarbital, concentration=1mg/ml
Oven temp.: 205°C isothermal **Inj. & det. temp.:** 200°C
Carrier gas: hydrogen **Linear velocity:** 50cm/sec. set @ 40°C
FID sensitivity: 8 x 10^{-11} AFS **Split ratio:** 42:1

Figure 3 - Enantiomeric components of ginger oil can be resolved with the Rt-βDEXm™ column.



30m, 0.25mm ID, 0.25µm Rt-βDEXm™(cat.# 13100)
wet needle split injection of ginger oil, concentration=neat
Oven temp.: 50°C (hold 2 min.) to 190°C @ 1°C/min.
Inj. & det. temp.: 200°C **Carrier gas:** hydrogen
Linear velocity: 50cm/sec. set @ 50°C
FID sensitivity: 8 x 10^{-11} AFS **Split ratio:** 71:1 (Split flow: 100cc/min.)

disciplines of scientific research. Natural products often differ from source-to-source, so a thorough analysis of each batch and blend is necessary. Pheromone production in insects has been linked to chiral components in essential oils which are injested while feeding on plant life. The volatility of these compounds makes gas chromatography the ideal analytical tool.

Many cyclic ketones, known as flavor compounds, occur as constituents of essential oils. In some cases, the enantiomers may be distinctly different in flavor and physiological activity. Classes of natural essential oils can also differ in volatile constituents from one another depending on geographic location. Adulteration of natural flavors and fragrances by synthetic additives may also be pinpointed if one can discriminate between ratios of enantiomeric pairs. Figure 3 shows the analysis of ginger oil on a Rt-βDEXm™ column. The enantiomeric selectivity of the Rt-βDEXm™ aids in the identification of the essential oil.

The Restek Rt-βDEXm™ column is highly selective for a wide variety of chiral separations. These chiral columns provide maximum efficiency and resolution between enantiomeric pairs, while the special test mix ensures high column-to-column reproducibility and inertness. ■

References
(1) Keim et. al., "Enantiomer Separation by Gas Chromatography on Cyclodextrin Chiral Stationary Phases", *HRC&CC*, Volume 14, August 1991, 507-529.
(2) Wilfried A. Konig, *Gas Chromatographic Enantiomer Separation with Modified Cyclodextrins*, Huethig, 1992, 137-139.
(3) FDA's Policy Statement for the Development of New Stereoisomeric Drugs, May 1992.

Product Listing

Rt-βDEXm™ 30m, 0.25mm ID, 0.25µm cat.# 13100, $425
Rt-βDEXm™ 30m, 0.32mm ID, 0.25µm cat.# 13101, $460
Chiral Column Test Mix, in methylene chloride, 1ml per ampul, cat.# 35001, $25

Clinical Corner (continued from page 7)

Effective protocols for opiate analysis include extensive sample preparation and optimized instrument parameters. Derivative formation and the use of deactivated glassware, sample vials, and inlet liners will ensure maximum recoveries and response. Optimized detector parameters using selected ions for detection will aid in the identification and of different compounds. ■

Product Listing

Rtx®-5 30m, 0.25mm ID, 0.25µm cat.# 10223, $370

Restek offers a large variety of inlet sleeves for numerous manufacturer's GCs. Please refer to our General Catalog or call customer service at 800-356-1688, ext. 3.

Coming Soon . . . Chemical Standards for Drug Analysis!

Standards Spotlight

EPA Quick Turnaround Method SOW Standards

• *High concentration for maximum value* • *Meet EPA specified quality criteria* • *Full data packs available* •

Restek now has stock chemical standards for all of the EPA Quick Turnaround Methods (QTM) specified in the most recent Statement of Work (SOW). These standards are prepared using precise gravimetric techniques, with concentration verification performed using state-of-the-art capillary chromatography methods.

Quick Turnaround Methods are designed to provide timely data to EPA project officers in several crucial situations: 1) Where field sampling teams have limited knowledge of a waste site and need to focus samples being taken from a particular area, and 2) Where remediation is being performed with heavy equipment on site waiting for sample analysis before proceeding. In all cases, QTM methods require laboratories to submit data to the EPA project officer within 24 hours.

System Monitoring Compounds (SMC) are included in the calibration mixtures at the specified level. Each SMC is also available in an appropriate solvent for matrix spike solution preparation.

QTM Volatiles Method

The QTM volatiles are available in two calibration solutions. Calibration Mix #1 contains all of the components except vinyl chloride. A separate solution containing just vinyl chloride is offered, since this compound is extremely volatile, allowing laboratories to replace vinyl chloride regularly without replacing the less volatile components in Calibration Mix #1.

QTM VOA Calibration Mix #1

2000µg/ml each in 1ml purge & trap grade methanol.
Packaged 1ml per ampul.

benzene
bromodichloromethane
bromoform
carbon tetrachloride
chloroform
chlorobenzene
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethene
trans-1,2-dichloroethene
ethylbenzene
1,1,2,2-tetrachloroethane
tetrachloroethene
toluene
o-xylene
p-xylene
1,1,1-trichloroethane
trichloroethene
cis-1,2-dichloroethene
4-bromofluorobenzene (SMC)

Cat.# 30088 $30 ea.
30088-500 $70 ea. w/data pack
30188 $270 10 pk. w/data pack

QTM VOA Calibration Mix #2

Contains vinyl chloride at 2000µg/ml in 1ml purge & trap grade methanol.
Packaged 1ml per ampul.

Cat.# 30089 $30 ea.
30089-500 $40 ea. w/data pack
30189 $270 10 pk. w/data pack

QTM VOA SMC Mix

4-bromofluorobenzene is the specified SMC in this method. The recommended working solution is to be prepared at a concentration of 50µg/ml in methanol. The following products may be used to prepare the SMC working solution with their respective dilution ratio.

conc. µg/ml	dilution ratio	each	each w/data pk.	10pk. w/data pk.
10,000	1:200	30082/$25	30082-500/$35	30182/$225
5,000	1:100	30003/$25	30003-500/$35	30103/$225
2,500	1:50	30067/$25	30067-500/$35	30167/$225

QTM Phenols Method

This method allows the laboratory to select one of two sample extraction procedures. Because the calibration mix should be prepared in the same solvent as the final sample extract, two different calibration mixes are offered. Use the QTM Phenols Calibration Mix A (in acetonitrile) when extracting samples using solid phase extraction (SPE), and the QTM Phenols Calibration Mix B (in methylene chloride) when using the liquid/liquid extraction procedure.

QTM Phenols Calibration Mix A

Contains 2500µg/ml of each compound in 1ml acetonitrile.
Packaged 1ml per ampul.

phenol
2-methylphenol
2-nitrophenol
2,4-dichlorophenol
2,4,6-trichlorophenol
4-nitrophenol
pentachlorophenol
2-bromophenol (SMC)
2-chlorophenol
3-methylphenol
2,4-dimethylphenol
4-chloro-3-methylphenol
2,4-dinitrophenol
2-methyl-4,6-dinitrophenol
2,3,4,6-tetrachlorophenol

Cat.# 31201 $30 ea.
31201-500 $70 ea. w/data pack
31301 $270 10 pk. w/data pack

QTM Phenols Method (cont.)

QTM Phenols Calibration Mix B

Contains 2500µg/ml of each compound in 1ml methylene chloride
Packaged 1ml per ampul.

phenol	2-chlorophenol
2-methylphenol	3-methylphenol
2-nitrophenol	2,4-dimethylphenol
2,4-dichlorophenol	4-chloro-3-methylphenol
2,4,6-trichlorophenol	2,4-dinitrophenol
4-nitrophenol	2-methyl-4,6-dinitrophenol
pentachlorophenol	2,3,4,6-tetrachlorophenol
2-bromophenol (SMC)	

Cat.# 31205 $30 ea.
31205-500 $70 ea. w/data pack
31305 $270 10 pk. w/data pack

QTM Phenol SMC Mix

Contains 2-bromophenol at 20,000µg/ml in 1ml methanol.
Cat.# 31202 $25 ea.
31202-500 $35 ea. w/data pack
31302 $225 10 pk. w/data pack

QTM Polynuclear Aromatic Hydrocarbons Method

QTM PAH Calibration Mix

Contains 1000µg/ml of each compound in 1ml methylene chloride.
Packaged 1ml per ampul.

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

Cat.# 31203 $45 ea.
31203-500 $85 ea. w/data pack
31303 $405 10 pk. w/data pack

QTM PAH SMC Mix

Contains 2-bromonaphthalene at 20,000µg/ml in 1ml methanol.
Cat.# 31204 $25 ea.
31204-500 $35 ea. w/data pack
31304 $225 10 pk. w/data pack

QTM Pesticides Method

QTM Pesticide Calibration Mix

Contains 25µg/ml of each compound in 1ml hexane
Packaged 1ml per ampul.

α-BHC	endosulfan sulfate
β-BHC	4,4′-DDT
δ-BHC	endrin ketone
γ-BHC (lindane)	methoxychlor
heptachlor	heptachlor epoxide (isomer B)
α-chlordane	γ-chlordane
endosulfan I	4,4′-DDE
endrin	endosulfan II
4,4′-DDD	endrin aldehyde
aldrin	decachlorobiphenyl (SMC)

Cat.# 32036 $30 ea.
32036-500 $70 ea. w/data pack
32136 $270 10 pk. w/data pack

QTM Pesticide SMC Mix

This method specifies preparing a working solution at 5µg/ml.
Contains decachlorobiphenyl at 125µg/ml in 1ml acetone.
Cat.# 32037 $25 ea.
32037-500 $35 ea.
32137 $225 10 pk. w/data pack

QTM PCB Method

This method requires the use of individual Aroclors® in solution with the exception of Aroclor® 1016 and 1260, which are analyzed together. The Aroclor® 1016/1260 mixture, along with the System Monitoring Compound (decachlorobiphenyl) are calibrated at three concentration levels. All other Aroclors® and toxaphene are calibrated at a single concentration.

Aroclor® 1016/1260 Mixture

Contains Aroclor® 1016 and Aroclor® 1260 at 1000µg/ml each in 1ml hexane.
Packaged 1ml per ampul.
Cat.# 32039 $25 ea.
32039-500 $35 ea. w/data pack
32139 $225 10 pk. w/data pack

(Please See Aroclor® & Toxaphene Product Listing Table Below.)

QTM PCB SMC Mix

The method specifies preparing a working solution at 2µg/ml.
Contains decachlorobiphenyl at 200µg/ml in 1ml acetone.
Cat.# 32029 $25 ea.
32029-500 $35 ea. w/data pack
32129 $225 10 pk. w/data pack

Aroclors® & Toxaphene

1000µg/ml in 1 ml hexane	Individual		Individual w/data pack		10pk. w/data pack	
Aroclor® 1221	32007	$25	32007-500	$35	32107	$225
Aroclor® 1232	32008	$25	32008-500	$35	32108	$225
Aroclor® 1242	32009	$25	32009-500	$35	32109	$225
Aroclor® 1248	32010	$25	32010-500	$35	32110	$225
Aroclor® 1254	32011	$25	32011-500	$35	32111	$225
Toxaphene	32005	$25	32005-500	$35	32105	$225

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

Hints for the Capillary Chromatographer

Selecting the Proper Ferrule for Capillary Columns

Proper ferrule selection is critical for capillary column installation. Characteristics such as thermal stability, ruggedness, and compressibility are determined by the different materials used to make ferrules. It is important to choose the right ferrule type and size to ensure proper column installation. The wrong ferrule type could cause damage to sensitive detectors such as ECDs, ELCDs, and MSDs. The wrong ferrule size or type can cause system leaks that result in decreased sensitivity and deterioration.

Ferrule Materials

Since metal ferrules would damage fused silica tubing, softer materials are used for capillary column ferrules. The two most common materials for capillary column ferrules are graphite and Vespel®. These materials can also be combined to form hybrid ferrules with the benefits of each material. Other ferrule materials, such as Teflon® and silicone, are commonly used with packed columns, but because of their limited thermal stability they are not typically used with capillary columns. Table I lists the maximum operating temperatures and the characteristics of common capillary ferrule materials.

Table I - Common Characteristics of Capillary Ferrules

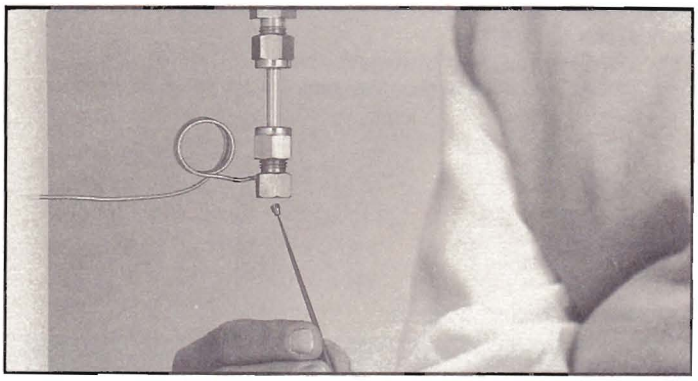
Material	*Max Temp.*	*Characteristics*
Graphite	450°C	Soft, easily conforms to all column sizes. Excellent for high temperature applications. Can flake or deposit particles in inlet & detector fittings. Easily deforms, resulting in limited reusability. Not recommended for vacuum interfaces.
Vespel®/Graphite	400°C	Hard, must be sized to exact column OD. Contracts when cooled causing leakage if not retightened after several thermal cycles. Excellent reusability.

Properties of Graphite Ferrules

Many chromatographers prefer graphite ferrules because they are soft and easily conform to any fitting dimension. Most graphite ferrules are made by tightly winding graphite ribbon around a pin and compressing it into a mold. The graphite ribbon increases ferrule pliability and allows it to deform easily. Increased pliability makes it possible to seal a 0.4mm OD (0.25mm ID) fused silica column with a 0.8mm ID ferrule. In addition, the ferrule can accomodate larger columns if the graphite bore is cored out. These features allow chromatographers to always have the right size ferrule on hand.

Graphite ferrules should be tightened using minimal force. Usually 1/4-turn past finger-tight is sufficient to form a leak-tight seal. If a graphite ferrule is over-tightened, it will extrude out of the bottom of the nut, deform into the fitting cavity, and create ferrule fragments. These particles can be driven further into the inlet or make-up gas fitting, causing adsorption or peak tailing when a column is reinstalled. Graphite ferrules can also flake or abrade and emit particles that can clog small orifices. Because graphite is porous, graphite ferrules leak under vacuum. Therefore, graphite ferrules are not recommended for detectors operated under vacuum, such as MSDs.

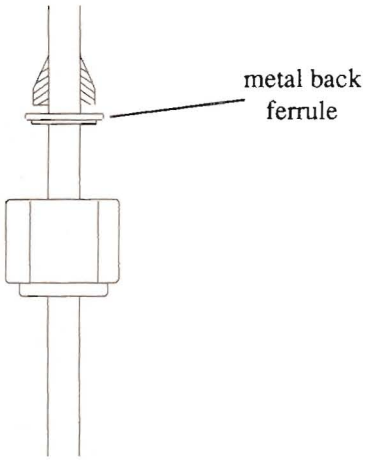
Graphite ferrules must be carefully removed, otherwise fragments and flakes remaining in the fitting can contaminate the GC system. Ferrules are easily dislodged by inserting a tapered needle file into the bore and moving it side-to-side. If the graphite ferrule does not come out in one piece, the inlet or detector fitting should be completely disassembled to ensure that no ferrule fragments remain.



Needle files easily remove graphite ferrules from injector and detector fittings or nuts. Gently insert the file into the ferrule bore and move it from side-to-side to dislodge the ferrule.

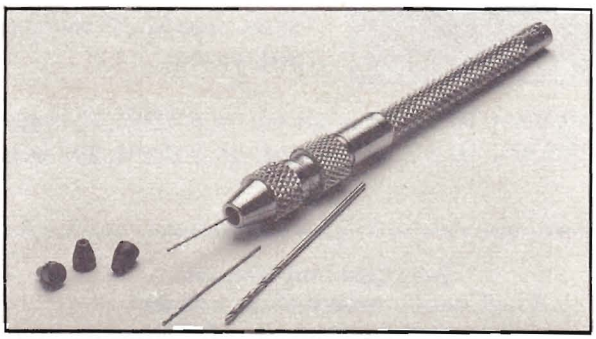
The life of a graphite ferrule is limited because they compress so easily. Some chromatographers obtain new life from a crushed ferrule by installing a reversed Swagelok®-type back ferrule between the fitting and the ferrule (Figure 1). The back ferrule raises the graphite ferrule higher in the fitting, allowing it to seal again.

Figure 1 - Give a used graphite ferrule new life by installing a reversed metal back ferrule in the fitting.



Both 100% Vespel® and Vespel®/graphite ferrules are available. Vespel®-type ferrules are often preferred because they do not flake, deposit particles, or fall apart in a fitting. Most chromatographers choose the Vespel®/graphite ferrule combination. These ferrules are made by compressing a graphite/polyimide powder under high pressure in a heated mold. They retain their shape and can easily be removed intact. Vespel®/graphite has a higher thermal stability than Vespel® (400°C vs. 350°C) and the graphite impregnation makes the ferrule feel softer and seal with less torque. Vespel®/graphite ferrules are currently available in combinations ranging from 85% Vespel®/15% graphite to 60% Vespel®/40% graphite. The 60/40 Vespel®/graphite combinations are preferred by most chromatographers because they seal with the least amount of torque.

Unlike graphite, the inside diameter of Vespel®-type ferrules must be very close to the column OD in order to seal properly. If the ID of a Vespel®-type ferrule is too large for the column OD, it will not compress properly and allow a leak. Usually, the ferrule forms an oval shape, gripping the tubing but not sealing at the ends of the oval. If the ID of a Vespel®-type ferrule is too small to fit over the column, the bore must be enlarged with a small drill.



If the Vespel®/graphite ferrule's ID is too small to fit over the column, a pin vise drill can be used to enlarge the bore.

Vespel®/graphite ferrules will deform to the exact fitting dimension when heated. Usually this deformation process causes the ferrule to become loose and leak during the cool down cycle of a GC oven. Therefore, they must be subsequently retightened after several thermal cycles or carrier gas leakage will occur. No additional shrinkage or loosening occurs once the ferrule has conformed to the internal dimensions of the fitting cavity.

Vespel® ferrules can be removed from a fitting using a tapered needle file in the same manner as a graphite ferrule. Vespel® ferrules sometimes stick to the fitting and column after they have been in use for a prolonged period. Stuck ferrules can be removed by tapping the fitting with a solid object such as a wrench and gently pulling outward on the column. This problem is greatly minimized by using Vespel®/graphite combination ferrules.

What are common ferrule sizes?

Most column connections in the GC inlet and detector are made using $^1/_{16}$" Swagelok®-type fittings. The ID or opening of the ferrule depends on the outside diameter of the column. Table II lists common fused silica capillary column IDs, ODs, and recommended ferrule sizes.

Table II - Common Ferrule Sizes for Fused Silica Capillary Columns

Column ID	*Column OD*	*Ferrule Opening*
0.18 to 0.25mm	0.35 to 0.40mm	0.4mm
0.32mm	0.45 to 0.48mm	0.5mm
0.53mm	0.69 to 0.72mm	0.8mm

The choice of ferrule material is often personal preference. If you are installing a capillary column for the first time, we suggest using a graphite ferrule. Graphite easily forms a leak-tight seal and conforms to any column OD. If you frequently install new columns, Vespel®/graphite is recommended to eliminate particle evolution and minimize maintenance downtime. However, when connecting columns to MSDs or Mass Spectrometer transfer lines, Vespel®/graphite is the only ferrule you should use to ensure a leak-free seal under vacuum. We recommend trying both ferrule types to choose a ferrule that best fits your needs. ■

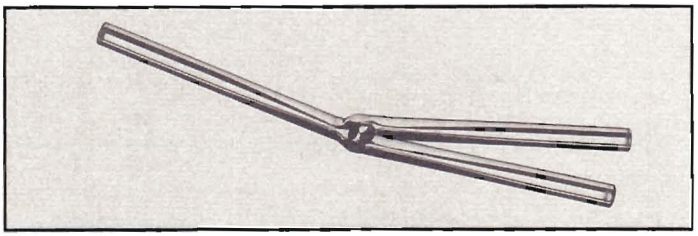
Suggestions?

Is there a topic you would like to see covered in "Hints for the Capillary Chromatographer"? If so, please call our technical service department toll-free at 800-356-1688, ext. 4.

Universal Angled "Y" Press-Tight® Connector

- Made from inert fused silica
- Fabricated at an angle approximating the radius of a capillary column.
- Does not place a strain on column end connections.

Universal "Y" Press-Tights® have become popular for splitting the sample between two columns for simultaneous confirmational analysis. They are also used for splitting the column effluent onto two different detectors. Our analysts had difficulty keeping the column ends sealed in the Press-Tight® because the standard straight "Y" creates strain on the fused silica tubing. To correct this problem, we have designed a "Y" connector bent at the appropriate angle to reduce the strain when connecting two columns or attaching a guard column or transfer line to an analytical column. Now both the inlet and outlet ends of the "Y" conform to the column radius. Fits fused silica tubing with ODs ranging from 0.3 to 0.8mm.



Universal Angled "Y" Press-Tight® Connector
cat.# 20403, $65 each
cat.# 20404, $175/3-pack

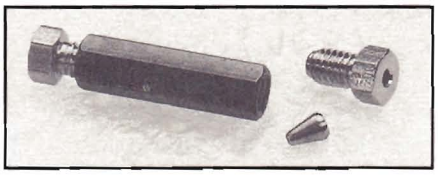
MXT® Low Dead Volume Connectors

In response to customer requests, we have developed metal connectors to join two MXT® columns, attach an MXT® guard column to an analytical column, or perform confirmational analysis with two MXT® columns.

These low dead volume connectors are Silcosteel®-treated, just like our MXT® columns, to make them inert to active compounds. We chose a 1/32" body size to minimize thermal mass and manufactured special metal ferrules that fit the OD of our 0.28 and 0.53mm ID MXT® columns perfectly.

The union connects two pieces of MXT® tubing and the "Y" connects two columns to a guard column or one column to two different detectors. These connectors will not cause peak tailing or affect system inertness and can be used up to 400°C without degrading the deactivation layer.

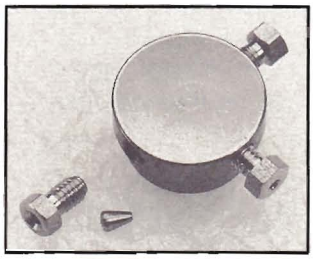
To connect a 0.53mm ID guard column to a 0.28mm ID MXT® analytical column, simply buy the appropriate ferrule sizes. The bodies of both the union and "Y" connectors are the same. A connector for 0.28mm ID MXT® columns will work for 0.53mm ID MXT® columns if the correct ferrules are used. See the chart below to determine what ferrule internal diameter fits the appropriate MXT® column.



MXT® Low Dead Volume Connector

- Connect guard columns/transfer lines to MXT® columns.
- Low thermal mass tracks rapid oven temperature programming.

for 0.28mm ID MXT® columns: cat.# 20397, $50 each
for 0.53mm ID MXT® columns: cat.# 20394, $50 each



MXT® Low Dead Volume "Y" Connector

- Connect two MXT® columns to one inlet.
- Connect one MXT® column to two detectors.

for 0.28mm ID MXT® columns: cat.# 20396, $90 each
for 0.53mm ID MXT® columns: cat.# 20395, $90 each

MXT® Connector Replacement Ferrules

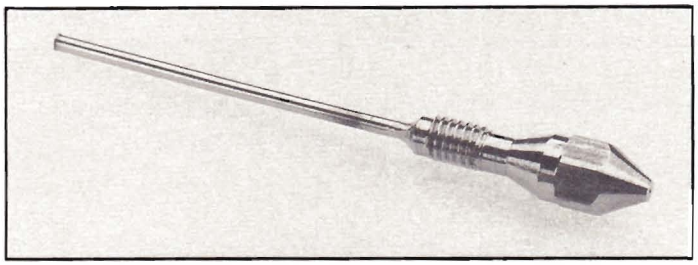
Ferrule ID	Fits column ID	cat.#	price
0.59mm	0.28mm	20398	$45/10-pk.
0.79mm	0.53mm	20399	$45/10-pk.

1/32" replacement nut: cat.# 20389, $15/5-pk

1/4"-3/16" Open End Wrench
A high quality wrench to use with the MXT® Low Dead Volume Connectors.
cat.# 20388, $20/2-pk.

FID Replacement Jet for Hewlett-Packard 5890 GCs

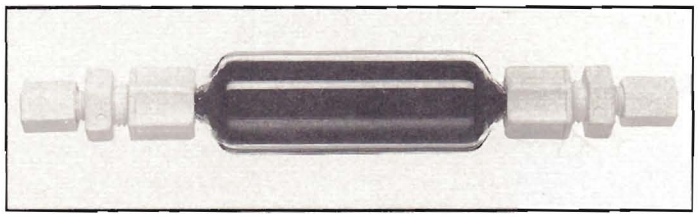
- Fluted jet tip easily guides capillary column into jet bore.
- High performance, Silcosteel® version eliminates adsorption of active compounds.
- Engineered to exceed original equipment specifications.
- Priced lower than HP replacement.



Restek has developed two versions of an HP 5890 FID jet. The standard version (replaces HP part# 19244-80560) is engineered with a fluted jet tip to guide the capillary column into the jet. This design prevents the fused silica column end from hitting the jet tip during installation. The high performance version is the same as the standard version, except that it has been treated with the Silcosteel® process to create an inert interior and exterior. This process coats the entire jet with a micron layer of silica and then further passivates the metal surface by deactivating it in the same manner as our MXT® columns. The high performance jet is extremely inert to active environmental or pharmaceutical compounds. Both versions are precisely machined and undergo stringent quality control to ensure the performance meets or exceeds the original specifications.

Standard HP 5890 Capillary Replacement FID Jet
cat.# 20670, $36 each
cat.# 20671, $95/3-pack
High Performance HP 5890 Capillary Replacement FID Jet
(treated with Silcosteel®, use with active compounds)
cat.# 20672, $48 each
cat.# 20673, $125/3-pack

New High Capacity Split Vent Trap



Potentially hazardous or carcinogenic chemicals can enter the lab atmosphere through the split vent in a capillary GC. As much as 99% of the sample injected vents to the air where chemists working nearby breath these pollutants. This problem is further magnified when multiple GCs are used in the same lab. Split vent traps, packed with charcoal, reduce the uncontrolled release of hazardous materials into the lab.

After examining many trapping materials, we chose a special type of activated coconut charcoal due to its tenacious trapping ability. Several trap designs were also evaluated. Narrow 1/4" trap bodies cause increased back pressure on the inlet system and severely retard retention times. In addition, the excessive backpressure on the split vent outlet can cause the back pressure regulator to perform erratically when the solvent expansion pulse occurs. Therefore, a large trap body design maximizes the quantitiy of charcoal that comes in contact with the sample vapor stream without causing unreasonable backpressure. Trap bodies made from solvent resistant plastics were investigated but continuous solvent exposure caused either cracking or leakage. A glass trap body provided the best resistance and longevity from repeated solvent injections.

When compared to other designs, the new high capacity split vent trap more than quadruples the number of injections that can be performed before solvent breakthrough occurs when compared to other designs (1300 vs. 300 injections). The trap provides protection for thirteen hundred injections or 50 days if one analysis is performed per hour. We recommend trap replacement every 1300 injections or at least every two months. The 1/8" female fittings accomodate most GCs and allow easy installation.

High Capacity Split Vent Trap Kit
includes 1/8" copper connecting tubing, Velcro® mounting strip, and 1/8" ferrule fittings
cat.# 20698, $25 each
cat.# 20699, $100/5-pack

To order any Restek product, call 800-356-1688 (ext.3).
For direct technical service, call 800-356-1688 (ext. 4).