The Advantage
RESTEK
Innovators of High Resolution Chromatography Products

Fast GC Using Microbore Capillary Columns

by Kristi Sellers

Reducing instrument & operator time for gas chromatographic analyses has become an important consideration for many laboratories.

The use of microbore (0.10mm ID) columns can significantly reduce analysis time without sacrificing resolution. The extremely high efficiency of microbore columns (~7000 plates/meter) can provide resolution of complex mixtures while using shorter lengths. Shorter columns are less expensive and reduce analysis times, resulting in a cost savings for the lab.

Some instrument companies have been promoting the benefits of fast screening columns, but the sacrifices required aren't always evident from their literature. The reduction of analysis time at the expense of resolution, sample capacity, and ease of use is not always an acceptable alternative. This article will discuss and demonstrate the benefits and limitations of 0.10mm ID columns.

Speed and Resolution

Table I compares the characteristics of microbore columns to conventional columns. This data holds the key to whether microbore columns are right for your analysis. The most striking difference of microbore columns is their high efficiency (plates/meter) compared to other diameters. **Table I** indicates that a 0.10mm ID column is 160% more efficient than a 0.25mm ID column. This high efficiency allows shorter columns to maintain excellent resolution and increase the speed of analysis. However, some of the other parameters in **Table I** illustrate limitations that may negate the usefulness of microbore columns in your laboratory. The effect of low flow rates, low sample capacity, and high operating pressures on your sample requirements will ultimately determine if microbore columns are an improvement for your laboratory.

Flow Rates

The low flow rates for microbore columns can be either an advantage or a limitation. Low flow rates are beneficial for GC/MS users because the flow rates are well within the pumping capacity of most systems. In addition, the microbore prevents "pumping out the column" or operation below atmospheric pressure. This provides more efficiency

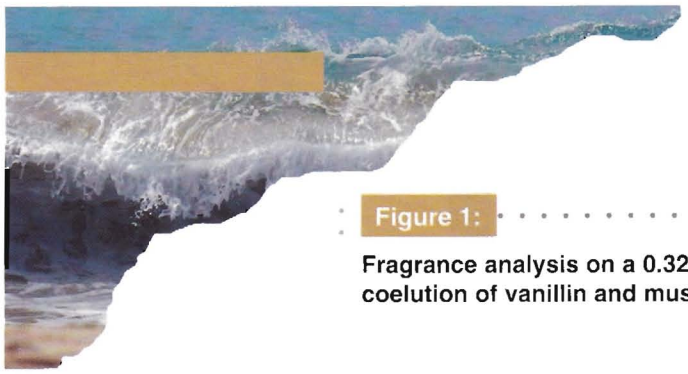
Table I:

Column Characteristics

Column ID	0.10mm	0.18mm	0.25mm	0.32mm	0.53mm
Theoretical plates/m	8,600	5,300	3,300	2,700	1,600
Effective plates/m	6,700	3,900	2,500	2,100	1,200
He flow @ 20cm/sec	0.1cc/min.	0.3cc/min.	0.7cc/min.	1.0cc/min.	2.6cc/min.
H_2 flow @ 40cm/sec	0.2cc/min.	0.6cc/min.	1.4cc/min.	2.0cc/min.	5.2cc/min.
Sample Capacity	5-10ng	10-20ng	50-100ng	400-500ng	1000-2000ng
Operating Pressures	40.0psig	21.0psig	12.5psig	7.5psig	3.0psig

in this issue

Summer 97



for the end user. However, low flow rates also translate into more flow path problems for the chromatographer. Unswept dead volume has disastrous consequences on the column performance.

Operating Pressures

Table I also shows that microbore columns require higher operating pressures which results in more ferrule leaks, septum leaks, and sample blow back through leaking syringe plungers. Connections need to be monitored for leaks more often. The pneumatic systems for older GCs are designed to operate at only 30psig and may need to be modified to handle higher pressures required for narrow bores. Operating microbore columns below optimum pressures will translate into poor resolution and poor performance.

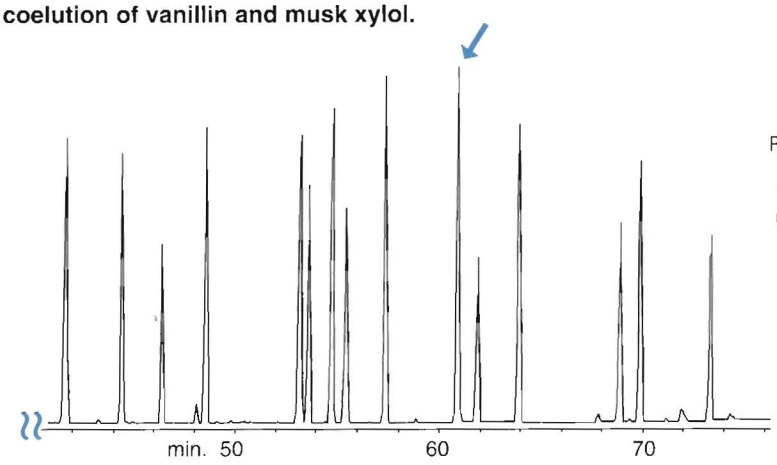
Sample Capacity

A limiting factor of a microbore column is the amount of sample that can be injected onto the column. **Table I** indicates that the sample capacity of a microbore column is ten times less than a 0.25mm ID column. Therefore, the on-column injection should be at least ten times lower for a microbore column.

Sample cleanliness is another important factor to take into consideration when using microbore columns. Because the surface area of the 0.10mm ID columns is much lower than a conventional column, contamination will occur more rapidly when dirty samples are injected. This means that 0.25 or 0.32 mm ID columns will be more rugged and require less maintenance for dirty samples than microbore columns. Whenever possible, samples containing non-volatile residue should be avoided. If dirty samples are a must, extensive column and injection port maintenance is required. Otherwise, loss of resolution, ghost peaks, and a high background signal will result.

Figure 1:

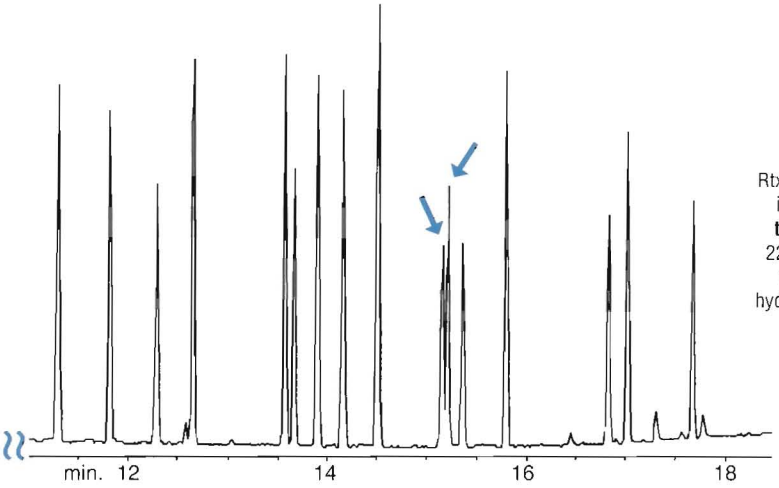
Fragrance analysis on a 0.32mm ID Rtx®-Wax column takes 75 minutes with complete coelution of vanillin and musk xylol.



30m, 0.32mm ID, 0.25µm Rtx-WAX (cat.# 12424). Splitless injection, 50mls/min. **Oven temp.:** 75°C to 225°C @ 70°C/min. (hold 15 min.). **Inj. & det. temp.:** 225°C. **Carrier gas:** helium, 30cm/sec. set @ 75°C.

Figure 2:

Fragrance analysis on the Rtx®-Wax microbore column reduces run times by 75% with increased resolution between vanillin and musk xylol.



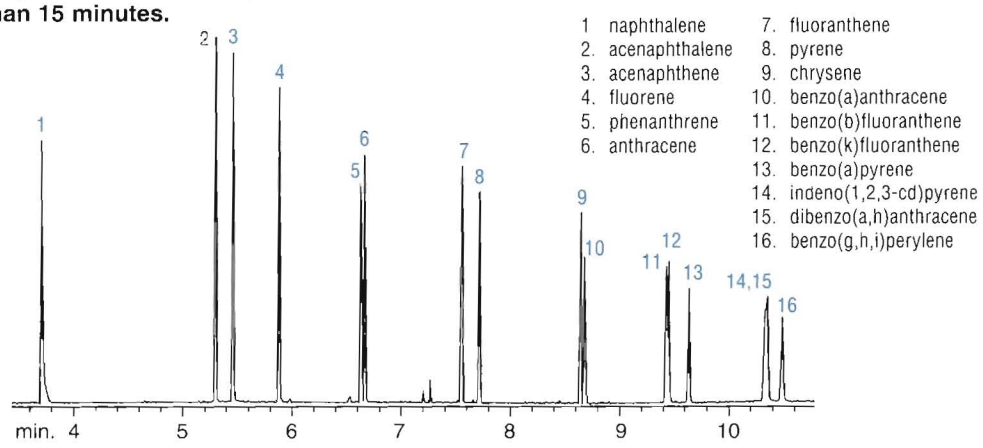
10m, 0.10mm ID, 0.20µm Rtx-WAX (cat.# 41603). Splitless injection. 250mls/min. **Oven temp.:** 55°C (hold 1 min.) to 225°C @ 10°C/min. **Inj. & det. temp.:** 225°C. **Carrier gas:** hydrogen, 70cm/sec. set @ 55°C.

Injector Considerations

Direct and on-column injection modes are not recommended due to the required low flow rates and small bore size of these columns. Therefore, trace analyses are difficult to perform with microbore columns. Split and splitless injections are the best alternatives. However, since microbore columns require low flow rates, speed of sample transfer through the liner to the column is a concern. Due to the high dead volume, poor peak shape, and response, loss of resolution will occur when 2 or 4mm ID liners are used in conjunction with microbore columns. Thus, 1mm ID inlet liners are a must for sharp, well resolved, and recovered peaks. Not only is the inlet liner a consideration when

Figure 3:

Polynuclear aromatic hydrocarbons on an Rtx®-5 microbore column are analyzed in less than 15 minutes.



10m, 0.10mm ID. 0.10µm Rtx-5 (cat.# 41201). 0.5µL splitless injection. 41psi initial pressure, hold 2 min. 8 psi/min. to 99psi (hold 1.87 min.). 275°C, vent open @ 1 min. **Oven temp.**: 40°C (hold 0.5 min.) to 90°C @ 70°C/min. then to 100°C @ 5°C/min. then to 310°C @ 30°C/min. (hold 2 min.).

using microbore columns for split or splitless injections, but other parameters specific to the type of injection method must also be optimized.

In a split injection, the choice of inlet liner and initial temperature will affect peak shape, response, and resolution the most. **Figure 1** shows part of a typical fragrance analysis on a conventional column (0.32mm ID). Under optimal conditions (4mm ID inlet liner and initial temperature of 75°C), the analysis time is more than 70 minutes and the separation of vanillin and musk xylol could not be achieved. By switching to a microbore column and optimizing run conditions (1mm ID inlet liner and initial temperature of 55°C), we were able to reduce the analysis time to 18 minutes and attain 80% resolution of the vanillin and musk xylol as shown in **Figure 2**. The 1mm ID inlet liner improved the recovery and peak shape of the early eluting compounds.

Figure 3 illustrates a splitless PAH analysis on a 10m, 0.10mm ID, 0.10µm Rtx®-5 using an optimized inlet liner and inlet pressure. When a 2mm ID inlet liner was used, high molecular weight discrimination occurred. By changing to a 1mm ID inlet liner, high molecular weight discrimination was eliminated. However, this change caused peak splitting of the early eluting compounds. The peak splitting was eliminated completely when pressure programming was applied in place of constant pressure.

Detector Considerations

Detector design and flows must be optimized when using microbore columns. Make up gas flows may need to be increased to minimize detector dead volume and compensate for the lower column flow rates. Since peak widths are approximately half compared to conventional columns (< 1 second), fast integrator and detector electrometers must be used. Integrator sampling rates must be increased over rates used for 0.25mm ID columns since the peaks are much narrower with microbores. If the sampling rate is too slow, then poor integration and non-reproducible peak areas will result. Check with your instrument company and data system manufacturer to be sure your system is capable of handling microbore sampling rates.

Microbore columns can produce shorter analysis times, equivalent resolution, and provide cost savings. But remember, converting your conventional system to a microbore system isn't as easy as changing columns. Column capacity, sample purity, and injector and detector conditions must be considered and optimized for a successful analysis. Keep in mind that when switching from conventional capillaries to microbore columns, there may be the need to optimize inlet temperatures, liners, and GC run conditions.

Product Listing:

Microbore Capillary Columns			
0.10mm ID, 0.10µm			
Column	**temp. limits**	**10-meter**	**20-meter**
Rtx®-1	-60 to 330/350°C	41101 $250	41102 $415
Rtx®-5	-60 to 330/350°C	41201 $250	41202 $415
Rtx®-Wax	20 to 250°C	41601 $250	41602 $415
0.10mm ID, 0.20µm			
Column	**temp. limits**	**10-meter**	**20-meter**
Rtx®-Wax	20 to 250°C	41603 $250	41604 $415
0.10mm ID, 0.40µm			
Column	**temp. limits**	**10-meter**	**20-meter**
Rtx®-1	-60 to 320/340°C	41103 $250	41104 $415
Rtx®-5	-60 to 320/340°C	41203 $250	41204 $415

Contact Restek's GC experts to discuss the suitability of Microbore or other GC columns for your specific application.

Rtx®-CLPesticides Column

The Complete Solution for Chlorinated Pesticides Analysis

by Frank Dorman

- **Baseline resolution of the 22 pesticides in EPA 8081.**
- **Analysis time under 25 minutes.**
- **Exceptional inertness for endrin, DDT, & methoxychlor.**
- **Temperature stability > 300°C.**
- **Exceeds performance criteria for EPA 8081, 608, and CLP.**

For years, environmental laboratories have struggled with various chlorinated pesticide analytical methods. They must keep track of resolution requirements and breakdown performance criteria while analyzing extracts which usually contain high-boiling contaminants. Historically, many laboratories have used cyanopropyl capillary column stationary phases (DB-1701, Rtx-1701) which very often provide the best resolution between target compounds, but have several limitations. The new Rtx®-CLPesticides column has been specially designed to overcome the resolution, breakdown, and temperature limitations of these other phases.

The Rtx®-CLPesticides column is capable of baseline resolution of the 22 common chlorinated pesticides (see **Figure 1**) listed in USEPA Methods 8081 and 608 and in the EPA's Contract Lab

Figure 1:

The Rtx®-CLPesticides column provides baseline resolution of 22 chlorinated pesticides in less than 25 minutes.

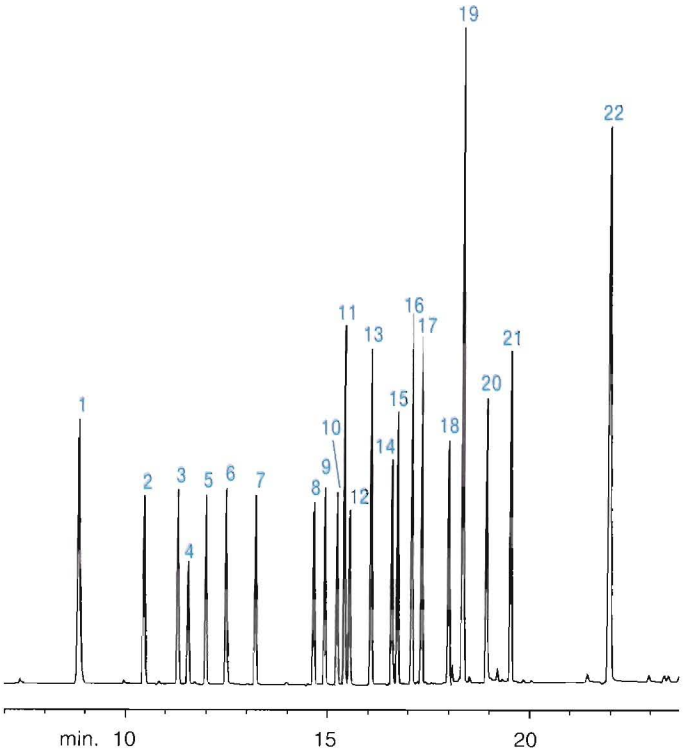
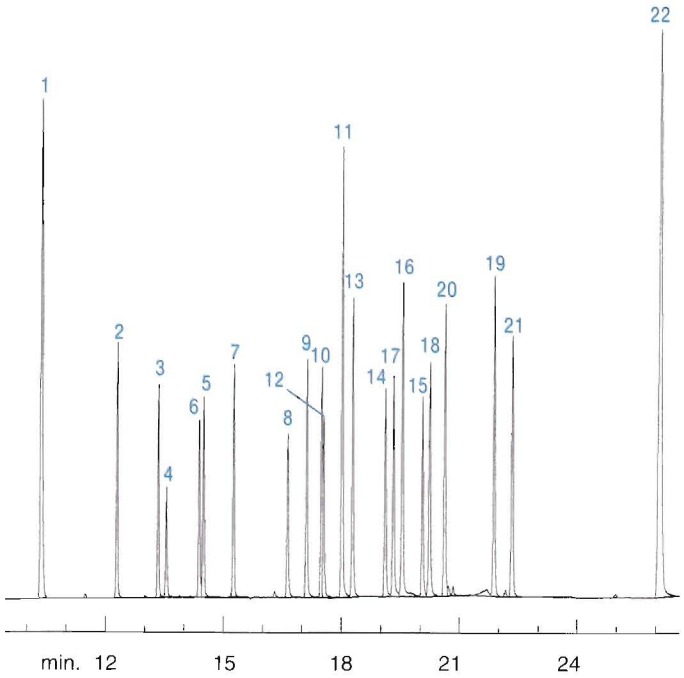


Figure 2:

The Rtx®-35 column provides elution order changes for 8 of the 20 chlorinated pesticides and is currently the best confirmational column for chlorinated pesticides.



Program (CLP) protocol. This column is available in 0.25, 0.32, and 0.53mm IDs, and has been optimized for ECD analysis resulting in very low bleed after conditioning. Analysis time has not been sacrificed in order to obtain baseline resolution. In less than 25 minutes, your analysis is complete with the Rtx®-CLPesticides column. This results in the laboratory's ability to make at least two injections per hour—an important factor for increasing sample throughput. In addition to its separating power, the Rtx®-CLPesticides column has a maximum temperature of 330°C, allowing you to bake out high molecular weight contaminants.

Although the Rtx®-CLPesticides column is clearly the column of choice for this analysis, most laboratories require a second column of different polarity for confirmation. To fulfill this requirement, Restek recommends using the Rtx®-35 column. It provides elution order changes for several compounds, while exhibiting high thermal stability and inertness. The Rtx®-35 only has two compounds that elute closely: endosulfan I and α-chlordane. Under the same temperature and flow conditions as the Rtx®-CLPesticides column, the Rtx®-35 column is an excellent choice for a confirmation column, see **Figure 2**.

The combination of the Rtx®-CLPesticides and Rtx®-35 columns provides unsurpassed performance for the analysis of chlorinated pesticides. They both exhibit the necessary resolution, low bleed, and thermal stability for this demanding analysis, as well as exceptional inertness for methoxychlor, endrin, and DDT. Both columns provide different elution orders for confirmation. If you are involved with the analysis of chlorinated pesticides and want to improve your resolution and increase your throughput, try the Rtx®-CLPesticides column.

Peak List & Conditions for Figures 1 & 2

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

30m, 0.32mm ID, 0.50µm Rtx®-CLPesticides (cat.# 11139)
30m, 0.32mm ID, 0.50µm Rtx®-35 (cat.# 10439)

Oven temp.: 120°C (hold 1 min.) to 285°C @ 8.5°C/min. (hold 6 min.).
Inj. port: Direct injection using a Uniliner® (cat.# 20335) at 200°C.
Detector: ECD at 300°C with Anode Purge
Flow rate: helium @ 2.1ml/min. set @ 120°C

Product Listing:

Rtx®-CLPesticides Columns		
	Cat.#	**Price**
30m, 0.25mm ID, 0.25µm	11123	$445
30m, 0.32mm ID, 0.50µm	11139	$475
30m, 0.53mm ID, 0.50µm	11140	$525

Rtx®-35 Columns		
	Cat.#	**Price**
30m, 0.25mm ID, 0.25µm	10423	$385
30m, 0.32mm ID, 0.50µm	10439	$415
30m, 0.53mm ID, 0.50µm	10440	$465

New CLP Pesticide Standards

Organochlorine Pesticide Mix AB #1

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

200µg/ml ea. in hexane/toluene (1:1), 1ml/ampul

	Each	**5-pk.**	**10-pk.**
	32291 $35	**32291-510** $157.50	—
w/data pack	**32291-500** $45	**32291-520** $175	**32391** $315

Organochlorine Pesticide Mix AB #2

aldrin	8µg/ml	dieldrin	16µg/ml
α-BHC	8	endosulfan I	8
β-BHC	8	endosulfan II	16
δ-BHC	8	endosulfan sulfate	16
γ-BHC (lindane)	8	endrin	16
α-chlordane	8	endrin aldehyde	16
γ-chlordane	8	endrin ketone	16
4,4'-DDD	16	heptachlor	8
4,4'-DDE	16	heptachlor epoxide (B)	8
4,4'-DDT	16	methoxychlor	80

At concentration listed in hexane/toluene (1:1), 1ml/ampul

	Each	**5-pk.**	**10-pk.**
	32292 $25	**32292-510** $112.50	—
w/data pack	**32292-500** $35	**32292-520** $125	**32392** $225

Optimizing Linear Velocity and Temperature Program for Capillary Chiral Analyses

by Sherry Sponsler

Chiral chromatography can be a useful tool for determining purity and authenticity in flavor, fragrance, and drug applications. As with all GC analyses, certain parameters should be considered to attain maximum enantiomeric separation and chiral capillary column performance. GC conditions such as linear velocity and temperature program must be optimized to ensure maximum chiral resolution.

Linear Velocity

"Faster is Better"

In many instances, the resolution between enantiomers can be improved with linear velocities that are faster than those considered optimum by the Van deemter curve. This has been demonstrated for five out of six racemic compounds that were evaluated on a 30-meter, 0.32mm ID, 0.25µm Rt-βDEXsa column using different linear velocity conditions. Although optimum linear velocity can be different for each chiral compound and column, the typical optimum linear velocity for maximum enantiomeric separation is ~80 cm/sec. with hydrogen carrier gas (**Figure 1**). This is twice the expected linear velocity required to achieve maximum column efficiency, as indicated by the Trennzahl values in **Figure 2**. Therefore, conditions providing maximum chromatographic peak efficiency do not always result in optimum chiral resolution.

Not all chiral components achieve maximum enantiomeric resolution at the same linear velocity, so this must be adjusted for specific target analytes. For instance, Grob [1] demonstrated optimum chiral resolution of γ-lactones at 50 cm/sec.[1] However, 1-octen-3-ol was an exception since Figure 1 shows no increase in chiral resolution from 40 cm/sec. to 80 cm/sec.

Temperature Program

"Slow Temperature Ramp Rates Are Better"

Several different temperature ramp rates were evaluated to determine the optimum linear velocity for enantiomeric resolution of 6 chiral compounds. The best chiral separations were achieved with temperature program rates between 1 and 2°C/min. (**Figure 3**). Unlike linear velocity, Trennzahl values increase along with enantiomeric resolution at these temperature program rates (**Figure 4**). Lower elution temperatures can provide increased enantiomeric resolution for chiral compounds. This suggests that selectivity of cyclodextrin columns improves with decreased temperature during the separation process.[1]

Flow rates that provide maximum separation efficiency do not always result in optimum enantiomeric selectivity. Higher linear velocities and slow temperature ramp rates promote lower elution temperatures, which can provide better chiral separation. Optimizing these conditions can enhance cyclodextrin column performance and the overall quality of chiral chromatography.

Figure 1:

Higher linear velocities provide maximum resolution of chiral pairs.

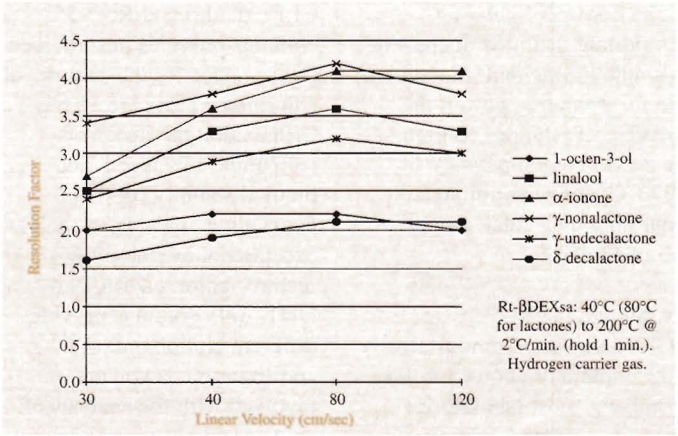
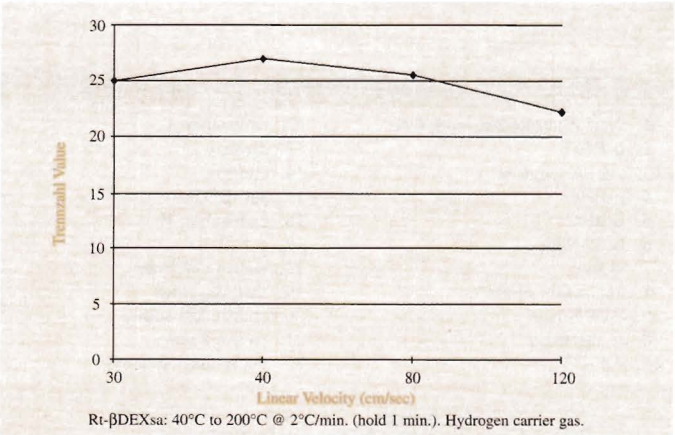


Figure 2:

Higher trennzahl values do not correspond with optimum enantiomeric separation.



What about helium? The observed optimum linear velocity for helium carrier gas is about 60 cm/sec for many of these chiral separations.

Figure 3:

Temperature program rates between 1 and 2°C/min. provide optimum enantiomeric separation.

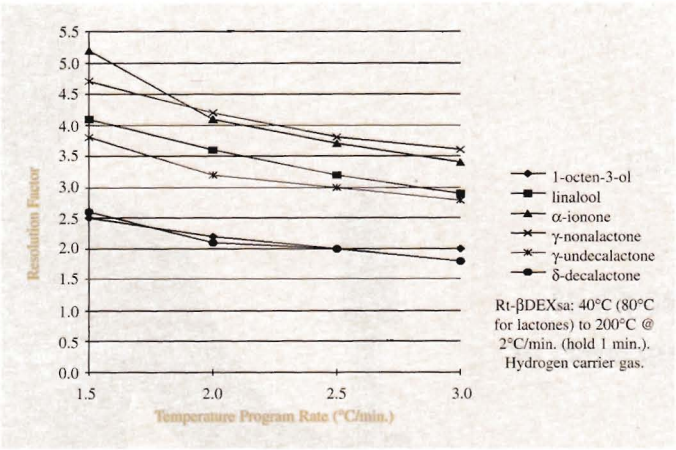
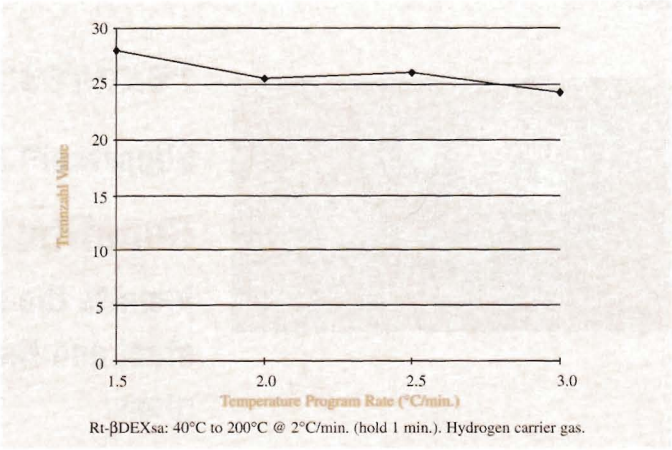


Figure 4:

Trennzahl values increase along with enantiomeric resolution at these slow temperature ramp rates.



References

[1] K. Grob, H. Neukom, H. Schmarr and Armin Mosandl, *Journal of High Resolution Chromatography* **13**, 433-436 (1990).

RESTEK IS THE LONG-LIFE LEADER. . .

Just Ask Chris Nelson and Richard Johnson at PPD Pharmaco!

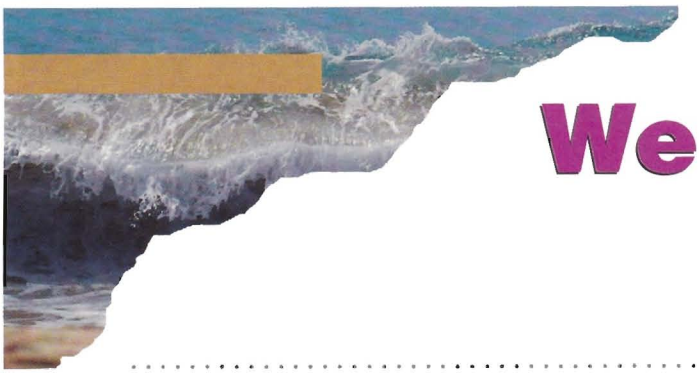
Chris Nelson and Richard Johnson, chemists at PPD Pharmaco in Madison, Wisconsin, truly believe that Restek is the ***Long Life Leader***! Chris was using a Gamma-cyclodextrin trifluoro-acetyl column for the analysis of dexfenfluramine and fenfluramine. Unfortunately, the columns he had been using for the analysis had so many problems, they really made his job difficult. They gave inconsistent performance, had poor resolution, were extremely expensive, were difficult to obtain because of very long delivery times, and once the seal was broken they could not be returned. But most importantly, the columns did not last very long. Chris only averaged about 150 injections before the column failed!

When he switched to Restek's Rt-βDEXcst columns, Chris was amazed. How could a column that costs less last ten times longer? On top of being less expensive and lasting longer, the columns were delivered quickly and showed more column-to-column consistency. According to Chris, "Every column we pull out of the box works".

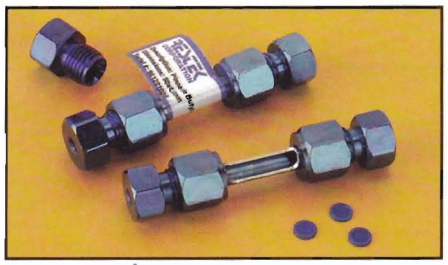
With Restek's Rt-βDEXcst columns, Chris averages 1,000 to 1,100 injections before the column's performance degrades, unlike the 150 injections from the other columns he was previously using. One Restek column actually performed for up to 2,000 injections!

Thanks Chris for sharing your information with everyone and showing that once again Restek is the ***Long-Life Leader***!

Richard Johnson and Chris Nelson have made 2,000 injections on Restek's Rt-βDEXcst column before replacement was necessary.



We put more into c



Features:

Silcosteel® Tubing

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Imparts the strength of steel and the inertness of glass.

Features:

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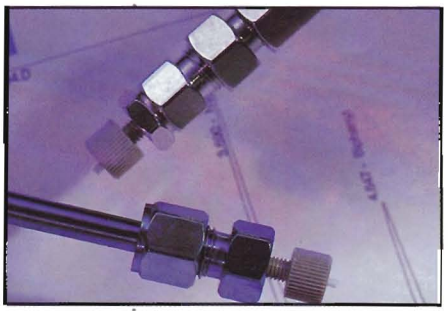
Benefits:

Reliable column protection without the hassle of connecting tubing.

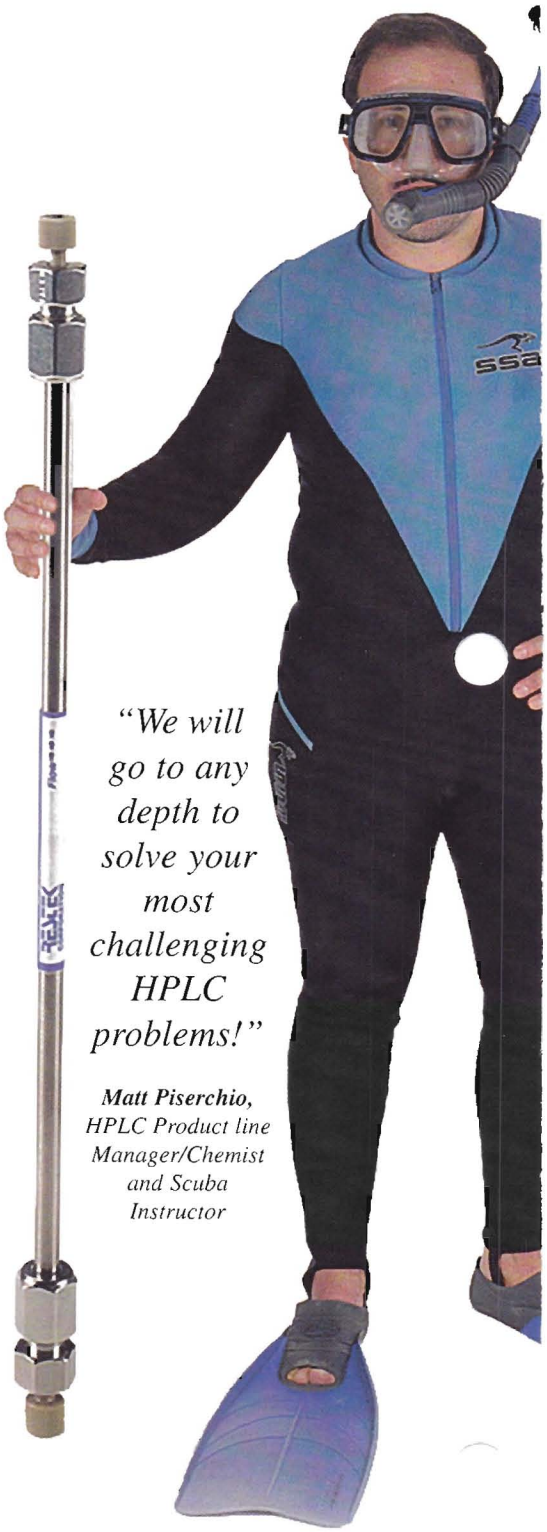


Features:

Stringent QA Specifications

Benefits:

Guarantee of High Performance



"We will go to any depth to solve your most challenging HPLC problems!"

Matt Piserchio,
HPLC Product line Manager/Chemist and Scuba Instructor

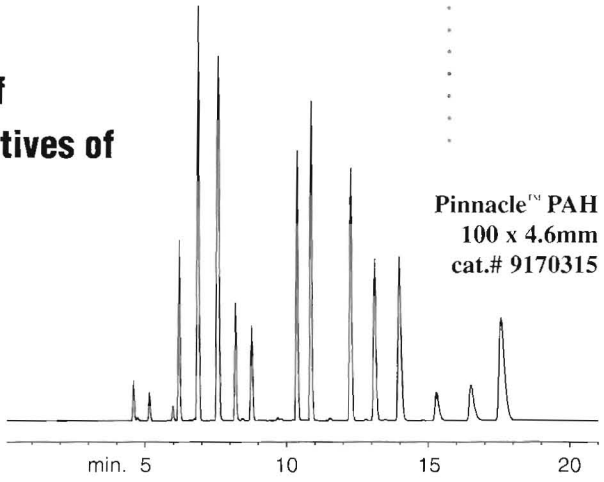
HPLC Columns...

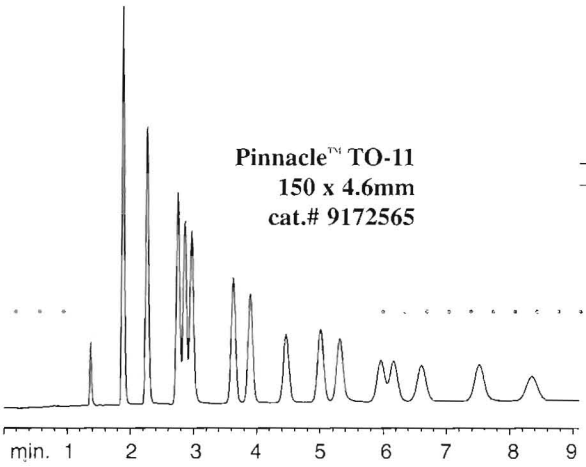
Features:

Application Specific Columns:
Pinnacle™ PAH, Pinnacle™ TO-11

Benefits:

Rapid separations of several classes of compounds, i.e. PAHs and DNPH derivatives of aldehydes and ketones.





Features:

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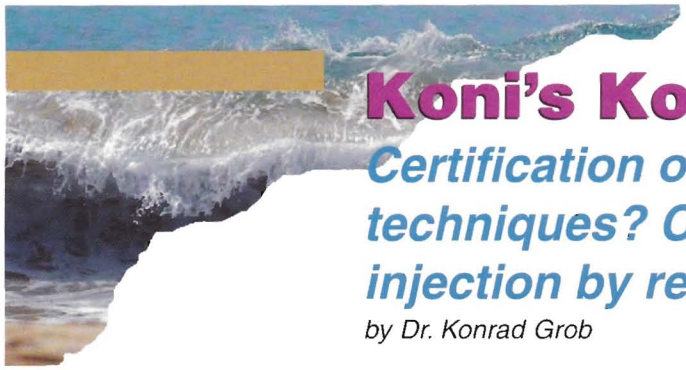
...So you get more out of them.



Koni's Korner

Certification of injectors and injection techniques? Comments on splitless injection by readers.

by Dr. Konrad Grob

In the spring and summer 1996 issues of *The Restek Advantage,* I posed the question of whether the most frequently used injection technique in capillary GC, splitless injection, is as mature as one tends to think. I traced its history to show that there has never been the systematic optimization and testing many think should have happened. Nobody felt responsible: Users assumed that instrument manufacturers provide exhaustively tested injectors and working instructions, whereas instrument companies just produce what they think "science" wants. But who is "science"?

Optimization of a technique as complex as splitless injection is work of such a volume that it cannot be accomplished on a single Friday afternoon, when the work of the week is completed. One of the open questions concerned sample evaporation in splitless injection. Should the liner be empty or packed? Should it have a constriction at the bottom? I was hoping for contributions by those routine users who must have found an answer in one way or another, but only received more general comments, three of which I want to bring up here.

Who introduced splitless injection?

Leslie Ettre was upset by my saying that my father introduced/invented splitless injection. Indeed, non-splitting injection was used from the very beginning of capillary GC, in particular before splitting was invented. I want to apologize for not having mentioned this. In response to him, my definition of "splitless" injection is not any non-splitting injection technique, but that of using an injector with a split outlet which is closed during the splitless period. At least in Europe, "direct" injection has always been distinguished from "splitless" injection.

Accelerated transfer through increased flow

E.H. Foerster, from Southwestern Institute of Forensic Science in Dallas, Texas, found that the analysis of low concentrations of certain active drugs (he named alprazolam, trazodone, and quinidine) was possible by split, but not by splitless injection (4 mm i.d. liner with glass wool). He could improve the results from splitless injection approximately four fold by increasing the carrier gas inlet pressure (gas flow rate) during a 1 min. transfer period after injection. He explained this by the reduced residence time in the injector during split or accelerated splitless injection. The same argument was used by Hewlett-Packard in favor of what they termed "pressure pulse".

An increase of the flow rate by a factor of four is possible only if initial inlet pressures are modest and, nevertheless, does not seem overwhelming: it reduces reaction time by a factor of four. However, the effect could be more than proportional, since the sample liquid deposited onto the packing initially forms an island cooled to the solvent boiling point. A high flow rate might remove the solute material from these surfaces before they have reached the injector temperature again. If evaporation occurred in the gas phase, the fog of the non-evaporated solute material could have been transferred into the column before it settled onto the packing material. Unfortunately, Mr. Foerster did not compare the performance of the packed liner with that of the empty liner, because gas phase evaporation is usually still most gentle (but not always complete).

Injector overloading

Gary Kellog, from the Springfield, Missouri Public Health Department, drastically illustrated the effect of overloading too small vaporizer chambers. "Last February we received a new GC/MS system, including a Varian 1078 temperature programmable split/splitless injector. At about the same time, I received my first copy of *The Restek Advantage* including your article on injector design and sample introduction. I had never used a split/splitless injector before. The old instrument was set up with a flash vaporization injector with a 0.53 mm ID column, and it didn't take long to realize that the old operating parameters would not work on the new system. When I began to calculate the vapor volumes and the liner volumes (54 mm x 0.8 mm ID with 9 mm column installed height, methanol as solvent), it was obvious that a lot of my sample was going into places other than the column. Due to the limited size of the 1078's liners (54 mm long), I chose the largest ID liner offered (3.4 mm), added a 1 cm plug of deactivated fused silica wool placed above the installed column height, and began to experiment with the temperature programming on the injector. I also switched to a lower vapor volume solvent, with a higher boiling point to take advantage of solvent effects (toluene)."

Gary Kellog used a mixture of pesticides to compare the peak areas obtained by the old conditions (0.8 mm i.d. liner, 250°C) with those he introduced recently (3.4 mm i.d. liner, injector programmed from 200 to 300°C). The detector, column, injection volume, and other conditions

	Peak area x 10^6		
Compound	**0.8 mm i.d.**	**3.4 mm i.d.**	**Difference**
alpha HCH	0.63	4.16	7.85
diazinon	0.69	6.34	9.19
heptachlor	0.49	3.98	8.12
endrin	0.41	2.32	5.66
p,p'-DDT	0.72	5.00	6.94
coumaphos	0.69	2.08	3.01

were identical. Results were obtained with toluene as the solvent, which must have improved them substantially. From a long list of results, I just want to cite a few.

The results show drastic (66-89 %) losses of solute material with the small vaporizer chamber, but also that losses are different for each component. This was no surprise. The usable volume in this vaporizing chamber was 23µl. 1µl of methanol (which he usually used) must have produced 600-700µl of vapor (@ 250°C injector temperature & intermediate inlet pressure). Even if the needle was only partially emptied, the injector was overloaded more than 40 times. 1µl of the toluene actually used produces only about 200µl of vapor. Losses of solute material are usually smaller than those of the solvent, because solutes may be deposited onto surfaces cooled by the evaporating solvent—but the process is poorly controlled. It is as if an analyst would spill more than 90 % of the solution during titration and then be surprised that results are poorly reproducible. There is no pool of liquid running out of the GC instrument, which in turn explains why so many people "spill" in the GC inlet without noticing it.

Gary Kellog's new injection technique might perform correctly, although it involves unusual conditions. He introduced his solution in toluene (b.p.110°C) into the PTV at 200°C. Standard working rules would require an injector temperature at, or below, the pressure-corrected solvent boiling point, in order to prevent rapid expansion of the vapors. He calculated that the chamber has a usable internal volume of 354µl, which should be sufficient to store the vapors even when considering that they will mix with the carrier gas present in the injector. A 2µl volume (or 1µl of a solvent producing more vapor), however, would again be too much. Further, he applied some glass wool, which might retain the solutes when solvent vapors expand out of the injector chamber.

Confusing injection conditions

Gary Kellog plans to carefully test his injection conditions, maybe by comparison with on-column injection. However, does it really make sense that every gas chromatographer develop his own conditions to get his sample into the column?

The comment by Gary Kellog demonstrates how chaotic injection in capillary GC still is. In HPLC, injection just requires filling of a sample loop without air bubbles and that the sample solvent is not too strong an eluent. It is standardized and essentially the same for all instruments. It is totally different in GC. Every instrument manufacturer seems to be proud of producing something different than the others and giving their injector another name. Did you ever count the names given to temperature-programmable injectors? Manuals do not provide sufficiently clear and safe rules on how to operate the device and warnings on where the limitations are. Confusion among the non-specialists is inevitable.

Why didn't anybody tell Gary Kellog that his old injector cannot be used in the way he used it—and how many others continue to do the same? Why didn't he know that with his new injector he can inject up to about 50µl (quite regardless of the vapor volume formed), provided he keeps the chamber below the solvent boiling point for the time of solvent evaporation?

Why are injectors and injection techniques not validated?

Today, splitless injection is frequently performed with too small vaporizing chambers, too short syringe needles, poorly suited carrier gas supply systems, excessively large samples, by the cool instead of the hot needle technique (or vice versa), by slow instead of rapid injection, with too low carrier gas flow rates, wrong column temperature during the sample transfer, too short splitless periods, packings in the liner at the wrong site, and without information on what all the critical parameters are. Properly written methods should specify all these conditions in at least as much detail as they specify sample preparation by saying that the flask must be rinsed twice and the solvent combined.

Analytical methods are validated in order to demonstrate the reliability of results. Chemicals, balances and pipettes are usually of certified quality and performance. Users check them ever so often. GCs are also checked. Oven temperatures are measured—as if this would be a critical parameter. Methods describe all steps of sample preparation in great detail, but when they reach the injection of the sample into GC, they become extremely short. Their authors would say that they cannot write as many versions as there are instrument manufacturers. True. But many users would badly need instructions, especially if their instruments work properly at best under special conditions.

The quality management people might not have realized the potential of the errors occurring during injection, as shown by the above example, it is many times larger than that of a balance. How can they validate methods if one of the principal sources of error remains out of control? Maybe they did realize the problem, but felt unable to make valid suggestions. Methods cannot be validated for all the different injectors on the market, nor can they require the use of an injector from a particular manufacturer. They must assume a properly working injection system and the application of validated working rules for that system. These rules do not exist. At least for the time being, the concept of validation reaches its limit at this point. It underlines that capillary GC is not a simple technique and it relies a great deal on the expertise of the operator.

3 Final Points

1) Does it really make sense that every gas chromatographer finds his own way to get his sample into the column?

2) How can methods be validated if one of the principal sources of error, injection, remains out of control?

3) Methods cannot be written in as many versions as there are instrument manufacturers.

I welcome your feedback.
Reach me by e-mail at
Konrad.grob@attach.ch.

Attention Heated Purifier Users!

Restek Improves a Laboratory Classic

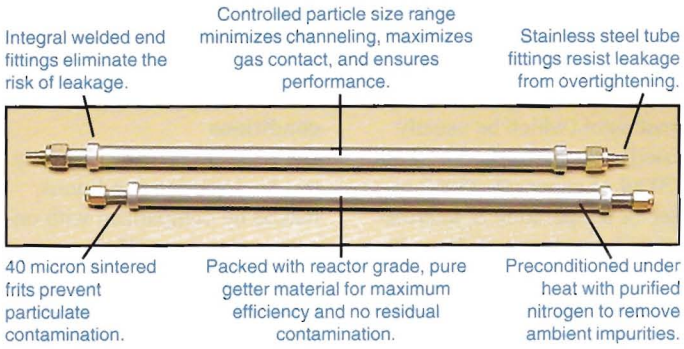
by Doug Elliott and Brad Rightnour

For decades, heated purifiers have provided unequaled service for contamination capacity, efficiency, and versatility. That is because nothing has been found to be more effective overall than heated non-evaporable getter materials for removing critical reactive contaminants from gas streams...***until now***.

Restek has taken current technology a step farther. Besides removing the same quantity of oxygen and water (14L and 35L) and purifying the same number of gas tanks at the same high 1100 ml/min. maximum flow rate as the original Supelco heated purifier tube, our attention to design detail eliminates leak sources, our control of material specifications optimizes performance, and our production processes remove potential contamination; essentially preconditioning the tube. This provides the analyst with hassle-free installation.

The next time you need a replacement tube, choose Restek's Straight Replacement Purifier Tube and appreciate the differences. You'll be glad you did! As a special offer, **SAVE 10% off the regular catalog price now through August 1997!**

Restek's replacement Purifier Tube has improved fittings, getter material, and manufacturing processes for optimum carrier gas purification.



Replacement Straight Purifier Tube		
1/8-inch	cat.# 21661, ~~$90~~ *$81*	Similar to Supelco #2-2396
1/4-inch	cat.# 21660, ~~$90~~ *$81*	Similar to Supelco #2-2398

Restek Designs a Thermal Gas Purifier Furnace and U-Shaped Getter Tube

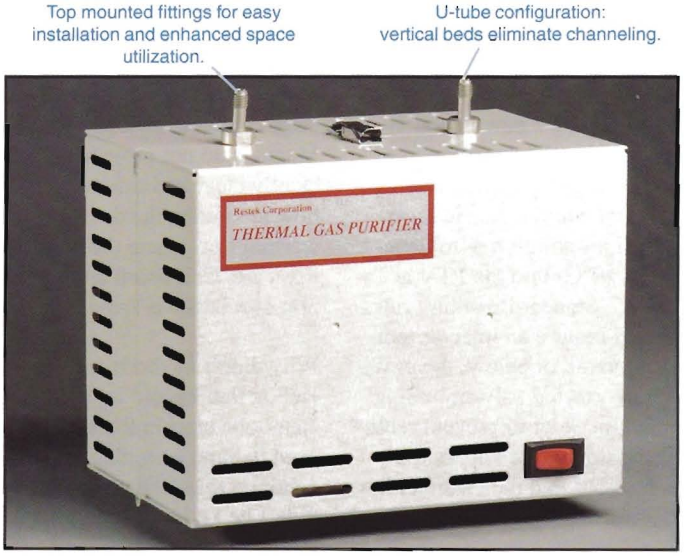
If you are not one of the analysts currently using heated getter technology to purify your carrier gas, try the Restek Thermal Gas Purifier. *It's everything you'd expect in a heated purifier plus more!* The unit:

- Completely removes up to 14 L of O_2 and 35 L of H_2O vapor.
- Purifies up to 50 cylinders at 50 ppm impurity at flow rates up to 1100 ml/min.
- Removes O_2, H_2O, CO, CO_2, sulfur compounds, and hydrocarbons.
- Saves you money by using less expensive grades of carrier gas.

Restek Thermal Gas Purifier		
Fittings	110v	220v
1/8-inch	cat.# 21664, $400	cat.# 21665, $420
1/4-inch	cat.# 21662, $400	cat.# 21663, $420

Restek Purifier Replacement U-Tube	
1/8-inch	1/4-inch
cat.# 21659, $90	cat.# 21658, $90

Restek's Thermal Gas Purifier combines the benefits of U-tube getter configuration with improved material handling to provide optimum carrier gas purification performance.



Rtx®-5

the Column of Choice for FIA Fuel Testing

by Lisa Serfass

Dr. Peter Tibbetts of M-Scan Ltd (Ascot, England) attends every Formula 1 Grand Prix Race. Although an avid racing fan, he doesn't actually get much time to enjoy the race. Dr. Tibbetts is the Director of Environmental Services at M-Scan Ltd and also serves as a Fuels Consultant for the FIA (Federation Internationale De l'Automobile). It is his responsibility to test fuel samples from the Formula 1 race cars in the FIA's Mobile Fuel Testing Lab.

The FIA is the governing body that sets the regulations that all Formula 1 competitors must comply with. It is the FIA's responsibility to ensure that each race car meets all technical regulations for safety as well as performance. Fuel testing is only one part of the process. The cars undergo intense scrutiny to ensure they meet all criteria set by the FIA. In addition to being measured and weighed, each car is checked for the proper engine capacity, aerodynamics, control systems, tires, and refueling systems.

Capillary gas chromatography is used to analyze the fuel and determine the exact pattern of components contained within the sample. Since race car fuel is composed of a complex mixture of hydrocarbons and other volatile organic compounds, each sample exhibits different chromatographic patterns. These patterns are known as fingerprints, since no two fuels are exactly alike.

The fuel specifications process begins with each racing team submitting a sample of the race fuel they wish to use for approval. If the sample is approved, its fingerprint is placed into a databank. The information is held for comparison with fuel samples at race time. Three replicate 250ml fuel samples are taken at the race. They can be taken at any time, usually during qualifying or just before or after the race. The containers are then sealed and witnessed by the team concerned to ensure there is no tampering with the samples. One of the samples is tested and compared with the fingerprint in the databank. The sample must match the fingerprint. If it does not, the race stewards are informed of the discrepancy and the sample is then sent on to the UK laboratory for a full mass spectral analysis. The second sample is sent to an independent laboratory for testing and the third sample is returned to the racing team so they can have their own analysis performed.

The fuel samples are analyzed using a 30 meter, 0.32 mm ID, 3.0µm Rtx®-5 capillary column. The Rtx®-5 column was chosen for this analysis because race car fuel is a highly complex mixture of very volatile compounds. The high separation efficiency of this column combined with the thick coating of stationary phase provides the necessary resolution needed to produce the unique fingerprints. The Rtx®-5 polymer was also chosen for its stability which results in extended column lifetime and very low background bleed level.

In addition to the GC analysis, additional testing is done to measure the density of the sample. The sample is injected into a PAAR DMA48 Density Meter and held at a constant temperature (15°C) in a glass U-tube. This is vibrated by a piezo-actuator and the natural frequency is measured. The natural frequency is directly proportional to the mass of the U-tube containing the fuel sample. Since the volume is known, the density can be calculated. Calibration is checked by injecting dodecane as a reference standard.

Usually, a minimum of three cars are randomly chosen from each race for testing. Each fuel analysis takes approximately 60 minutes and all testing must be completed before the race results can be confirmed. With all that is required for fuel testing, the drivers and cars aren't the only ones in a race. Dr. Tibbetts is in his own race —a race of time.

Figure 1:

Restek's high performance Rtx®-5 column is used to characterize fuel samples from Formula 1 racecars in a mobile fuel testing lab.

Rtx®-5

30-meter, 0.32mm ID, 3.0µm: cat.# 10284 $415

Peak Performers

New Inlet Liners for Fisons 8000 & Shimadzu 17A Injectors

- Deactivated & tested for guaranteed inertness.
- Low internal volume makes them ideal for purge & trap or Fast GC analysis.
- Guaranteed to meet original equipment specifications.

Fisons 8000 1mm Split Sleeve		
8.0mm OD x 1.0mm ID x 105mm		
Each	**5-pack**	**25-pack**
20916 $42	20917 $168	—

Shimadzu 17A 1mm Split Sleeve		
5.0mm OD x 1.0mm ID x 94mm		
Each	**5-pack**	**25-pack**
20976 $32	20977 $128	20978 $512

Stainless Steel Capillary Nuts for HP 5890 GCs

Stainless Steel Capillary Nut (inlet)	
For use with Standard Ferrules	**For use with Compact "HP-type" Ferrules**
20883 $25/2-pack	21884 $25/2-pack

New Compact Vespel®/ Graphite Ferrules for HP GCs

Restek now offers the compact Vespel®/graphite (60%/40%) ferrules for HP GC capillary injection ports. The Vespel®/graphite ferrules are designed to seal with minimal torque, are reusable, and are stable to 400°C. They are available in economical 10 & 50 packs and meet original equipment specifications.

Compact Vespel®/Graphite Ferrules for HP GCs				
Ferrule ID	**Fits Column ID**	**V/G 10-pack**	**V/G 50-pack**	**Similar to HP part #**
0.4mm	0.25mm	20238 $45	20239 $180	5062-3516
0.5mm	0.32mm	20248 $45	20249 $180	5062-3514
0.8mm	0.53mm	20263 $45	20264 $180	5062-3512

Compact Graphite Ferrules for HP GCs				
Ferrule ID	**Fits Column ID**	**Graphite 10-pack**	**Graphite 50-pack**	**Similar to HP part #**
0.4/0.5mm	0.25/0.32mm	20250 $25	20251 $100	HP0100-8853
0.8mm	0.53mm	20252 $25	20253 $100	HP0100-1042

Graphite Capillary Ferrules			
Ferrule ID	**Fits Column ID**	**Graphite 10-pack**	**Graphite 50-pack**
0.4mm	0.25mm	20200 $25	20227 $100
0.5mm	0.32mm	20201 $25	20228 $100
0.8mm	0.53mm	20202 $25	20224 $100

Vespel®/Graphite Capillary Ferrules			
Ferrule ID	**Fits Column ID**	**V/G 10-pack**	**V/G 50-pack**
0.4mm	0.25mm	20211 $30	20229 $120
0.5mm	0.32mm	20212 $30	20231 $120
0.8mm	0.53mm	20213 $30	20230 $120

Please direct comments & suggestions on this publication to my attention:
Sherry Wenrick, Newsletter Editor, Ext. 2113, or e-mail to sherw@restekcorp.com

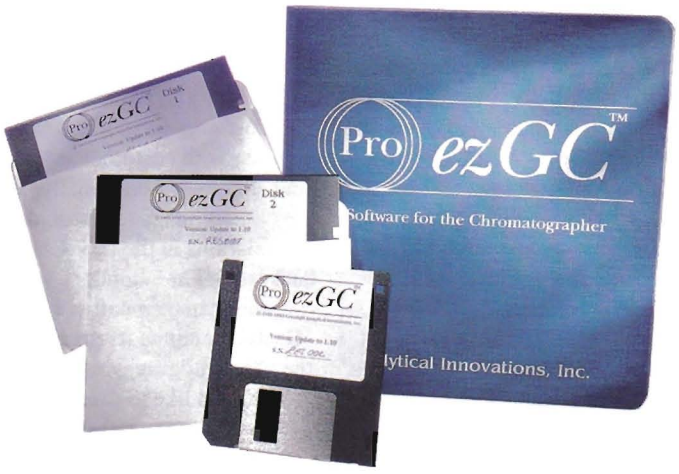
ISO 9001

Pro ezGC™ Software (Ver. 2.0) for Windows®.

The most powerful GC optimization software has new features!

by Mike Feeney

- **Optimize temperature and flow programs from a single analysis.**
- **Model retention gap and guard column applications including Restek's Integra-Guard™ columns.**
- **Model columns in series for process GC and multi-dimensional GC analyses.**
- **Software routines expanded for Fast GC and Windows® 95.**



Pro ezGC™ software is a powerful optimization software and retention index database for gas chromatography. This program accurately predicts GC separations on any capillary column and is useful for selecting the best column and conditions for your GC analysis. Pro ezGC™ software utilizes either your chromatographic retention data or you can use the extensive library containing thousands of commonly analyzed compounds. From this data, it is possible to automatically evaluate thousands of combinations of column dimensions, oven temperature program, and carrier gas pressure program to determine the best separation with the fastest analysis time. This saves days of method development time for new analyses and can decrease analyses times of existing analyses by more than 50%. Several new features incorporated into version 2.0 make Pro ezGC™ software for Windows® even more powerful and easy to use.

Optimize separation and analysis time from a single GC run

In order to accurately simulate separations and analysis time, the software requires either selecting a compound from an existing library or entering retention times from a GC analysis. This new version of Pro ezGC™ software for Windows® can calculate the thermodynamic retention indices from a single chromatographic run. This analysis can be your current method, retention data from a scientific journal, or retention times from publications such as Restek's chromatography products catalog. This makes it possible to select a GC column for your analysis and estimate the separation quickly and easily.

Model GC separation including guard columns and capillary restrictors

The analysis of contaminated samples often requires the use of a guard column to minimize contamination of the analytical column. Cool on-column injection often utilizes retention gaps to improve peak widths and to facilitate injection using standard gauge syringes. These "precolumns" can either be connected using a low-volume union or built-in as with Restek's Integra-Guard™ columns. In GC/MS applications, a length of deactivated tubing is sometimes added to the end of the analytical column to either increase the GC inlet pressure or to decrease background bleed from the transfer line tube. Pro ezGC™ software for Windows® now permits accurate modeling when these types of columns are used, even when the diameters are not identical to the analytical column.

Predict the optimum column for dual columns connected in series or parallel

Many times, resolution of all components in a sample is not possible using a single column. This is where the power of Pro ezGC™ software becomes especially obvious because the program can either model two columns connected in parallel to two separate detectors or two columns connected in series. With series connections, the lengths of the two columns are often cut to give the best combination. This approach would be costly and time consuming without Pro ezGC™ software. Now, any laboratory can quickly evaluate whether serially coupled columns will give the separation without cutting up valuable capillary columns.

Take the guesswork out of selecting the best column and conditions for your GC analysis with Pro ezGC™ software for Windows®. Increase the productivity of your existing gas chromatographs by improving separations and shortening analysis time with optimized temperature and carrier gas programs. For more information, download a copy of the demo software directly from Restek's website at http://www.restekcorp.com or call Technical Service at (800) 356-1688, ext. 4, and request a demo disk of Pro ezGC™ software for Windows®.

Pro ezGC™ for Windows® version 2.0
cat.# 21487 $795

Upgrade from Pro ezGC™ for DOS to Pro ezGC™ for Windows®
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RESTEK Behind the Scenes

Restek On The Road

Restek On the Road Seminar...June 9, Minneapolis, MN

Restek On the Road Seminar...June 10, Detroit, MI

Restek On the Road Seminar...June 11, Rockford, IL

Restek On the Road Seminar...June 12, Chicago, IL

Restek On the Road Seminar...June 13, St. Louis, MO

Air & Waste Management Association's 90th Annual Meeting and Exposition...June 8-13, Toronto, Canada.

IFT Food Expo...June 14-18, Orlando, FL.

NERM '97...June 23-25, Saratoga, NY.

Pesticide Residue Workshop...July 13-16, St. Pete Beach, FL.

111th AOAC International Annual Meeting and Exposition...September 8-10, San Diego, CA.

Gulf Coast Conference...September 9-11, Galveston Island, TX.

Welcome, Barbara Lyke and Chris Lope!

Barbara Lyke is our new Manger of Information Services. Her background in IS has been diverse and includes positions held at The Pennsylvania State University, Office of Business Services; Murata-Erie North America, Inc.; and International Business Machines, T.J. Watson Research Center. Barb is a graduate of Marymount College, Tarrytown, NY, with a B.S. in Business and has continued her education in computer science and business logistics at Pace and Penn State Universities. Barb will be leading Restek's IS group into the next century. No Y2K bugs here!

In his new role as Director of Sales and Domestic Distribution, ***Chris Lope*** will be responsible for Restek's growing Technical Sales Force and its outstanding Customer Response Team, along with customer service and shipping. Additionally, he will manage distributor relations in the U.S. Chris' background in laboratory sales and management will help Restek continue its growth in the chromatography market. If you would like to discuss any issues relating to the sales and service of Restek products, please contact Chris at 800-356-1688, ext. 2175.

Team Pennsylvania Comes to Restek

Team Pennsylvania is comprised of a 32-member board made up of top corporate executives and Governor Ridge's cabinet members that form a central business resource center. The purpose of this "team" is to visit businesses throughout the state, listen to their needs, and coordinate efforts by people across Pennsylvania to improve the business climate. It is intended to allow businesses to be treated as customers and to get the attention of state business leaders and elected officials. Restek, first on the list of businesses to visit, was selected because of the company's environmental track record, recent expansion, growth rate, and overall success.

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