THE RESTEK ADVANTAGE

Innovators of High Resolution Chromatography Products

MXT®-500 Sim Dist

New carborane stationary phase for High Temperature GC

by Andy Schuyler

Hot

- **MXT columns will not break like brittle Al-Clad columns.**
- **Safe for Hydrogen carrier gas.**
- **Low bleed and long life to 430°C.**

Gas chromatographic analysis at temperatures above 400°C requires stationary phases and tubing that can withstand temperatures beyond the limits of most conventional polymers and tubing used in GC. By incorporating carborane into the backbone of the polymer chain, the thermal stability is increased (**Figure 1**). Because these slightly polar polymers are not pure dimethyl polysiloxanes like MXT-1, Restek uses the phase designation MXT-500 Sim Dist for this new stationary phase.

Tubing Constraints

An improved, high-temperature stationary phase is not enough: the tubing used is also an important consideration. The polyimide coating that keeps fused silica tubing flexible breaks down rapidly at oven temperatures above 360°C and is unsuitable for high temperature gas chromatography. Aluminum-clad tubing overcomes the problems with the polyimide, but has limitations. When repeatedly temperature programmed above 400°C or

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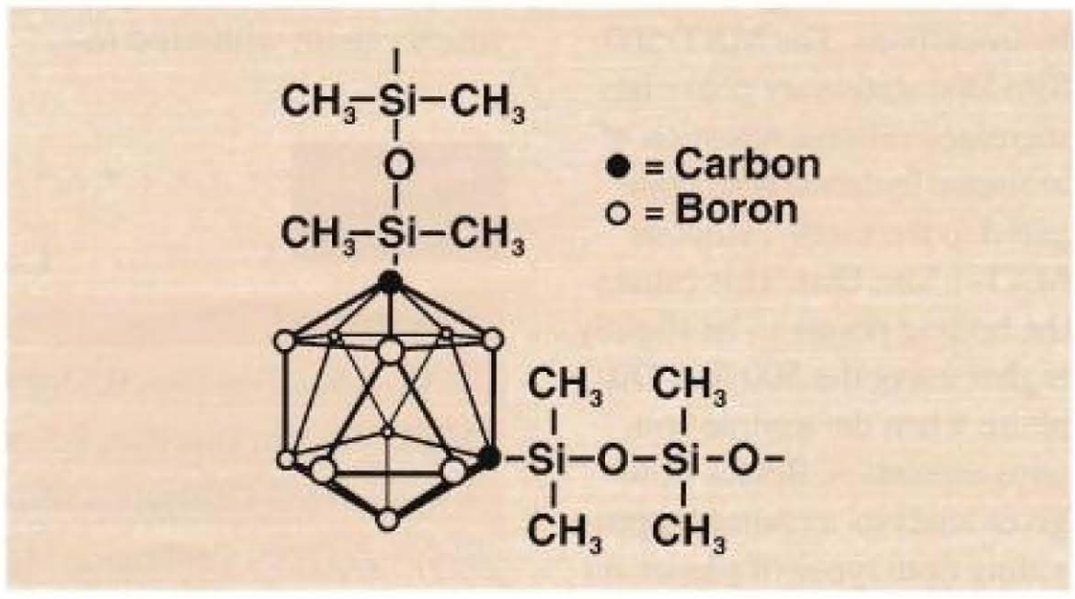
Figure 1:

Carborane dimethyl polysiloxane—MXT-500 Sim Dist stationary phase.



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MXT®-500 Sim Dist

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Tubing stable at ANY GC temperature!

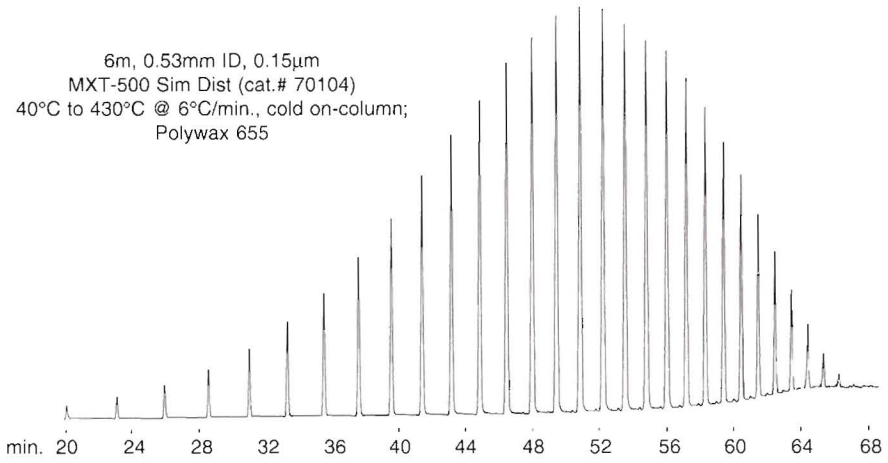
allowed to cool below 50°C, the aluminum sheath becomes brittle and eventually breaks. The most durable capillary columns available are Restek's MXT columns, which are manufactured using Silcosteel® tubing (metal tubing with the inertness of fused silica and the robustness of metal).

MXT columns are designed for High Temperature Simulated Distillation

Simulated distillation (Sim Dist), one of the most common high temperature GC applications, is a good demonstration of the durability of MXT columns. Simulated distillation is a technique in which the GC is calibrated using the retention times of hydrocarbons that have published boiling points. The analysis of a high molecular weight petroleum sample, such as lubricating oil, is compared to the calibration by a special software program and the boiling range distribution is determined. Simulated distillation requires stable retention times under temperature programmed conditions and a baseline with low bleed that is repeatable with multiple temperature programmed analyses. **Figure 2** illustrates low and stable column bleed, excellent peak symmetry, and good recovery of the high molecular weight hydrocarbons in Polywax 655 on an MXT-500 Sim Dist column. Retention time and baseline stability are excellent indications that the polymer is stable. This column has been operated at 430°C isothermal for over 100 hours without significant retention time shift or baseline increase. And, of course, the MXT tubing will never become brittle!

Figure 2:

The MXT-500 Sim Dist column demonstrates low bleed and stable baseline to 430°C for high temperature Sim Dist calibration and analysis.



Restek offers MXT-1 and MXT-500 Sim Dist stationary phases for high temperature Sim Dist

Although the carborane 500 Sim Dist stationary phase is the most stable phase available for Sim Dist, many analysts prefer to use a true methyl silicone column for this analysis. Differences in polarity of the stationary phases cause a shift in the calculated boiling range distribution for petroleum samples containing aromatic hydrocarbons. The MXT-500 Sim Dist stationary phase has increased relative retention of aromatic hydrocarbons compared to the methyl silicone MXT-1 Sim Dist. This causes the boiling points to be slightly higher using the 500 Sim Dist phase when the sample contains aromatics. Restek now gives analysts a choice by providing both types of phases on high temperature MXT columns. While both columns can be operated to 430°C, the MXT-500 Sim Dist column has lower bleed and longer lifetime, while the MXT-1 offers methyl silicone polarity that matches many laboratories' historical data.

Durable Silcosteel® tubing and stable stationary phases for High Temperature GC

High temperature GC challenges the limits of existing column and stationary phase technology. Restek's MXT tubing is ideally suited to the task when compared to fused silica or aluminum clad tubing, which cannot withstand repeated temperature programmed operation to 430°C. These temperatures also push GC polymers to the limit of thermal decomposition. But Restek's new MXT-500 and MXT-1 Sim Dist columns hold up well under these extreme conditions. When properly conditioned to 430°C, these columns give stable baselines with low bleed and repeatable retention times needed for high temp Sim Dist and other HTGC analyses.

Product Listing:

Columns

Description	cat.#	price
MXT-1 Sim Dist (6m, 0.53mm ID, 0.15µm)	70101	$300
MXT-500 Sim Dist (6m, 0.53mm ID, 0.15µm)	70104	$300
Polywax (655 Calibration Material 1 gm)	36225	$10
Polywax (1000 Calibration Material 1 gm)	36227	$10

The Rtx®-OPPesticides Column

Fast, efficient analysis of Organophosphorus Pesticides in EPA Method 8141A

by David Smith

- **Efficient analysis of 55 components in EPA Method 8141A. Includes: 49 organophosphorous pesticides, 4 internal standards, and 2 triazines.**
- **Quick analysis—less than 20 minute run time for 55 compounds.**
- **Flexible column configuration—0.32mm ID allows for direct injection with FPD or NPD detection *and* GC/MS confirmation.**
- **Maximum Temperature of 300°C.**

In contract analytical labs, time is money when analyzing client samples. The efficient throughput of samples in shorter periods of time results in higher revenue. Using the latest in method and phase development technologies, the chemists at Restek have designed a unique phase for the efficient analysis of organophosphorus (OP) pesticides. The Rtx-OPPesticides can analyze the 55 components in EPA Method 8141A in under 20 minutes, which is 50% faster than other published analysis times.[1]

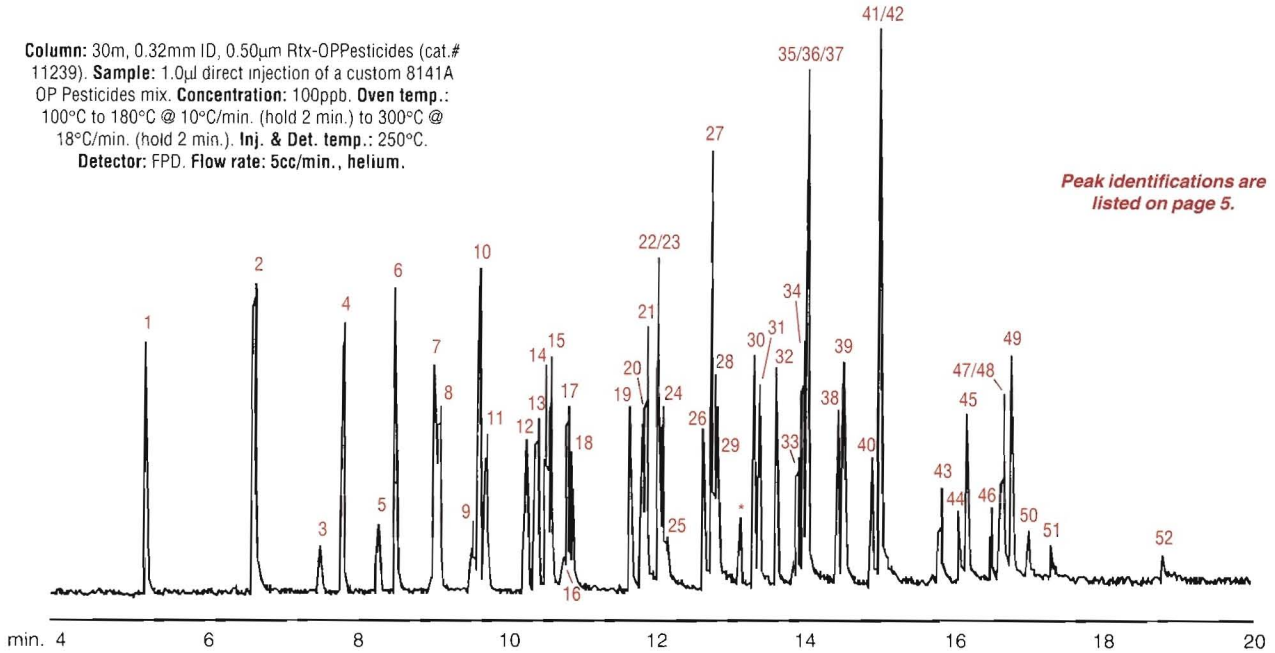
When the Rtx-OPPesticides column is run under the specified conditions, it exhibits four coelutions involving nine compounds. The coelutions will not affect identification and quantitation of GC/MS analysis. For more element-specific detectors, such as the NPD or FPD, these coelutions may be of concern if the full compound list is analyzed, although the 27 compounds used for method 8141A validation are all resolved. In order to present a complete method for organophosphorus pesticides, the Rtx-35 was researched and chosen as a confirmational column (see Figure 3 on page 4).

An illustration of the organophosphorus pesticide separations achieved with the 30m, 0.32mm ID, 0.5µm Rtx-OPPesticides is shown in **Figure 1**. The concentration of the standard is 100ppb, which is

Continued on page 4.

Figure 1:

The Rtx®-OPPesticides column separates 43 organophosphorus pesticides at 100ppb in less than 20 minutes by FPD.



The Rtx®-OPPesticides Column

Continued from page 3.

near the lowest concentration used in the development of calibration curves. The 100ppb standard shows the inertness of the column when analyzing low levels of organophosphorus pesticides. With this column, it is possible to analyze the OP pesticides in Method 8141A using a direct injection with run times under 20 minutes. The two triazine herbicides listed in Method 8141A, atrazine and simazine, are not detected using an FPD, but are resolved as shown in **Figure 2**.

The ion trap chromatogram shown in **Figure 2** exemplifies the flexibility of the Rtx-OPPesticides. The same dimension column used to

Continued on page 5.

Figure 2:

The 0.32mm ID Rtx®-OPPesticides allows GC/MS separation of OP pesticides and triazines.

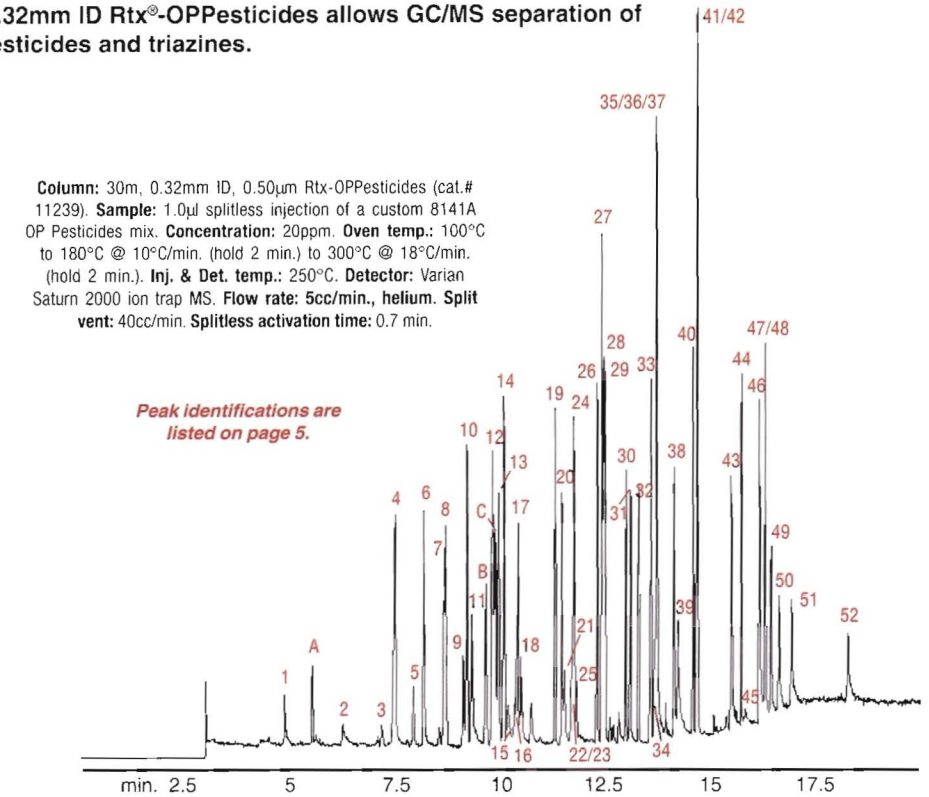
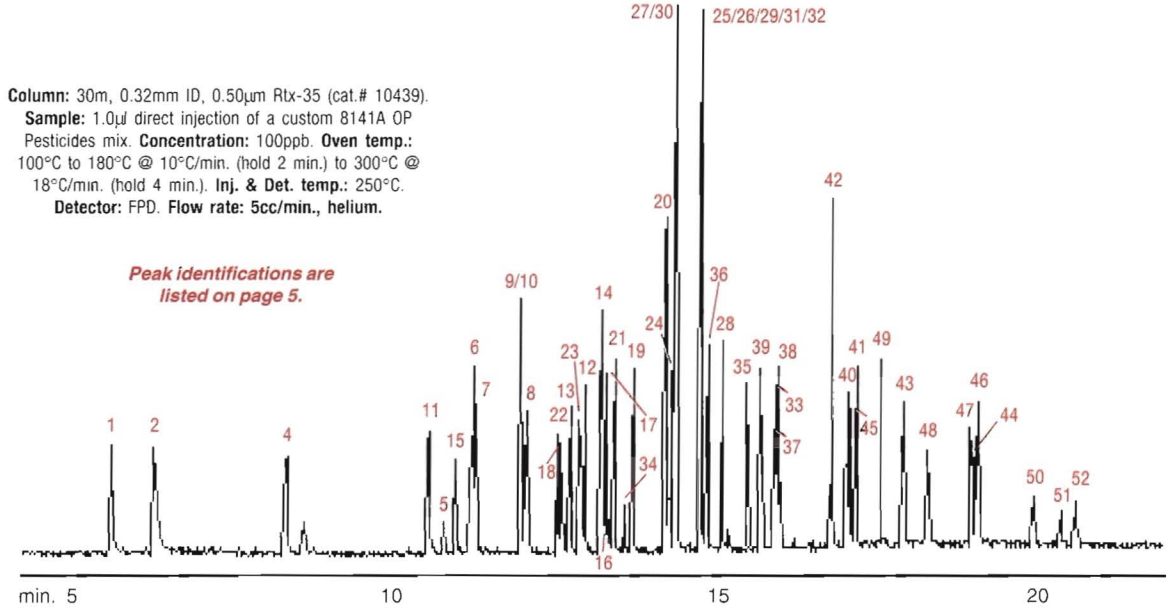


Figure 3:

An analysis time of less than 21 minutes makes the Rtx®-35 an excellent confirmational column.



achieve fast separation of OP pesticides with element-specific detection is used to gain valuable structural information with mass spectrometer detection.

An outstanding confirmational column for OP pesticide analysis is a standard Rtx-35 of the same Rtx-OPPesticides dimension (30m, 0.32mm, 0.5µm). Using the identical temperature and flow conditions shown in **Figures 1 and 2**, the Rtx-35 chromatogram in **Figure 3** has a run time of just over 20 minutes, and resolves all coeluting compounds shown in the Rtx-OPPesticides chromatogram.

The Rtx-OPPesticides is the latest in GC stationary phase innovation. This phase gives an efficient analysis of 55 organophosphorus pesticides in under 20 minutes. And one column can do it all—direct injection on FPD or NPD for high sensitivity and splitless injection on a GC/MS for structural identity. Only from Restek!

[1]C. George, *Separation Times*, 11, 1 (1997) 8-10.

Peak List for Figures 1-3

1. dichlorvos
2. hexamethylphosphoramide
3. trichlofon
4. mevinphos
5. demeton-S
6. zinophos
7. ethoprop
8. phorate
9. naled
10. sulfotepp
11. tributylphosphate (standard)
12. diazinon
13. terbufos
14. fonofos
15. TEPP
16. dioxation
17. disulfoton
18. demeton-O
19. dichlofenthion
20. chlorpyrifos methyl
21. dimethoate
22. dicrotophos
23. monocrotophos
24. ronnel
25. merphos
26. chlorpyriphos
27. aspon
28. fenthion
29. trichloronate
30. methyl parathion
31. malathion
32. fenitrothion
33. tokuthion
34. phosphomidon
35. chlofenvinphos
36. parathion
37. merphos oxone (merphos breakdown product)
38. stirophos
39. crotoxyphos
40. bolstar
41. carbophenthion
42. ethion
43. triphenylphosphate (standard)
44. leptophos
45. fensulfothion
46. tri-o-cresyl phosphate
47. phosmet
48. EPN
49. famfur
50. azinphos methyl
51. azinphos ethyl
52. coumaphos

* phosphomidon breakdown product

Nitrogen-containing compounds
A. 1-bromo-2-nitrobenzene
B. simazine
C. atrazine

Product Listing:

30m, 0.32mm ID, 0.50µm Columns

Rtx®-OPPesticides: Cat.# 11239, $475

Rtx®-35: Cat.# 10439, $415

Analytical Reference Materials

8140/8141 OP Pesticide Calibration Mix A

azinphos methyl
bolstar (sulprofos)
chlorpyrifos
coumaphos
demeton, O and S
diazinon
dichlorvos
disulfoton
ethoprop
fensulfothion
fenthion
merphos
methyl parathion
mevinphos
naled
phorate
ronnel
stirofos
tokuthion (prothiofos)
trichloronate

200µg/ml ea. in hexane/acetone (95%/5%), 1ml/ampul

	Each	5-pk.	10-pk.
	32277 $90	**32277-510** $405	
w/data pack	**32277-500** $100	**32277-520** $450	**32377** $810

8141 OP Pesticide Calibration Mix B

dimethoate
EPN
malathion
monocrotophos
parathion
sulfotepp
TEPP

200µg/ml ea. in hexane/acetone (95%/5%), 1ml/ampul

	Each	5-pk.	10-pk.
	32278 $55	**32278-510** $247.50	
w/data pack	**32278-500** $65	**32278-520** $275	**32378** $495

8140/8141 Internal Standards & Surrogates

1000µg/ml in acetone, 1ml/ampul

1-bromo-2-nitrobenzene Standard

(Recommended 8141A NPD Internal Standard)

	Each	5-pk.	10-pk.
	32279 $20	**32279-510** $90	
w/data pack	**32279-500** $30	**32279-520** $100	**32379** $180

tributylphosphate Standard

(Recommended 8141A FPD Surrogate)

	Each	5-pk.	10-pk.
	32280 $20	**32280-510** $90	
w/data pack	**32280-500** $30	**32280-520** $100	**32380** $180

triphenylphosphate Standard

(Recommended 8141A FPD Surrogate)

	Each	5-pk.	10-pk.
	32281 $20	**32281-510** $90	
w/data pack	**32281-500** $30	**32281-520** $100	**32381** $180

4-chloro-3-nitrobenzotrifluoride Standard

(Recommended 8141A NPD Surrogate)

	Each	5-pk.	10-pk.
	32282 $20	**32282-510** $90	
w/data pack	**32282-500** $30	**32282-520** $100	**32382** $180

SilcoCan™—The Ideal Canister for Sulfur Compound Storage

by Dave Shelow

7-Day Stability at Concentrations as Low as 1 ppm

Sulfur compounds are emitted from a variety of sources including petrochemical processes, land fills, and stack emissions. Because of their odor, these compounds are a nuisance. They frequently require air monitoring and analysis.

Collection of air samples containing trace levels of sulfur compounds is difficult because they readily react with stainless steel sampling vessels such as Summa® Canisters. Because of this reactivity with stainless steel, Tedlar® bags have been used for collection of sulfur compounds. However, the stability of these compounds in Tedlar® bags is limited to 24-48 hours.

Restek's Silcosteel®-lined Silcocan™ canister is the ultimate solution for long term storage of air samples containing sulfur compounds. Silcosteel is a unique process that chemically bonds a layer of fused silica material to the stainless steel surface, reducing adsorption and breakdown of active compounds. The Silcocan air sampling canister has been shown to maintain the stability of trace level sulfur compounds up to seven days with little or no degradation.

A stability study of six common sulfur compounds was recently conducted by the Bay Area Air Quality Management District. These compounds were spiked at two concentration levels into Silcocan air sampling canisters and measured at time intervals of 1, 2, 3, 4, and 7 days. The results of this study are shown in **Figures 2 and 3**. The data clearly shows that even after seven days of storage in a Silcocan canister, over 90% of these six sulfur compounds were successfully recovered.

Since any stainless steel surfaces that come into contact with sulfur compounds will cause adsorption, a Silcocan canister with a Silcosteel-treated valve is recommended. **Figure 4** shows a Silcosteel-treated diaphragm valve. All internal parts that come into contact with the sample have been Silcosteel-treated. Also, any portion of the sampling pathway, such as the flow controller or tubing, should also be Silcosteel-treated. For more information about Restek's Silcosteel process, please contact our Technical Service team or your local Restek representative.

Collection and storage of highly adsorptive sulfur compounds is no longer a problem with Restek's Silcocan canister. Silcosteel technology reduces the adsorptive characteristics of stainless steel. Even trace levels of sensitive sulfur compounds can be stored for up to seven days without significant loss using Restek's innovative technology.

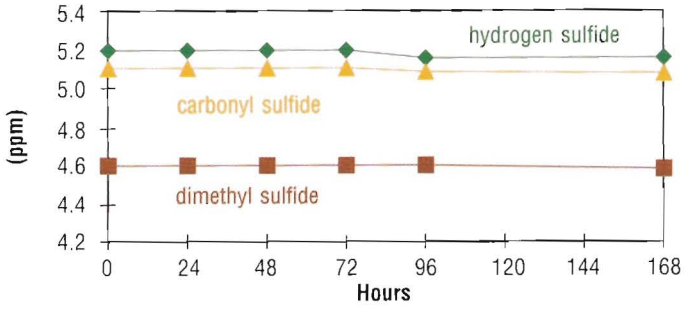
Figure 1:

The Silcosteel® lining in the SilcoCan™ canister reduces adsorption of sulfur compounds.

For more information, request a free copy of Restek's Air Monitoring Products Guide.

Figure 2:

No significant loss of sulfur compounds when stored in a SilcoCan™ for up to 7 days.



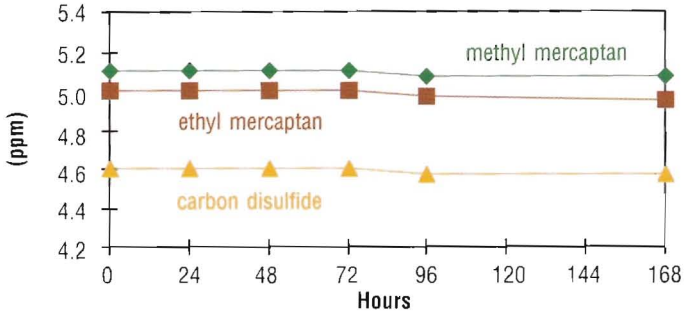
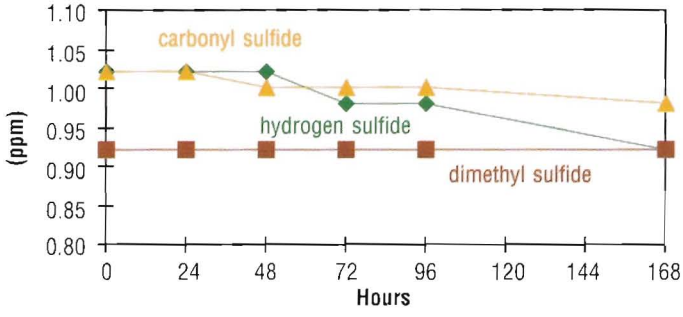
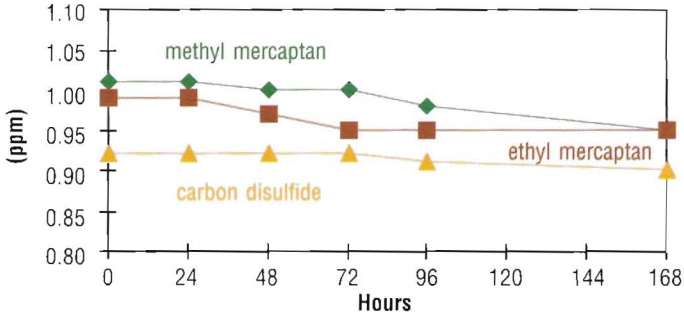


Figure 3:

Even 1ppm of sulfur compounds is recovered from a SilcoCan™ canister after 7 days.

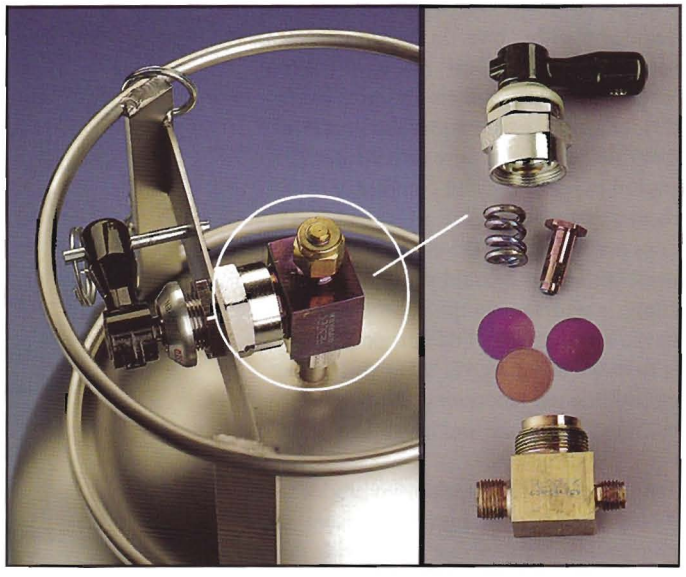




Since any stainless steel surfaces that come into contact with sulfur compounds will cause adsorption, a Silcocan canister with a Silcosteel-treated valve is recommended.

Figure 4:

A Silcosteel®-treated diaphragm valve insures a completely inert sample pathway.



Product Listing:

SilcoCan™ Canisters with Silcosteel®-treated Valves

Sizes	Cat.#	Price
1.0 Liter	24201-650	$510
1.8 Liter	24202-650	$520
3.0 Liter	24203-650	$530
6.0 Liter	24200-650	$550
15.0 Liter	24204-650	$850

Silcosteel® Replacement Diaphragm Valve: cat.# 24221, $305

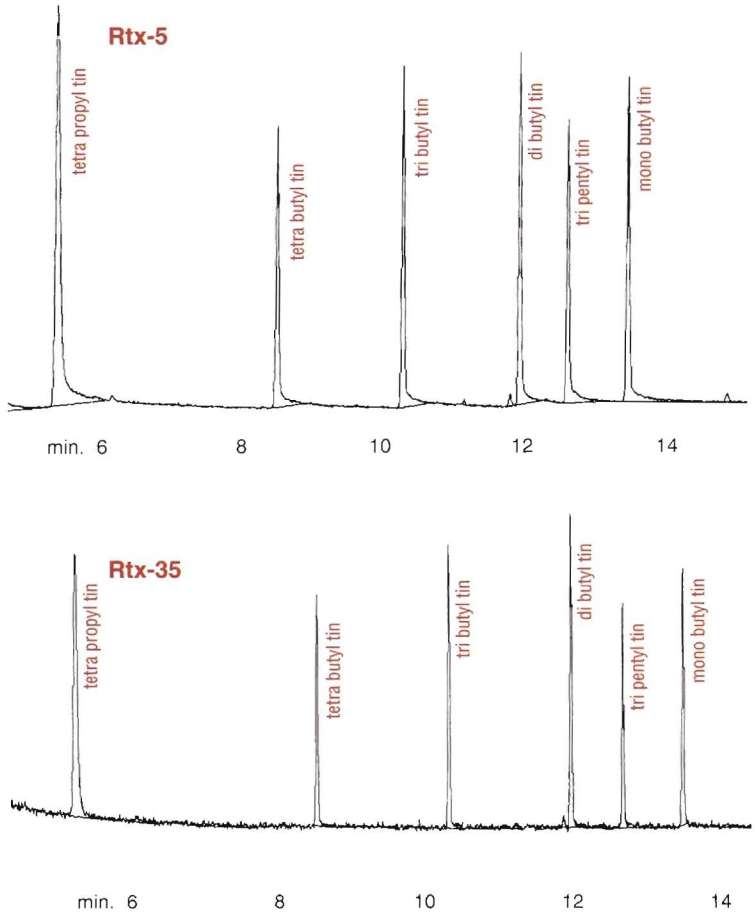
Organo Tin Analy

by Frank Dorman

Tributyl tin was commonly used as an antifouling agent in marine paint, as well as a pesticide and fungicide before its use was discontinued in the 1980's. Tributyl tin has since been found to bioaccumulate and cause a number of health-related problems, and has been recently added to the growing list of possible endocrine disrupting compounds.[1] As awareness of endocrine disrupters grows, and shipyards are remediated, many environmental laboratories are faced with requests for the analysis of tributyl tin and its breakdown products. Unfortunately, there is no "EPA accepted" method for this, so most laboratories have decided to either pass on these requests, or subcontract the analysis to one of the few laboratories that perform this test. Generally, laboratories are under the impression that this analysis requires a considerable capital expenditure and complex techniques that would result in high cost. This does not have to be the case, however, and most laboratories could perform this analysis with the equipment they already have. The purpose of this proposed method is to make this analysis "available" to an environmental laboratory at low cost using common glassware and instrumentation.

Figure 1/2:

500 pg Organo Tin Compounds on the Rtx®-5 and Rtx®-35 Columns by GC-FPD.



30m, 0.32mm ID, 1.0µm Rtx-5 and Rtx-35 columns (cat.# 10254 and 10454)
3µl direct injection. **Concentration:** ~500pg on-column. **Head pressure:** 15 psi, constant.
Oven temp.: 100°C (hold 1 min.) to 285°C @ 10°C/min. hold 10 min., **Inj. & det. temp.:** 250°C; **Carrier gas:** He

Tributyl tin and its breakdown products of dibutyl tin and monobutyl tin present a preparation problem due to the wide range of polarity. These compounds usually exist as chlorides, and it is difficult to completely extract all of them quantitatively from the sample matrix, although there has been some promising data from open-vessel microwave extraction techniques. What is possible, however, is to quantitatively extract the tetra, tri, and di butyl tin, and achieve reasonable and reproducible extraction of the monobutyl tin.

For this method it is very important to remove as much of the potential interferents as possible through a thorough extract cleanup. The primary interference is from sulfur-containing compounds, and these can be at relatively high concentration compared to the organo tin compounds. The 16 gram Florisil and 5 gram silica gel method[2] has a large capacity and works well for all three sample matrices, water, soil, and biota. The cleanup column can be made in glass prep-scale chromatography columns, or purchased as SPE cartridges from Restek (cat.# 53305). In either case, the extract is applied to a hexane-wetted column, and eluted using 100 mls of hexane. The extract is again collected and the internal standard, tetra-n-propyl tin, is added before final concentration.

There are many reported methods of analysis in the literature, but since the goal of this method was to be adaptable to an environmental laboratory, gas chromatography (GC) with flame photometric detection (FPD) was chosen. The FPD must be operated under fuel-rich conditions for efficient conversion of the alkyl tin compounds into tin hydrides. The only other necessary modification is to use a

ic by Capillary GC

New Method from RESTEK!

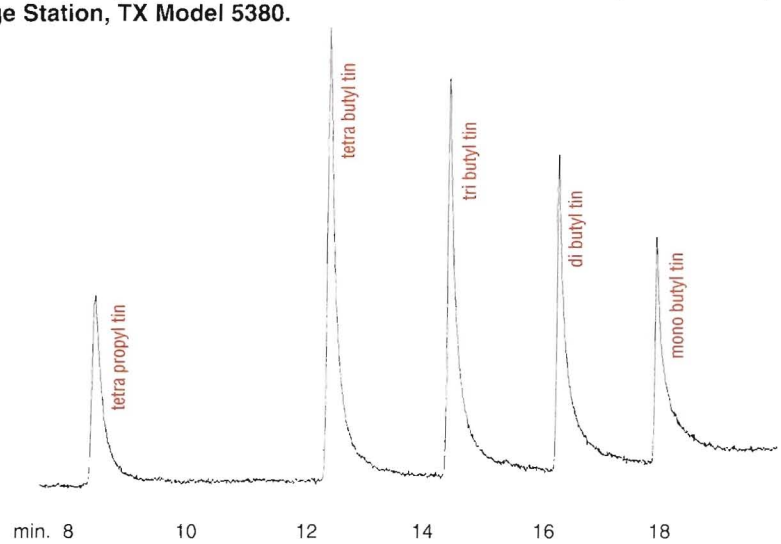
Figure 3:

Commercial Laboratory Results using the Proposed Method from Restek.

Compound	Water Extraction Recovery (%)	Soil Extraction Recovery (%)	Restek SPE-Cleanup Recovery (%)	MDL Liquid (ng/L)	MDL Soil (µg/Kg)
tetrabutyl tin	83	86	92	29.9	0.45
tributyl tin	110	96	99	20.9	0.39
dibutyl tin	75	66	96	15.7	0.46
tripentyl tin (SSTD)	NA	NA	101	NA	NA
monobutyl tin	38	36	118	19.6	0.14

Figure 4:

Organo Tin compounds using Pulsed FPD. Detector courtesy of O.I. Analyical , College Station, TX Model 5380.



30m, 0.32mm ID, 1.0µm Rtx-35 (cat.# 10454). **Concentration:** 5pg on-column. **Head pressure:** 15 psi, constant. **Oven temp.:** 100°C (hold 1 min.) to 285°C @ 10°C/min. hold 10 min., **Detector:** PFPD from O.I. Analytical Corp.

610 nm wavelength filter to collect the molecular emission from the tin hydride. Tin chlorides are analyzed as hexyl derivatives which are formed by a Grignard reaction using n-hexyl magnesium bromide. The surrogate tri-pentyl-tin chloride is added to the sample prior to extraction and tetra-n-propyl-tin is the recommended internal standard. The calibration compounds, surrogate and internal standard solutions are available from Restek as custom reference materials.

Figures 1 and 2 show the resulting chromatograms from the mid-point calibration standard on the Rtx-5 and the Rtx-35 columns by GC-FPD. **Figure 3** shows the method performance obtained by a commercial laboratory using this method.[3]

The method presented easily meets the requirements of 50 ng/L. In order to meet a possible proposed detection limit of 1ng/L, some method modification will be necessary. The easiest modification would be to switch from using a regular FPD to a pulsed FPD (PFPD) detector. This detector gives a sensitivity enhancement of 10 to 100 times over the standard FPD for the organo tin compounds. **Figure 4** shows the chromatogram obtained for 5 pg of each tin compound on the Rtx-35 using PFPD detection. Comparing this chromatogram to **Figure 1 and 2** it is observed that a similar signal to noise ratio is obtained with 100 times less material. This demonstrates roughly an increase of 100 in sensitivity using the PFPD with the same preparation method, resulting in the ability to meet the proposed 1 ng/L detection limit being considered by the EPA.

In summary, this method allows laboratories to perform organo tin analysis with minimal start up and implementation costs. It is reliable, rugged, and utilizes equipment that most laboratories already have. To meet current and proposed detection limits, it is not necessary to use tandem MS or GC-AED which have high purchase and operating costs, and are typically not found is most laboratories. Finally, Restek can provide the technical training, and supplies required to perform this method so that literature and vendor research is not required.

[1] Special Report on Environmental Endocrine Disruption: An Effects Assessment and Analysis, EPA/630/R-96/012, Feb. 1997.
[2] Sampling and Analytical Methods of the National Status and Trends Program, National Benthic Surveillance and Mussel Watch Projects 1984-1992, Vol. IV, NOAA Technical Memorandum, NOS ORCA 71
[3] ITS - Environmental, 55 South Park Drive, Colchester, VT 05446.

Product Listing:

Columns and Accessories

30m, 0.32mm ID, 0.50µm Rtx-5: cat.# 10239, $415
30m, 0.32mm ID, 0.50µm Rtx-35: cat.# 10439, $415
Florisil/silica gel SPE cartridge: cat.# 24049, $120 16-pack

Koni's Korner

Carrier Gases for GC

by Dr. Konrad Grob

Probably more than 90% of the present GC instruments run with helium as carrier gas. Some people use hydrogen or nitrogen, maybe because the first ones are hidden pyromaniacs (some GC ovens actually exploded) and the second still have nitrogen mounted on the instrument from the times they worked with packed columns. These gases serve to produce wind through the column to move our solutes forward. The solute molecules evaporate from the stationary phase surface, i.e. enter the open space of the capillary column, are hit by a carrier gas molecule and start traveling down the tube. After a short distance, however, they touch the sticky surface of the stationary phase and go through another partitioning process. Does the choice of the carrier gas interfere with this? Yes, it does, through its diffusivity and viscosity. You want to know why hydrogen is the best carrier gas?

Diffusivity

Diffusivity provides a measurement for the diffusion speed of a solute vapor in a given gas. For helium and hydrogen, diffusivities are similar, but that of nitrogen is about four times lower (see **Table I**).

The diffusion speed of the solute in the carrier gas determines the speed of chromatography. A solute molecule evaporating from the stationary phase surface into the gas stream should be given enough time to diffuse back to the stationary phase (**Figure 1**) before having gone far in order to undergo another partitioning process - it is these contacts which differentiate between different substances, and a large number of contacts are needed to obtain the best separation. We get more of them if the solute diffuses more rapidly and/or when we give it more time, i.e. reduce the gas velocity. However, there is a limit: giving it more time for the diffusion towards the stationary phase (radial diffusion) also provides more time for spreading within the open bore of the column, i.e. for band broadening through longitudinal diffusion. This is why there is an optimum gas velocity: it provides a maximum number of contacts with the stationary phase with a minimum of band broadening in the gas phase.

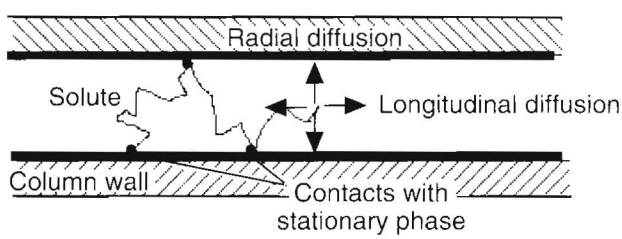
Table I:

Relevant characteristics of carrier gases[1]

Carrier gas	Viscosity at 50°C [kg/s m]	Diffusivity (butane, 100°C [m2s])
Hydrogen	9.4	$6\ 10^{-6}$
Helium	20.8	$5.5\ 10^{-6}$
Nitrogen	18.8	$1.5\ 10^{-6}$

Figure 1:

Diffusion of a molecule in the gas phase of the column.



This kind of logic applies to all gases. In fact, all carrier gases provide similar separation efficiencies - provided conditions are adjusted correspondingly. The time needed is different: since diffusion in hydrogen and helium is much faster than in nitrogen—for (wanted) radial as well as (unavoidable) longitudinal diffusion—GC is 2-3 times faster with the former. If we users of hydrogen wait for one hour, users of nitrogen should wait for 2-3 hours to get the same performance. Nitrogen is for those who own a comfortable arm chair in the lab or who are afraid of the result. Usually users of nitrogen are not really that patient and run their chromatography at similar speed as others using hydrogen and helium. **Table II** shows what they get. It compares separation efficiencies measured in terms of Trennzahl (TZ) indicating the number of peaks which could be fully separated between two components to be defined, in this case, the alkanes C_{13} and C_{14}. At the gas velocities most commonly used with hydrogen (40-60 cm/s), nitrogen produced hardly more than half as many peaks. When using

Table II:

Separation efficiencies in terms of separation numbers (Trennzahl, TZ) for the n-alkanes C_{13} and C_{14} and a 12m, 0.25mm ID column coated with a methyl silicone.

Gas velocity	Hydrogen	Nitrogen
50 cm/s	24	13
40 cm/s	25	15
30 cm/s	23	17
20 cm/s	20	23

hydrogen, the same result could have been obtained from a column roughly 3 times shorter in a third of the time. To give an impression of how the chromatograms look like, an example is shown in Figure 2. At halved velocity, nitrogen provided good performance also.

In this application, nitrogen just requires extra time. However, long retention times also produce low peaks, i.e. poor sensitivity (see **Figure 2**). Additionally, do not try to run triglycerides or other labile compounds with nitrogen as carrier gas: they are largely degraded during the long run time required.

Viscosity

The other difference between the carrier gases concerns the viscosity that determines the inlet pressure required for a given gas velocity. High inlet pressures strongly compress the gas in the column inlet, which causes the problems shortly outlined below. This explains why hydrogen is preferable to helium.
You have certainly seen the h/u curves, also called van Deemter curves, plotting HETP (plate height) against gas velocity. Their peculiarity: the best is at the bottom, i.e. the optimum gas velocity is at the lowest point of the curve; the larger the plate heights, the worse the separation. The curves say that separation is poor when the gas velocity is below the optimum velocity (left of the optimum in **Figure 3**, the result of excessive longitudinal diffusion) and that it worsens again beyond that optimum (the curve rising at the right, the result of insufficient radial diffusion).

For columns of a given diameter, the optimum velocity is highest when the column is short. This is because inlet pressure is low. For hydrogen or helium, with about the same diffusivity, the optimum is almost the same, i.e. around 40-50 cm/s. Further, the losses in performance upon speeding, i.e. using excessive gas velocity, are relatively small. The longer the column, the higher is the inlet pressure required. This shifts the optimum gas speed to lower values and, as if there were a strict educator behind the chromatographer, speeding is punished more strongly when the velocity must be low anyway. Hence, using a column of doubled length requires more than twice as much run time, because the gas velocity must be lower. In this respect, helium is worse than hydrogen because its viscosity is about twice as high: the higher inlet pressure requires a lower gas velocity and if you do not obey, the punishment is harder.

Figure 2:

Separation of a kerosene fraction using hydrogen or nitrogen as carrier gas at the same average gas velocity (40 cm/s).

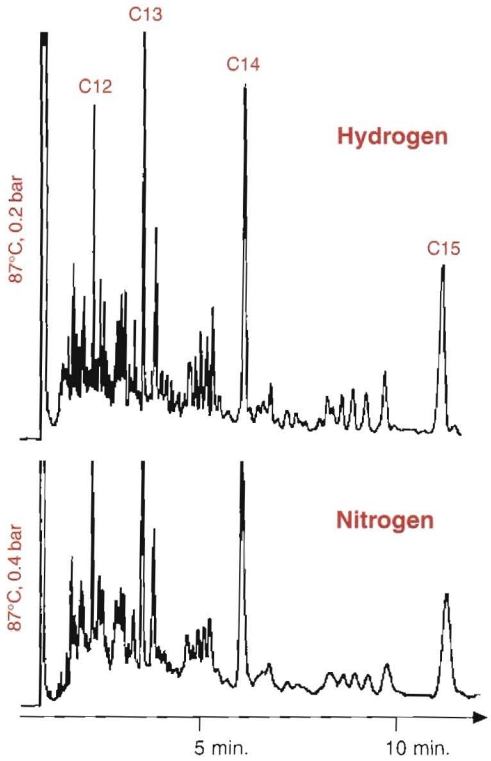
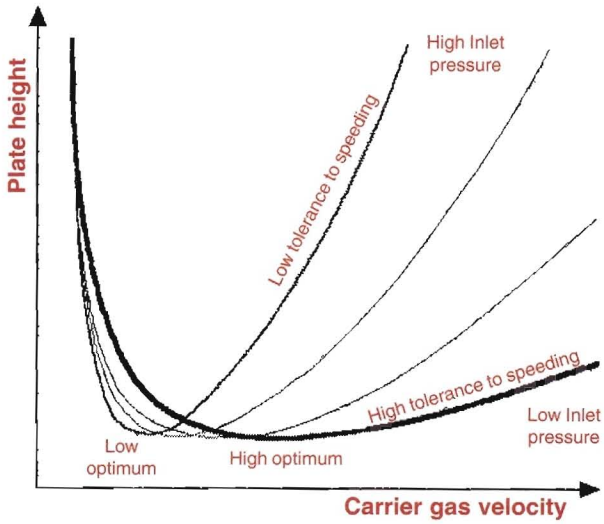


Figure 3:

High inlet pressures cause the optimum gas velocity to be low and the loss in separation efficiency when exceeding this optimum to be high.



Koni's Korner

Continued from page 11.

What is the reason for this? If the column head pressure is, e.g., 1 bar, corresponding to 2 bar absolute pressure, the carrier gas in the inlet is compressed to half the volume compared to the column outlet (assuming the latter is at ambient pressure, 1 bar absolute, **Figure 4**). Hence the plug corresponding to 2 ml in the outlet is only 1 ml and is half as long. To displace 1 ml, half the velocity is required compared to displacing 2 ml at the outlet. Hence optimization must compromize between a low velocity in the inlet and a higher one at the outlet.

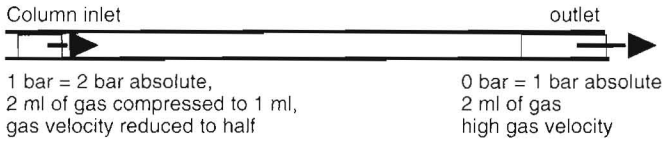
Conclusions are against intuition. From short columns we know that 40-50 cm/s are best. In the last, e.g., 15 m of a long column, pressure conditions are the same as in a short column, i.e. the optimum gas velocity and tolerance to speeding must be the same. The problem resulting from the compressibility of the gas is obviously in the inlet of the long column. We are tempted to assume that it is related to the fact that the gas velocity is 20-25 cm/s only and would conclude that a compromize should be chosen between maybe 30 cm/s in the inlet and 70 cm/s in the outlet in order to result in some 50 cm/s as an average. Experiments show that this is wrong: the best average velocity is only 20-25 cm/s. Hence the system wants an even lower velocity in the inlet: about 10 cm/s. And it insists in that: it forces to choose a velocity at the outlet lower than found to be optimum, and if you do not obey to the 10 cm/s in the inlet, punishment is hard. A rapid glance into the above h/u curve shows that 10 cm/s would provide extremely poor performance at the column outlet. Thus the correct conclusion is that optimum velocities are far lower in a compressed gas. This is not really new: GC with vacuum at the outlet, e.g. with GC-MS, is even faster.

Nitrogen has only drawbacks and is not suitable for capillary GC. Helium is as good as hydrogen if inlet pressures are below about 50 kPa, but requires slower GC at higher inlet pressures (for longer columns), the difference being roughly a factor of two when 150-200 kPa must be applied for helium.

The most important argument against the use of hydrogen concerns safety. The next "Korner" will report on how our lab solved that problem.

Figure 4:

Compressibility of the carrier gas causes the gas velocity in the inlet to be lower than in the outlet.



[1]*from Rohrschneider, Ullmanns Enzyklopädie der technischen Chemie, Vol. 5.*

I welcome your feedback. Reach me by e-mail at Koni@grob.org.

Capillary GC Reference Books

by Dr. Konrad Grob

On-Column Injection in Capillary Gas Chromatography, 2nd Edition

Basic Technique; Retention Gaps; Solvent Effects *(Konrad Grob)*

On-column injections minimize detrimental adsorption and non-linearity problems associated with split/splitless techniques. Grob's text is a *must*-read treatise for the novice as well as for the experienced chromatographer. Basic technique is explained clearly with excellent schematics.

Huethig Publishing, Ltd., 1987 • 591pp.
cat.# 20453, $130.75 ea.

Split and Splitless Injection in Capillary GC, 3rd Edition

(Konrad Grob)

Represents one of the most comprehensive, single-volume treatment of all aspects of split and splitless injection. The book is divided into four sections: split injection, splitless injection, problems arising from the heated syringe needle in vaporizing injection, and Programmed Temperature Vaporizing (PTV) injection.

Huethig Publishing, Ltd., 1993 • 547pp.
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Figure 1:

Sulfa Drugs

1. sulfathiazole
2. sulfamethizole
3. sulfadimethoxine
4. sulfaquinoxaline

Inertsil ODS-2
5µm, 150 x 4.6mm ID
(cat.# 9000006). **Eluent:** 5mM 1-heptanesulfonate Na in 28% CH_3OH. **Flow rate:** 1.2 mL/min. **Detector:** 254nm. **Col. temp.:** RT.

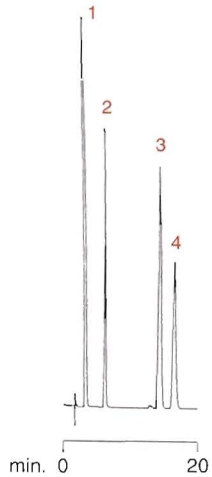
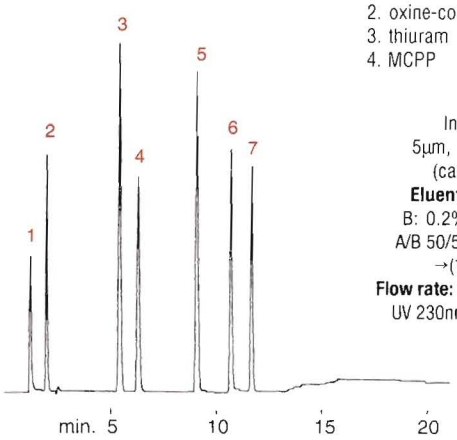


Figure 2:

Golf Course Pesticides

1. asulam
2. oxine-copper
3. thiuram
4. MCPP
5. iprodione
6. bensulide
7. pencycuron

Inertsil ODS-3
5µm, 150 x 4.6mm ID
(cat.# 9000001).
Eluent: A: 0.2% H_3PO_4
B: 0.2% H_3PO_4 in CH_3CN
A/B 50/50→(10min)→10/90 →(10min)→10/90
Flow rate: 1.0 mL/min. **Detector:** UV 230nm. **Col. temp.:** 40°C.



Each lot of Inertsil reverse-phase bonded phases must pass several critical tests for inertness and stability prior to release for sale. First, packings are evaluated for inertness by observing the elution order and peak symmetry of pyridine in a mixture with phenol, methylbenzoate, and 3-methylindole. Then, a mixture of oxine copper and caffeine is injected and evaluated for peak shape and reproducibility to check for residual metals that can irreversibly adsorb certain compounds. After a check for selectivity, all bonded phases are subjected to both pH 2 and pH 9 to determine their chemical stability. Finally, columns are packed using proprietary techniques developed to produce highly efficient, reproducible HPLC columns.

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Product Listing:

Inertsil Columns

Phase	Particle Size	Dimensions	Cat.#	Price
ODS-3	5µm	150 x 4.6mm	9000001	$360
	5µm	250 x 4.6mm	9000002	$395
ODS-2	5µm	150 x 4.6mm	9000006	$360
	5µm	250 x 4.6mm	9000007	$395
C8	5µm	150 x 4.6mm	9000010	$360
	5µm	250 x 4.6mm	9000011	$395
C4	5µm	150 x 4.6mm	9000014	$360
	5µm	250 x 4.6mm	9000015	$395
Phenyl	5µm	150 x 4.6mm	9000018	$360
	5µm	250 x 4.6mm	9000019	$395
Silica	5µm	150 x 4.6mm	9000022	$360
	5µm	250 x 4.6mm	9000023	$395

Inertsil Guards

Phase	Particle Size	1cm Cartridges (Pack of 2)		Guard Set (Holder + Guards)	
ODS-3	5µm	9000004	$160	9000005	$250
ODS-2	5µm	9000008	$160	9000009	$250
C8	5µm	9000012	$160	9000013	$250
C4	5µm	9000016	$160	9000017	$250
Phenyl	5µm	9000020	$160	9000021	$250
Silica	5µm	9000024	$160	9000025	$250

Cartridge Guard Holder: cat.# 9000003, $110

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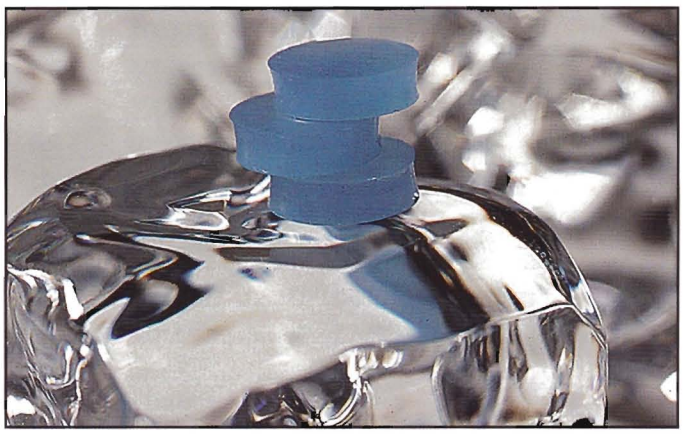

Product Listing:

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10mm	22390	$35	22391	$65
11mm	22392	$35	22393	$65
12.5mm	22394	$35	22395	$65
17mm	22396	$35	22397	$65
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Description	Cat. #	Price
PID Lamp, Model 103 C	20676	$390
PID Lamp, Model 108-10.0/10.6	20675	$479
PID Lamp Cleaning Kit	20674	$15

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

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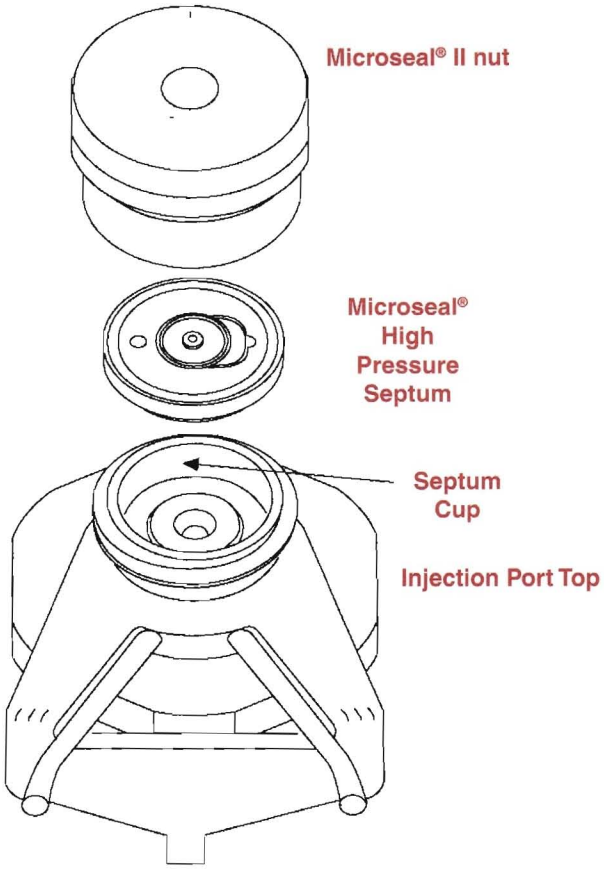
INSTALLATION



Product Listing:

Merlin Microseal® High Pressure Septa

Description	Cat.#	Price	Merlin#	Similar to HP#
Nut Kit (1 Nut, fits 300 & 400 series septa):	22809	$100	403	5182-3445
Standard Kit (Nut, 2 High Pressure Septa):	22810	$340	404	Not offered
Starter Kit (Nut, 1 High Pressure Septum):	22811	$240	405	5182-3442
High Pressure Replacement Septa (1-septum):	22812	$140	410	5182-3444

Merlin Microseal® Septa

Description	Cat.#	Price	Merlin#	Similar to HP#
Standard Kit (Nut, 2 Septa):	22813	$250	304	5181-8833
Starter Kit (Nut, 1 Septum):	22814	$190	305	5181-8816
Microseal Replacement Septa (1-septum):	22815	$90	310	5181-8815
Replacement PTFE Washers (2-pack):	22808	$5	311	5181-0853

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ASTM E-19 Meeting...Oct. 12-17, San Diego, CA.

Northeast Regional Chromatography Discussion Group (NERCDG)...Oct. 15, Rochester, NY.

Northeastern Association of Forensic Scientists...Oct. 16-18, White Plains, NY.

FACSS '97...Oct. 25-31, Providence, RI.

California Association of Toxicologists...Nov. 3-7, Las Vegas, NV.

Eastern Analytical Symposium...Nov. 16-21, Somerset, New Jersey.

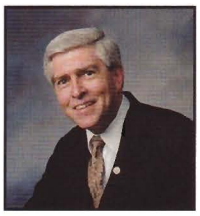
Chiral GC Seminar...Nov. 21, E. Brunswick, New Jersey.

Literature cat.# 59711

Welcome Jingzhen Xu and Doug Elliott!

Restek is pleased to have Jingzhen Xu join its dynamic research and development team. Jingzhen received his bachelor in chemical engineering from Tsinghua University, Master in chemistry from Beijing Polytechnic University and Ph.D. from Southern Illinois University at Carbondale.

Jingzhen has extensive experience in chromatography, especially multidimensional gas chromatography. His knowledge in hydrocarbon analysis and instrumentation will help him develop new products and applications. He is now working on PLOT columns. If you would like to discuss your PLOT column applications, don't hesitate to call Jingzhen at extension 2158.



During 10 years in analytical Services at Air Products, Doug was involved in various disciplines including gas chromatography and instrumental microanalysis. He left to join the instrument and supplies side of analytical chemistry as Manager of Sales and Marketing at Control Equipment Corp. and then Accessories Marketing Manager at Supelco. Doug's experience has provided him with the skills to develop new products and the personal contacts in our industry to bring the best commercial products to you. Doug is here to support you with your questions to optimize the performance of your chromatography accessories as well as to develop products that better meet your needs. Call him at extension 2159.