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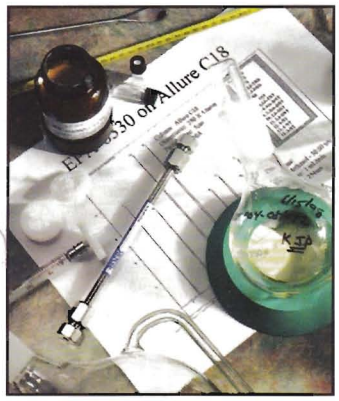
Innovators of High Resolution Chromatography Products

LC/MS COLUMNS

New, Highly Retentive HPLC Columns Designed for LC/MS

by David Bell and Keith Duff, Senior Research Chemists

Liquid chromatography/mass spectrometry (LC/MS) has developed into one of the most powerful analytical techniques available. Although the MS can, in itself, provide enhanced selectivity, quality chromatographic separations are essential in order to take full advantage of the technique. Many column manufacturers are simply repackaging and marketing their existing, standard HPLC phases as LC/MS columns. Restek, on the other hand, is actively involved in real-world research and development of new LC/MS stationary phases and columns.



Among the advantages of LC/MS technology is an increased level of sensitivity compared to traditional ultraviolet (UV) detection. For electrospray-interfaced LC/MS systems, the sensitivity is related to the efficiency of the ionization/evaporation process. A greater percentage of organic modifier in the mobile phase improves the ionization/evaporation efficiency and thus provides increased sensitivity.

The Allure™ family of HPLC columns has been designed to provide the high retention and resolution required for successful LC/MS method development. Research efforts have concentrated on obtaining maximum retention for analytes based on their functional group, *(cont. on pg. 2)*

Figure 1 **LC/MS Column Selection Based on Analyte Functional Group.**

High Bonding Density of Conventional HPLC Phase Chemistries

Novel Bonding Chemistries Engineered for Maximum LC/MS Sensitivity

Analyte Functional Group

Neutral/ Slightly Polar	Base	Acid/Amino Acid	Very Polar
Allure™ C18 Ultra IBD	Allure™ Basix Ultra IBD	Allure™ Acidix Ultra IBD	Allure™ Acidix Ultra IBD

Neutral/ Slightly Polar	Base (Low MW)	Base (High MW)	Acid (Low MW)	Acid (High MW)	Amino Acid
Ultra C18 Ultra C8 Ultra C4 Ultra C1 Ultra CN Ultra Phenyl	Ultra Phenyl Ultra CN (NP) Ultra Silica (NP)	Ultra C18 Ultra C8 Ultra C4 Ultra C1 Ultra CN Ultra Phenyl	Ultra CN (NP) Ultra Silica (NP)	Ultra C18 Ultra C8 Ultra C4 Ultra C1 Ultra Phenyl	Needs Derivatization

Low Molecular Weight (MW) = <200 Daltons High Molecular Weight (MW) = >200 Daltons NP = Normal Phase

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LC/MS Columns (continued from pg. 1)

without compromising quality and reproducibility. The research produced three superior products—Allure™ C18, Allure™ Basix, and Allure™ Acidix columns—that target four classes of analytes as diagrammed in Figure 1 (front). Because these columns retain analytes longer than conventional columns, a higher percentage of organic modifier in the mobile phase may be employed. The result is a significant increase in MS sensitivity (Figure 2).

We also offer Ultra HPLC columns. The high-percentage carbon and phase coverage provides heightened solute retention compared to other manfacturers' columns. Additionally, the bonding chemistry of each phase is reproducible and reliable.

Try unique stationary phase chemistries for enhanced, state-of-the-art selectivity. We offer the ultimate in HPLC column quality and reproducibility, guaranteed lot-to-lot confidence, and minimal phase bleed.

For Allure™ columns with Trident™ inlet fittings and Allure™ guard cartridges, as well as a complete listing for Ultra columns, please request the *LC/MS Columns Catalog* (lit. cat. #59607*), or visit our web site at www.restekcorp.com.

*International customers please request lit. cat.# 59606.

Product Listing

Allure™ Acidix Columns

Particle Size: 5µm	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm length	9162531	9162532	9162533	9162535
50mm length	9162551	9162552	9162553	9162555
100mm length	9162511	9162512	9162513	9162515
150mm length	9162561	9162562	9162563	9162565
200mm length	9162521	9162522	9162523	9162525
250mm length	9162571	9162572	9162573	9162575

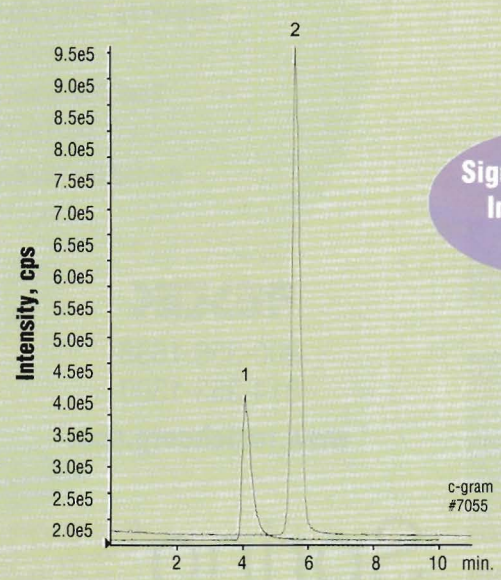
Allure™ C18 Columns

Particle Size: 5µm	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm length	9164531	9164532	9164533	9164535
50mm length	9164551	9164552	9164553	9164555
100mm length	9164511	9164512	9164513	9164515
150mm length	9164561	9164562	9164563	9164565
200mm length	9164521	9164522	9164523	9164525
250mm length	9164571	9164572	9164573	9164575

Allure™ Basix Columns

Particle Size: 5µm	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm length	9161531	9161532	9161533	9161535
50mm length	9161551	9161552	9161553	9161555
100mm length	9161511	9161512	9161513	9161515
150mm length	9161561	9161562	9161563	9164[illegible]
200mm length	9161521	9161522	9161523	91[illegible]
250mm length	9161571	9161572	9161573	9161[illegible]

Figure 2 Achieve 240% Signal-to-Noise Increase Using an Allure™ Basix LC/MS Column.



Signal-to-Noise Increase of 240%!

Amitriptyline LC/MS

Mass Spectrometer: PE SCIEX API 150 EX
LC/MS Interface: PE SCIEX TURBO IONSPRAY®
Flow: 0.2mL/min.
Injection: 25ng Amitriptyline HCl

1) Column: Conventional C18, 50 x 2.1mm, 5µm

Mobile phase: 5mM NH_4CH_3COO, pH 4.5: ACN (65:35, v/v)
Intensity: 288373.72 cps
Area: 2.794 x10^5 cps^2
Noise: 580.620 cps

2) Column: Allure™ Basix, 50 x 2.1mm, 5µm

Mobile phase: 5mM NH_4CH_3COO, pH 4.5: ACN (20:80, v/v)
Intensity: 962220.81 cps
Area: 1.544 x 10^7 cps^2
Noise: 798.673 cps

0.45mm ID Rtx®-502.2 Column Provides Faster GC Volatile Analysis

by Christopher English, Environmental Applications Chemist

The Rtx®-502.2 column is one of the most popular choices for a long list of volatile Environmental Protection Agency (EPA) methods. An optimized 0.45mm ID Rtx®-502.2 column now provides even better resolution and faster analysis times. The Rtx®-502.2 column originally was designed to address the growing number of compounds that the EPA added to its GC volatile methods; namely the transition from Methods 502.1/503.1 (40 compounds) to Method 502.2 (60 compounds).

Although the 502.2 method has remained the same, many of the SW-846 8000-series methods have changed the target analytes. Additionally, states have required monitoring of other compounds, such as methyl-*tert*-butyl ether (MTBE), a gas additive, and Freon®113. The most recent change is the revision of Method 8021A to Method 8021B. This newly promulgated method removes 13 compounds from the previous list—mostly the branched aromatics—and adds 10 more target analytes. Many environmental labs are interested in analyzing the 502.2 compound list, the added compounds introduced in Method 8021B, and state-regulated compounds. This results in a target list that may exceed 70 compounds.

The new 0.45mm ID Rtx®-502.2 column has excellent resolution for the volatile compounds and a 15-minute faster analysis time compared to the 0.53mm ID equivalent. Figures 1 and 2 illustrate optimized run conditions using a 75m, 0.45mm ID, 2.55µm df, Rtx®-502.2 column. By using a smaller internal diameter, a faster run is possible with enough column flow to effectively sweep the volatiles off of the trap, resulting in excellent chromatography.

Advantages of the Rtx®-502.2 Column

The Rtx®-502.2 column is unique in that it resolves

Analysis Conditions for Figures 1 & 2

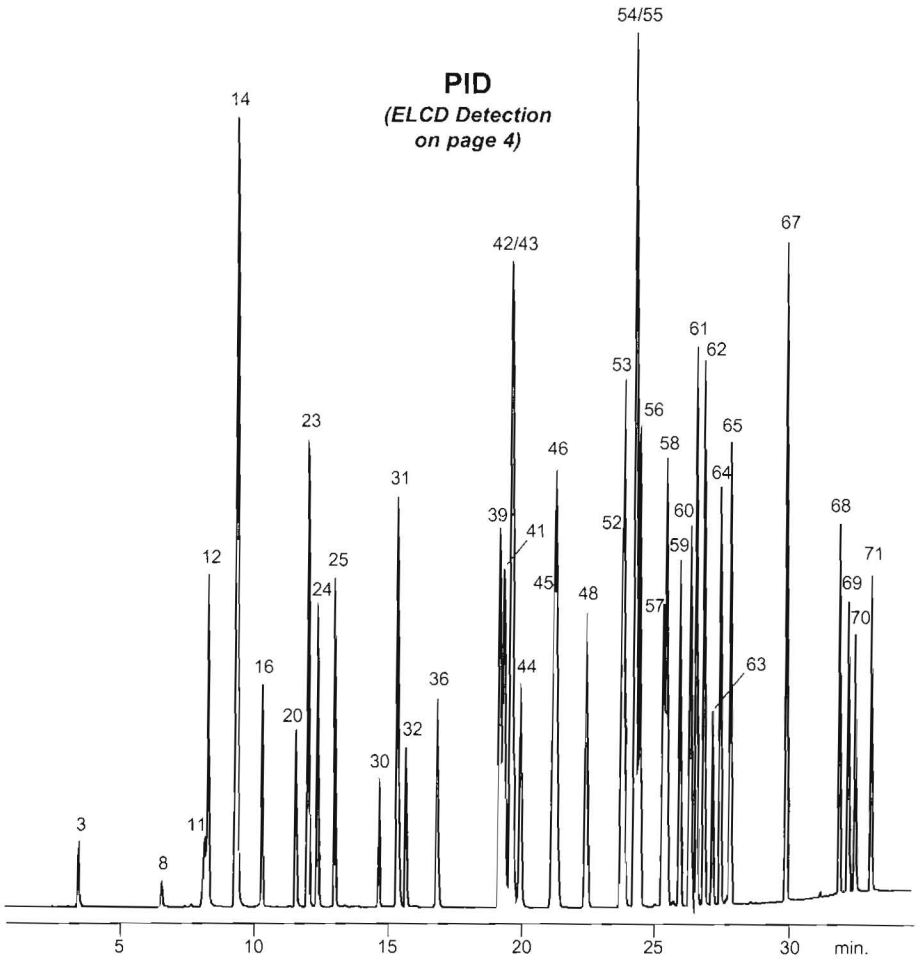
Column: 75m, 0.45mm ID, 2.55µm Rtx®-502.2 (cat.# 10986).
Concentration: 20ppb in 5mL of RO water (unless otherwise noted, peak 14).
Injection: a combination of the following reference materials was used:
502.2 Cal2000 MegaMix™ (cat.# 30431); 502.2 Calibration Mix #1A (cat.# 30439); 502.2 Internal Standard Mix #2 (cat.# 30041); 1-chloro-2-fluorobenzene (cat.# 30040); 4-bromochlorobenzene (cat.# 30230); 2-chlorethyl vinyl ether (cat.# 30265); 1,4-dichlorobutane (cat.# 30227); MTBE (cat.# 30402); and custom mixtures of Freon® 113, allyl chloride, chloroprene, and benzyl choride.

Purge & Trap concentrator: Tekmar LSC-3000 purge and trap; **Trap:** VOCARB™ 3000; **Purge:** 11 min. @ 40mL/min.; **Dry purge:** 1 min. @ 40mL/min. (MCS off); **Desorb preheat:** 245°C; **Desorb:** 250°C for 2 min.; **Bake:** 260°C for 8 min.

GC: Finnigan 9001
Oven temp: 35°C (hold 6 min.) to 115°C @ 11°C/min. (hold 7 min.) to 130°C @ 7°C/min. (no hold), to 220°C @ 9.2°C/min. (hold 4 min.); **Carrier gas:** helium (9mL/min.); **Detectors:** µGold Tandem PID/HALL; **PID:** makeup 7mL/min., purge 7mL/min. set @ 0.35mV, base temp. 200°C; **ELCD:** (Hall 2000) Rxn gas 25mL/min., Rxn temp. 940°C, propanol flow 470µL/min.

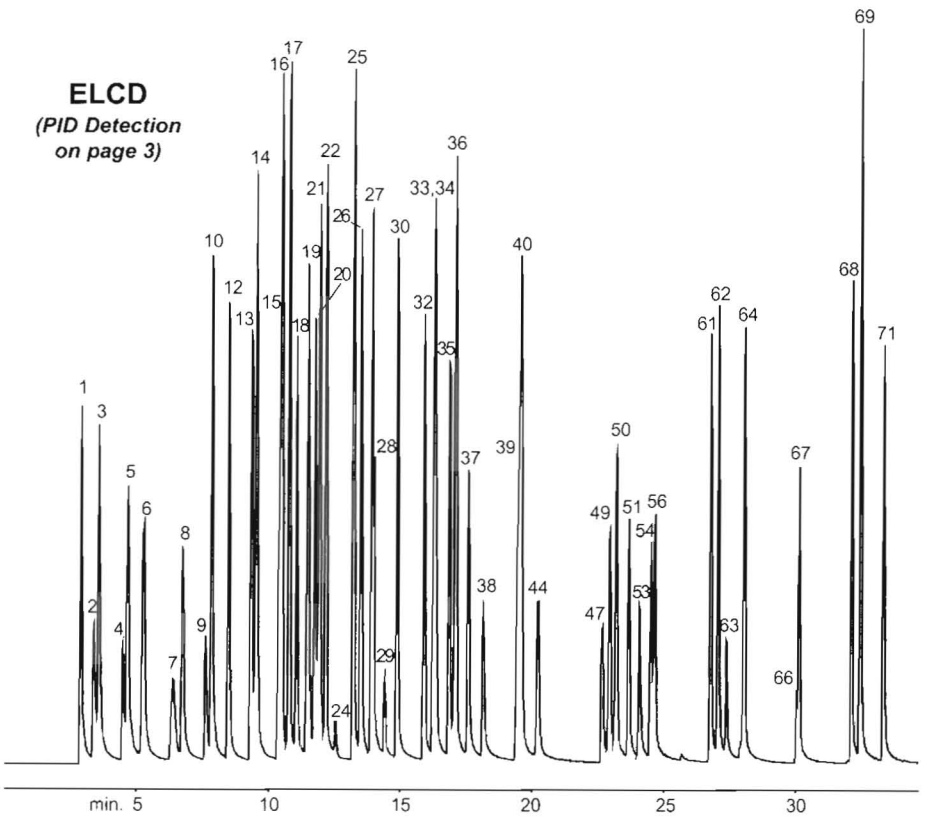
Acknowledgement: *Finnigan 9001 GC, µGold Tandem Photoionization Detector & Hall 2000 Detector provided courtesy of ThermoQuest/CE Instruments.*

Figure 1 **The New 0.45mm ID Rtx®-502.2 Column Provides a 15-Minute Faster Analysis Time Than the 0.53mm ID Column.**



0.45mm ID Rtx®-502.2 Column

Figure 2 The New 0.45mm ID Rtx®-502.2 Column Provides a 15-Minute Faster Confirmational Analysis Than the 0.53mm ID Column.



MTBE, Freon®113, and the trihalomethanes (THMs) from Method 502.2 analytes. These compounds are commonly found in "real world" samples. MTBE in groundwater is considered widespread and is currently regulated in 15 states across the country, with others expected to follow. Several states have added MTBE to their Total Petroleum Hydrocarbon (TPH) methods.

Freon®113 is used in industrial air conditioners and for cleaning metal surfaces. In the environment, it is a contributor of chlorine atoms to the atmosphere, which ultimately damages the earth's ozone layer. Freon®113 is still produced in 25 states nationwide. Both of these early eluting compounds have characteristically broad peaks when analyzed by purge and trap.

The THMs are disinfectant byproducts and are found in drinking water, which accounts for half of the workload in the gas chromatography (GC) lab. Column selection becomes very important when analyzing these added analytes along with the regular EPA volatile compounds. Figures 1 and 2 were achieved using a photoionization detector (PID) and the electrolytic conductivity detector (ELCD) in series. Additional compounds from Method 8021B, such as allyl chloride, chloroprene, 2-chloroethylvinylether, and benzyl chloride also are well resolved. Because these were analyzed under ambient purge conditions, the alcohols were not added as part of our target list. Suggested surrogates for the Rtx®-502.2 and Rtx®-1 column pair include fluorobenzene, 1-chloro-2-fluorobenzene, and 1,4-dichlorobutane. These performance-monitoring compounds are almost baseline resolved and will produce excellent recoveries. The outstanding thermal stability of the Rtx®-502.2 column minimizes bleed, which improves PID and ELCD performance and reduces instrument downtime. These bonded phase columns can be solvent rinsed if contaminated and have long lifetimes.

For more information on the new 0.45mm ID Rtx®-502.2, including chromatograms of confirmational analysis on the Rtx®-1, request lit. cat.# 59808. Call Technical Service at 800-356-1688, ext. 4, or contact your local Restek representative.

Peak List for Figures 1 & 2

1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. Freon® 113
8. 1,1-dichloroethene
9. allyl chloride
10. methylene chloride
11. methyl-*tert*-butyl-ether
12. *trans*-1,2-dichloroethene
13. 1,1-dichloroethane
14. chloroprene (40ppb)
15. 2,2-dichloropropane
16. *cis*-1,2-dichloroethene
17. chloroform
18. bromochloromethane
19. 1,1,1-trichloroethane
20. 1,1-dichloropropene
21. carbon tetrachloride
22. 1,2-dichloroethane
23. benzene
24. fluorobenzene (surrogate)
25. trichloroethene
26. 1,2-dichloropropane
27. bromodichloromethane
28. dibromomethane
29. 2-chloroethylvinylether
30. *cis*-1,3-dichloropropene
31. toluene
32. *trans*-1,3-dichloropropene
33. 1,1,2-trichloroethane
34. 2-bromo-1-chloropropane (surrogate)
35. 1,3-dichloropropane
36. tetrachloroethene
37. dibromochloromethane
38. 1,2-dibromoethane
39. chlorobenzene
40. 1,1,1,2-tetrachloroethane
41. ethyl benzene
42. *m*-xylene
43. *p*-xylene
44. 1-chloro-2-fluorobenzene (surrogate)
45. *o*-xylene
46. styrene
47. bromoform
48. isopropyl benzene
49. 1,4-dichlorobutane (surrogate)
50. 1,1,2,2-tetrachloroethane
51. 1,2,3-trichloropropane
52. *n*-proplyl benzene
53. bromobenzene
54. 2-chlorotoluene
55. 1,3,5-trimethylbenzene
56. 4-chlorotoluene
57. *tert*-butylbenzene
58. 1,2,4-trimethylbenzene
59. *sec*-butylbenzene
60. *p*-isopropyl toluene
61. 1,3-dichlorobenzene
62. 1,4-dichlorobenzene
63. benzyl chloride
64. *n*-butylbenzene
65. 1,2-dichlorobenzene
66. 1,2-bromo-3-chloropropane
67. 4-bromo-1-chlorobenzene (surrogate)
68. 1,2,4-trichlorobenzene
69. hexachlorobutadiene
70. naphthalene
71. 1,2,3-trichlorobenzene

Faster GC Volatile Analysis

Product Listing

Rtx®-502.2 Fused Silica Columns

ID	df (µm)	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	1.40	10915	10916	-----	-----
0.32mm	1.80	10919	10920	-----	10921
0.45mm—**NEW!**	2.55	-----	-----	10986	-----
0.53mm	3.00	10908	10909	-----	10910

ID	df (µm)	20-Meter	40-Meter
0.18mm	1.00	40914	40915

Most column configurations are available as metal MXT® columns. See pg. 46 of the *1999 Annual Product Guide*.

502.2 CAL2000 MegaMix™ Mixture

benzene
bromobenzene
bromochloromethane
bromodichloromethane
bromoform
n-butylbenzene
sec-butylbenzene
tert-butylbenzene
carbon tetrachloride
chlorobenzene
chloroform
2-chlorotoluene
4-chlorotoluene
dibromochloromethane
1,2-dibromo-3-chloropropane
1,2-dibromoethane
dibromomethane
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethene
cis-1,2-dichloroethene
trans-1,2-dichloroethene
1,2-dichloropropane
1,3-dichloropropane
2,2-dichloropropane
1,1-dichloropropene
cis-1,3-dichloropropene
trans-1,3-dichloropropene
ethylbenzene
hexachlorobutadiene
isopropylbenzene
p-isopropyltoluene
methylene chloride
naphthalene
n-propylbenzene
styrene
1,1,1,2-tetrachloroethane
1,1,2,2-tetrachloroethane
tetrachloroethene
toluene
1,2,3-trichlorobenzene
1,2,4-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethene
1,2,3-trichloropropane
1,2,4-trimethylbenzene
1,3,5-trimethylbenzene
m-xylene
o-xylene
p-xylene

In P&T methanol, 1mL/ampul

200µg/mL Ea.	Each	5-pk.	10-pk.
	30432	30432-510	-----
w/ data pack	30432-500	30432-520	30532

2000µg/mL Ea.	Each	5-pk.	10-pk.
	30431	30431-510	-----
w/ data pack	30431-500	30431-520	30531

Custom single or multiple component mixes are available. Please call for a quote.

502.2 Calibration Mix #1A

bromomethane
chloroethane
chloromethane
dichlorodifluoromethane
trichlorofluoromethane
vinyl chloride

In P&T methanol, 1mL/ampul

200µg/mL Ea.	Each	5-pk.	10-pk.
	30439	30439-510	-----
w/ data pack	30439-500	30439-520	30539

2000µg/mL Ea.	Each	5-pk.	10-pk.
	30042	30042-510	-----
w/ data pack	30042-500	30042-520	30142

502.2 Internal Standard Mix #1

1-chloro-2-fluorobenzene
2000µg/mL in P&T methanol, 1mL/ampul

	Each	5-pk.	10-pk.
	30040	30040-510	-----
w/ data pack	30040-500	30040-520	30140

502.2 Internal Standard Mix #2

2-bromo-1-chloropropane
fluorobenzene
2000µg/mL in P&T methanol, 1mL/ampul

	Each	5-pk.	10-pk.
	30041	30041-510	-----
w/ data pack	30041-500	30041-520	30141

VOA Individual Standards for EPA Methods, (2000µg/mL ea. in P & T Methanol)

1mL/ampul	Ind.	Ind. w/Data Pack	5-pk.	5-pk. w/data pack	w/data pack
4-bromochlorobenzene	30230	30230-500	30230-510	30230-520	30330
2-chloroethyl vinyl ether	30265	30265-500	30265-510	30265-520	30365
1,4-dichlorobutane	30227	30227-500	30227-510	30227-520	30327
MTBE	30402	30402-500	30402-510	30402-520	30502

GC Analysis of Organic Volatile Impurities According to USP <467>

by Christopher Cox, Senior R&D Chemist

The analysis of residual solvents using US Pharmacopoeia (USP) Method <467> presents many technical challenges to the pharmaceutical analyst with limited gas chromatography (GC) experience. Some of these challenges include poor injection reproducibility at high temperatures, poor reproducibility of peak area response, and difficult standard preparations. Issues related to USP <467> have been addressed in the Pharmacopoeial Forum, including sample introduction, standards preparation, detection limits, sample amounts, and column selection. We would like to further clarify these issues in the following article.

Methods I and V of USP <467> are the most commonly used methods for OVI analysis. One of the drawbacks associated with these methods is the use of aqueous injections for sample introduction. High injection port temperatures can produce large expansion volumes for injections of water as low as 1µL. When the expansion volume of the sample exceeds the buffer volume of the injection port liner, backflash can occur and some sample can be lost through the septum purge line. Because of backflash, injection reproducibility is poor at high temperatures. Originally, injection port temperatures were specified to be 180°C for Method I, and 140°C for Method V. Data supplied by Bergren and Foust demonstrated that a decrease in injection port temperature from 180°C to 70°C yielded lower relative standard deviations for peak area response on replicate injections.[1] Revisions have been made to Method I to lower the injection port temperature to 70°C, but the injection port temperature for Method V has not been revised as of the publication of this article.

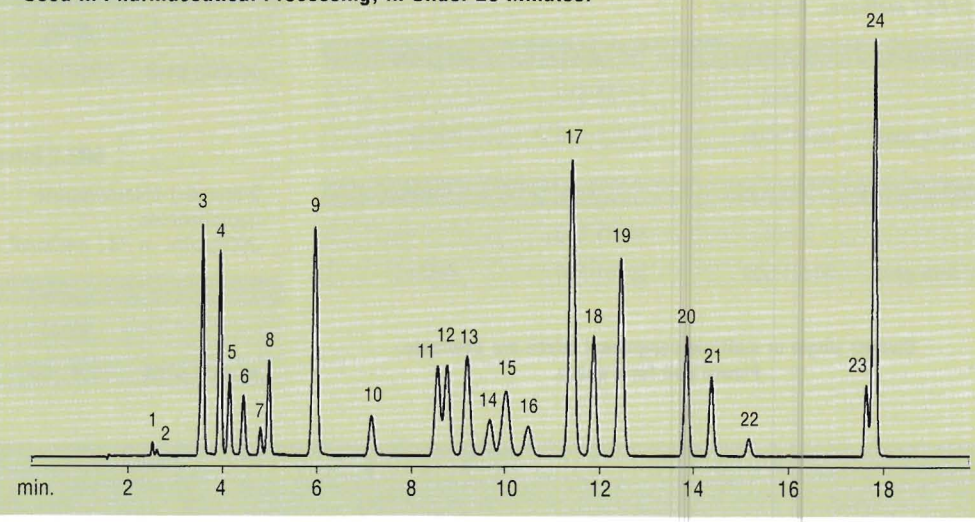
For more information and additional chromatograms, call 800-356-1688 or 814-353-1300, ext. 4 for *"GC Analysis of Organic Volatile Impurities According to USP <467>"* (Lit. Cat.# 59577).

Poor reproducibility of peak area response is also related to the amount of analyte reaching the column. The response of chloroform on an FID is very poor. At the concentrations originally specified for the standard solutions, the chloroform response cannot be reproducibly detected above the baseline noise. Krasowski et al.[2] proposed two solutions that previously have been mentioned in the *In Process Revision*[3] comments in the Pharmacopoeial Forum. The answer was to double the concentration of OVIs in the standard solution, and double the amount of bulk pharmaceutical used to produce the test solution. This modification increased the on-column concentration and produced a more consistent peak area response for each analyte. USP also increased the allowable relative standard deviation to 15% after finding that 10% was too demanding.

Figure 1 **The Rtx®-G43 Column Provides Good Resolution of Solvents Commonly Used in Pharmaceutical Processing, in Under 20 Minutes.**

Run Conditions:
Rtx®-G43 (30m, 0.53mm ID, 3.0µm) with 5m phenylmethyl Integra-Guard™ (cat.# 16085-126). Headspace injection of 24 common residual solvents for pharmaceutical processing. Prepared to equal approximately 500ppm in the bulk pharmaceutical. Samples shaken and heated at 90°C for 15 minutes, 1mL headspace injection. **Oven temp.:** 35°C (hold 10 min.) to 100°C @ 5°C/min., to 240°C @ 25°C/min. (hold 5 min.); **Inj./det. temp.:** 220°C/240°C; **FID sensitivity:** 1.25 x 10^{-11} AFS; **Carrier gas:** helium, 35cm/sec. set @ 35°C; **Split ratio:** 2:1.

Acknowledgement: *Thermoquest Trace GC and HS2000 Autosampler provided courtesy of ThermoQuest/CE Instruments.*



Peak List for Figure 1

1. ethylene oxide
2. methanol
3. diethyl ether
4. 1,1-dichloroethene
5. acetone
6. isopropanol
7. acetonitrile
8. methylene chloride
9. *n*-hexane
10. *n*-propanol
11. methyl ethyl ketone
12. ethyl acetate
13. tetrahydrofuran
14. chloroform
15. 1,1,1-trichloroethane
16. carbon tetrachloride
17. benzene
18. 1,2-dichloroethane
19. heptane
20. trichloroethylene
21. n-butanol
22. 1,4-dioxane
23. pyridine
24. toluene

In the Fifth Supplement, USP-NF[4], the requirement to analyze for trace levels of ethylene oxide was removed from Method <467>, and a test for ethylene oxide now only is performed when specified in the individual monograph.

In the May /June 1993 edition of Pharmacopoeial Forum[5], additional method modifications were made. The limit test concentration for methylene chloride was increased from 100ppm to 500ppm. The solvent used to prepare stock calibration standards also was changed from dimethyl sulfoxide to methanol.

Furthermore, USP has simplified the standard preparation procedure. The solubility for OVIs in water is very poor and direct dissolution of these compounds in water is difficult. Standard stability and lifetime can be improved by using stock solutions of the OVIs prepared in dimethyl sulfoxide, and then making dilutions of the stock standard into water to produce working standards. Comments in the September/October 1992 Pharmacopoeial Forum[6] propose the use of dimethyl sulfoxide as the solvent for stock standard, but this has not been approved as of the date of this publication.

Choosing a capillary column to perform OVI analysis also has been a subject for debate. Method I uses a capillary column interfaced with an FID, and sample introduction via a direct injection of aqueous sample. The column specified is a 30m x 0.53mm ID x 3µm, 5% phenyl/95% methyl polysiloxane (G27) column (Restek's Rtx®-G27 column). When Method I originally was published, a 1.5 resolution factor was included in the system suitability parameters. Many analysts have difficulty achieving baseline resolution between trichloroethylene and 1,4-dioxane. The 1.5 resolution factor was reduced to 1.0 in the Fifth Supplement of Method I, in order to make the system suitability requirements easier to achieve.

Method V also was introduced in the Fifth Supplement and incorporated the use of a 30m x 0.53mm ID x 3µm, 6% cyanopropylphenyl/94% dimethylpolysiloxane (G43) column (Restek's Rtx®-G43 column) as an alternative to the 5% phenyl/95% methyl polysiloxane column. Figure 1 shows the use of an Rtx®-G43 column for the analysis of OVIs. Baseline resolution of all the compounds is obtained and the resolution criteria of 3.0 is easily met on the Rtx®-G43 column.

In the Sixth Supplement, USP-NF[7], USP <467> changes were made to overcome the difficulties resulting from unregulated solvents coeluting with regulated solvents, thereby over-representing their concentration using GC/FID methods. GC/MS, or a second validated column with a different stationary phase, may be used to confirm the presence of the coeluting unregulated solvent and report the correct concentration of regulated solvent.

In addition to giving superior resolution for the OVIs listed in USP <467>, the Rtx®-G43 column also shows improved performance for analyzing other commonly used solvents, including many listed by the International Conference on Harmonization of Technical Requirements for Registration of Pharmaceutical for Human Use, Impurities: Guidelines for Residual Solvents.[8]

In a review of the current status of USP <467>[9], the USP is advocating the conversion of monograph requirements from Method I to Method V, to allow the use of the cyanopropyl stationary phase for better resolution. They are also anticipating that more laboratories will begin to use the static headspace technique as a means of replacing the direct injection of aqueous samples and its associated problems. The European Pharmacopoeia lists only static headspace methods for the testing of OVIs.[10] These changes, along with the current revisions to USP <467>, should result in an easier to use and more reproducible method for the future.

References

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2. J.A. Krasowski, H. Dinh, T.J. O'Hanlon, R.F. Lindauer, "Comments on Organic Volatile Impurities, Method I, <467>," *Pharmacopoeial Forum*, May/June 1991, Vol. 17, No. 3, pp. 1969-1972.
3. *Pharmacopoeial Forum*, March/April 1991, Vol. 17, No. 2, p. 1653.
4. Fifth Supplement, USP-NF, Organic Volatile Impurities <467>, Nov. 15, 1991, pp. 2706-2708.
5. "Organic Volatile Impurities <467>," *Pharmacopoeial Forum*, May-June 1993, Vol. 19, No. 3, pp. 5335-5337.
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7. Sixth Supplement, USP-NF, "Organic Volatile Impurities <467>," May 15, 1997, pp. 3766-3768.
8. "ICH Harmonized Tripartite Guideline, Impurities: Guideline for Residual Solvents," *The Fourth International Conference on Harmonization*, July 17, 1997.
9. V. Gray, "Organic Volatile Impurities Testing Initiative: An Update," *Pharmacopoeial Forum*, March/April 1992, Vol. 18, No. 2, p. 3205.
10. *European Pharmacopoeia*, Supplement 1999, pp. 14-15, 208.

References not available from Restek.

Product Listing

Rtx®-G27 Integra-Guard™ Column (with built-in 5m phenylmethyl guard column)*
(5% phenyl/95% methyl polysiloxane)

Dimensions	Temp. Limits	cat.#
30m x 0.53mm ID x 5µm	-60 to 270/290°C	10279-126

Rtx®-G43 Integra-Guard™ Column (with built-in 5m phenylmethyl guard column)*
(6% cyanopropylphenyl/94% dimethyl polysiloxane)

Dimensions	Temp. Limits	cat.#
30m x 0.53mm ID x 3µm	-20 to 240°C	16085-126

*Restek is the only capillary column manufacturer that offers Integra-Guard™ columns. These innovative columns come with a built-in guard column that eliminates the need for a connector and assures a leak-free connection between the guard column and analytical column. In addition to capillary columns, Restek offers a variety of calibration standards, headspace vials, and inlet liners for this application. Request Applications Note "GC Analysis of Organic Volatile Impurities According to USP <467>" (lit. cat.# 59577) for a complete product listing.

USP 467 Calibration Mixture #3

benzene	100µg/mL
chloroform	50
1,4-dioxane	100
methylene chloride	500
trichloroethene	100

Prepared in DMSO, 1mL/ampul

Each: cat.# 36004
10-Pack: cat.# 36104

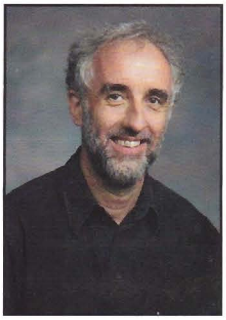
Restek offers a complete line of
reference materials for USP <467> analysis,
and other OVI Methods. Custom standards are also available.

Call 800-356-1688, ext. 4, for more information or contact your local Restek representative.

KONI'S KORNER

The Value of Education

by Dr. Konrad Grob, Contributing Author



In my last Korner, I concluded that only an education and qualification system could prevent chromatography from further decline. Instead of devoting time and effort to force better chromatography through quality management schemes, validation, accreditation, and bureaucratic piles of paper, I suggested that institutions invest in improving the quality of their employees. In this way, both the employees and the employers share the responsibility for improvement. Employers should realize that knowing how to manage a crimper for closing autosampler vials (despite what some instrument vendors may claim) is not "all" it takes to make a gas chromatography (GC) laboratory successful. At the same time, if analysts want to be considered valuable assets, then they should be ready to take an examination that affirms their education and training. How much education is necessary?

I received an overwhelming response to my article. Almost all of which confirmed my conclusion. For the sake of argument, though, I would have liked to hand over the microphone to somebody who disagrees. However, without refuting my point, I will take a moment to gain a perspective on the questions of 1) How simple is GC really? and 2) How much education is necessary? When observed from a safe distance, the work of a GC analyst appears simple. A gas chromatographer performing routine analyses should be able to help troubleshoot when results are not appropriate. The analyst should, in addition, be able to "rapidly" analyze x in sample y, and know how to select the right column, the injection technique, and all the many parameters finally determining whether the analysis will be successful. If capable of developing methods, the analyst needs to overview the possibilities and the problems to be expected; the clever choosing of strategies, tools, and conditions that may save trouble over years and reduce the time needed per sample by a factor of more than two.

No doubt analysts who are able to answer common GC questions achieve more reliable results because they can find the pitfalls. While other people waste several days because an analysis does not turn out adequately, these analysts find tests that can rapidly localize the problem. They know beforehand that aqueous samples are more difficult to analyze and should be injected in small volumes.

In industrial countries, every working day costs around $1,000. Eliminating three days of expense per month by improved troubleshooting saves $3,000 for that month. You easily can save several days per month by using better methods or improving performance of given methods. A knowledgeable gas chromatographer can prevent visits by the service engineer and avoid other delays disturbing production or delivery. If half of the profit generated by more competent work goes to shareholders, the analyst's salary should increase by $3,000 at least.

I would like to substantiate my call for education by asking some questions about a specific technique in capillary GC: split injection. Test your knowledge; see if you think more education is required for efficient and appropriate GC analysis. A lab supervisor who deals with GC should be able to answer ALL of the questions on page 9. Please review the answers; if you have difficulty with any of them, perhaps a convenient refresher course would be helpful.

Coming to a Location Near You...

Comprehensive GC Seminar

by Andy Schuyler, Seminar Coordinator

This is a great opportunity to learn tips for saving time and money, such as when columns can be operated above their maximum temperatures and how to convert a packed column GC to a capillary GC in under 5 minutes! The seminar will also cover the sample/column/GC system—from sample introduction to detection—and will extensively survey proper injection techniques, column selection, column installation, system maintenance, and detector operation.

If you have trouble with the questions on page 9, help is available! As a direct response to your interest in improved GC education, "Restek On-the-Road" presents its new *"Comprehensive GC for the Practicing Chromatographer Seminar Series."* The new seminars are a full-day course, presented in an engaging multimedia format that teaches key chromatographic concepts, tricks of the trade, and little-known secrets that are of benefit to the novice or the seasoned veteran. There is no sales pitch presented during the seminar, just the facts on how to make your chromatography results better.

Restek guarantees that after you apply what you have learned in our seminar, the savings you create in the first month alone will exceed the registration fee!

Contact the Restek representative nearest you for information on educational seminars and training in your area.

Do you need a brush-up on your chromatography basics?

Take this simple test to see if you would benefit from Restek training.

Concept Questions

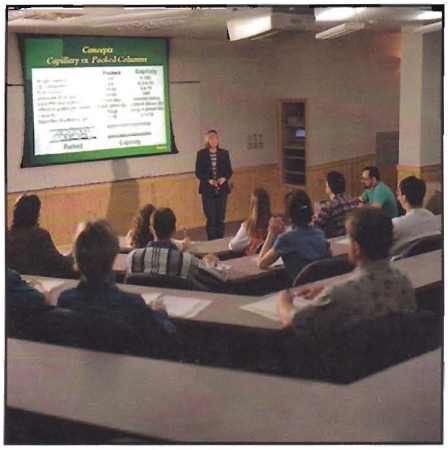
1. Describe split injection and what determines the split ratio?
2. Name the principal parts of a split injector.
3. Describe two concepts of pneumatic systems controlling the gas supply and split flow rate.
4. Does an increase of the split ratio increase or decrease peak areas?
5. What are the two steps required to adjust the split ratio?
6. Does the split ratio depend on a) the injector temperature or b) the column temperature?
7. Do autosamplers produce the same results as manual injections (within the normal standard deviation)?
8. What are the problems resulting from partial sample evaporation in the syringe needle?
9. Why is an empty, straight liner usually used for manual injections?
10. If performed manually: does a 1µL injection produce twice as large peaks as a 0.5µL injection, to within 10%?

Questions on Selecting Conditions

11. When does the liner need to be packed?
12. What are the factors determining the best sample volume for split injection?
13. What is the best injector temperature?
14. What is the effect of solvent recondensation in the column inlet?
15. What should you be careful about when using split injection with the external standard methods?
16. What length of inserted syringe needle is suitable for split injection?

Troubleshooting Questions

17. What can cause absolute peak areas to have relative standard deviations exceeding 10%?
18. What is "non-linear splitting" and how can this disturb quantitative results?
19. How do you test for a deviation of the effective split ratio?
20. How do you test for discrimination by losses inside the syringe needle?
21. Does a minor leak in the septum have an effect on quantitative analysis?
22. What is the effect of a leaking fitting between the liner and the injector body?
23. If the plunger of the syringe moves with friction and the manual injection is slower, will it have an effect on quantitative results?



We'll Bring the Seminar to You!

"Restek On-the-Road" presents its new *"Comprehensive GC for the Practicing Chromatographer Seminar Series"* at 41 locations nationwide in 1999. However if these aren't convenient for you, we'll bring the seminar to you! Seminars at your facility are presented by experienced chromatographers and provide customized instruction But don't take our word for it...

> "As the Training Coordinator for the University of Iowa Hygenic Laboratory, I worked with Restek to coordinate a Gas Chromatography Workshop at our facility. The workshop was very informative and was well received by all. I would recommend this training to anyone using a GC—regardless of education or experience level."
>
> - Beth Hochstedler
> University of Iowa Training Coordinator

Check Your Answers!

Visit us online at
www.restekcorp.com/konitest.htm
- or -
*Call us at **800-356-1688, ext. 4.***
We'll fax or email you the answers.

REFERENCE MATERIALS

CLP Volatiles OLM 04.1

by Eric Steindl, Analytical Reference Materials Product Line Manager

Standards for the EPA Superfund Contract Lab Program (CLP):

- ✔ Meets latest Statement of Work.
- ✔ Fewest number of calibration solutions possible.
- ✔ Immediately available.
- ✔ Maximum shelf life.

CLP 04.1 VOA CAL2000 MegaMix™

2000µg/mL each in P & T methanol
benzene
bromodichloromethane
bromoform
carbon disulfide
carbon tetrachloride
chlorobenzene
chloroform
1,2-dibromo-3-chloropropane
cyclohexane
dibromochloromethane
1,2-dibromoethane
1,2-dichlorobenzene
1,3-dichlorobenzene
1,4-dichlorobenzene
1,1-dichloroethane
1,2-dichloroethane
1,1-dichloroethene
cis-1,2-dichloroethene
trans-1,2-dichloroethene
1,2-dichloropropane
cis-1,3-dichloropropene
trans-1,3-dichloropropene
ethylbenzene
isopropylbenzene
methyl acetate
methylcyclohexane
methylene chloride
styrene
methyl-*tert*-butyl-ether (MTBE)
1,1,2,2-tetrachloroethane
tetrachloroethene
toluene
1,2,4-trichlorobenzene
1,1,1-trichloroethane
1,1,2-trichloroethane
trichloroethylene
1,1,2-trichloro-1,2,2-trifluoroethane
m-xylene
o-xylene
p-xylene

Each	5-pk.	10-pk.
30456	30456-510	-----
	w/ data pack	
30456-500	30456-520	30556

VOA Calibration Mix #1

5000µg/mL each in P & T methanol
acetone
2-butanone
2-hexanone
4-methyl-2-pentanone

Each	5-pk.	10-pk.
30006	30006-510	-----
	w/ data pack	
30006-500	30006-520	30106

502.2 Calibration Mix #1

2000µg/mL each in P & T methanol
bromomethane
chloroethane
chloromethane
dichlorodifluoromethane
trichlorofluoromethane
vinyl chloride

Each	5-pk.	10-pk.
30042	30042-510	-----
	w/ data pack	
30042-500	30042-520	30142

CLP 04.1 VOA Internal Standard/SMC Spike Mix

2500µg/mL each in P & T methanol
bromochloromethane
1,4-difluorobenzene
chlorobenzene-d5
4-bromofluorobenzene
1,2-dichloroethane-d4
toluene-d8

Each	5-pk.	10-pk.
30457	30457-510	-----
	w/ data pack	
30457-500	30457-520	30557

VOA Surrogate Spike Mix

(System Monitoring Compounds Spike Mix)
2500µg/mL each in P & T methanol
4-bromofluorobenzene
1,2-dichloroethane-d4
toluene-d8

Each	5-pk.	10-pk.
30004	30004-510	-----
	w/ data pack	
30004-500	30004-520	30104

VOA Matrix Spike Mix

2500µg/mL each in P & T methanol
benzene
chlorobenzene
1,1-dichloroethene
toluene
trichloroethene

Each	5-pk.	10-pk.
30005	30005-510	-----
	w/ data pack	
30005-500	30005-520	30105

VOA Screening Mix # 1

1000ug/mL each in P & T methanol
benzene
ethylbenzene
toluene
o-xylene
p-xylene

Each	5-pk.	10-pk.
30001	30001-510	-----
	w/ data pack	
30001-500	30001-520	30101

VOA Screening Mix # 2

1000µg/mL each in P & T methanol
n-dodecane
n-nonane

Each	5-pk.	10-pk.
30002	30002-510	-----
	w/ data pack	
30002-500	30002-520	30102

VOA Tuning Compound

5000µg/mL in P & T methanol
4-bromofluorobenzene

Each	5-pk.	10-pk.
30003	30003-510	-----
	w/ data pack	
30003-500	30003-520	30103

VOA Internal Standard Mix

2500µg/mL each in P & T methanol
bromochloromethane
chlorobenzene-d5
1,4-difluorobenzene

Each	5-pk.	10-pk.
30011	30011-510	-----
	w/ data pack	
30011-500	30011-520	30111

CLP 04.1 VOA Kit #1

Contains 1mL each of:	*Restek Cat. #*
VOA Screening Mix #1	30001
VOA Screening Mix #2	30002
VOA Tuning Compound	30003
VOA Surrogate Spike (System Monitoring Compounds [SMC])	30004
VOA Matrix Spike Mix	30005
VOA Calibration Mix #1 (ketones)	30006
VOA Internal Standard Mix	30011
502.2 Calibration Mix #1 (gases)	30042
CLP 04.1 VOA CAL2000 MegaMix™	30456

Each	Ea. w/Data Pack
30458	30458-500

CLP 04.1 VOA Kit #2

Contains 1mL each of:	*Restek Cat. #*
VOA Screening Mix #1	30001
VOA Screening Mix #2	30002
VOA Tuning Compound	30003
VOA Matrix Spike Mix	30005
VOA Calibration Mix #1 (ketones)	30006
502.2 Calibration Mix #1 (gases)	30042
CLP 04.1 VOA CAL2000 MegaMix™	30456
VOA Internal Standard/SMC Mix	30457

Each	Ea. w/Data Pack
30459	30459-500

CLP 04.1 VOA Kit #3

Contains 1mL each of:	*Restek Cat. #*
VOA Calibration Mix #1 (ketones)	30006
502.2 Calibration Mix #1 (gases)	30042
CLP 04.1 VOA CAL2000 MegaMix™	30456

Each	Ea. w/Data Pack
30460	30460-500

SILCOSTEEL® TREATMENT

Silcosteel®-Treated Tubing & Fittings

by Gary Barone, Metals Passivation Group Product Line Manager

The inertness and flexibility of Silcosteel®-treated tubing makes it ideal for sample transfer lines. In fact, Restek first developed this inert, deactivated stainless steel tubing specifically for transfer lines used in purge-and-trap systems. Our application chemists frequently found that standard fused silica lines became brittle and chemically active after repeated cycling. This often led to extended down-time of the instruments. Because we are in the business of developing new phases and applications, we needed better durability and performance from our transfer lines; the kind that Silcosteel®-coated tubing provides.

Today Restek has expanded the Silcosteel®-treated product line to include a wide variety of tubing and fittings for many process and lab instruments: purge-and-trap transfer lines, heated headspace transfer lines, sample loops, sample storage vessels, valving, and more. Restek has been working extensively with instrument manufacturers to incorporate Silcosteel®-treated components to improve system performance.

When peak performance and inertness are required for your analysis, use Silcosteel®-coated tubing and fittings.

Silcosteel®-Treated Tubing & Fittings are Ideal for Transfer Lines from Purge-and-Trap and Headspace

Silcosteel®-Treated Welded/Drawn 304* Grade Stainless Steel Tubing

ID, OD	5-24 ft.	25-199 ft.	200-399 ft.	>400 ft.
0.011" ID (0.28mm ID), 0.022" OD (0.56mm OD) **cat.# 20590**	ft.	ft.	ft.	ft.
0.021" ID (0.53mm ID), 0.029" OD (0.74mm OD) **cat.# 20591**	ft.	ft.	ft.	ft.
0.010" ID (0..25mm ID), 1/16" OD (1.59mm OD) **cat.# 20592**	ft.	ft.	ft.	ft.
0.020" ID (0.51mm ID), 1/16" OD (1.59mm OD) **cat.# 20593**	ft.	ft.	ft.	ft.
0.030" ID (0.76mm ID), 1/16" OD (1.59mm OD) **cat.# 20594**	ft.	ft.	ft.	ft.
0.040" ID (1.02mm ID), 1/16" OD (1.59mm OD) **cat.# 20595**	ft.	ft.	ft.	ft.
0.085" ID (2.16mm ID), 1/8" OD (3.18mm OD) **cat.# 20596**	ft.	ft.	ft.	ft.
0.210" ID (5.33mm ID), 1/4" OD (6.35mm OD) **cat.# 20597**	ft.	ft.	ft.	ft.

Silcosteel®-Treated Seamless 316* Grade Stainless Steel Tubing

0.055" ID (1.40mm ID), 1/8" OD (3.18mm OD) **cat.# 20598**	ft.	ft.	ft.	ft.
0.180" ID (4.57mm ID), 1/4" OD (6.35mm OD) **cat.# 20599**	ft.	ft.	ft.	ft.

*The surface properties of 304 and 316 stainless steel are virtually indistinguishable when Silcosteel®-treated.

Tees

sizes	cat.#	
1/16"	20513	ea.
1/8"	20514	ea.
1/4"	20515	ea.

Unions

sizes	cat.#	
1/16"	20510	ea.
1/8"	20511	ea.
1/4"	20512	ea.

Reducing Fittings

sizes	cat.#	
1/16" to 1/8"	20519	ea.
1/16" to 1/4"	20520	ea.
1/8" to 1/4"	20521	ea.

Elbows

sizes	cat.#	
1/16"	20516	ea.
1/8"	20517	ea.
1/4"	20518	ea.

Zero Dead Volume Unions

sizes	cat.#	
1/16"	20580	ea.
1/8"	20582	ea.

Zero Dead Volume Tees

sizes	cat.#	
1/16"	20581	ea.
1/8"	20583	ea.

Part I
In a Series

INLET LINERS

What Type of Inlet Liner is Best for My Analysis?

by Brad Rightnour, GC Accessories Product Line Manager

With so many different inlet liner designs and deactivation chemistries available, how do you determine which one is best suited for your analysis? Each liner geometry offers the analyst a unique sample flow, from the liner to the analytical column, through cups, cyclos, and packings designed in the inner bore of the liner. How does each design affect sample flow? Which deactivation chemistry is best for your particular analysis?

Let's look at the these questions to determine the answers to liner geometry selection and deactivation.

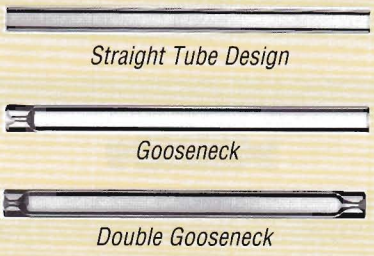
Splitless Inlet Liners (Figure 1)

Splitless liners are designed to hold the sample in the liner from between 0.5 and 2.0 minutes. A large surface area is not critical for splitless injections.

It is common to use packing materials whenever dirty samples are analyzed.

Figure 1

Splitless liners are designed to hold the sample in the liner for 0.5 to 2 minutes. A large surface area for sample vaporization is not a factor in splitless injections.



The *straight tube design* is the most common splitless sleeve design. This liner is ideal for low molecular weight samples that are not prone to thermal decomposition. If used for high molecular weight sample analysis, packing material is recommended to aid in sample vaporization. The drawback with using packing material is that the resulting increased residence time of the sample can cause adsorption of the high molecular weight compounds.

The *gooseneck liner* isolates the sample from the metal injection port parts situated at the base of the injector. This design funnels the sample onto the analytical column for increased splitless efficiency and decreases the breakdown of highly active compounds, such as endrin and DDT. A *double gooseneck design* helps to contain the sample cloud in the liner, for increased performance with larger sample introductions, but cannot be packed with wool.

Split Inlet Liners (Figure 2)

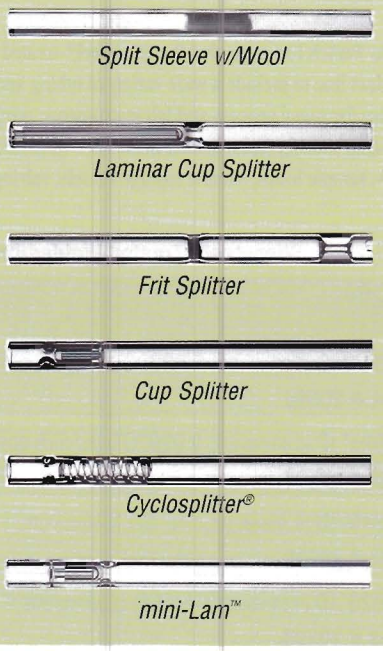
Split liners are designed to help vaporize the sample before it enters the column using mixing chambers and tortuous flow paths. Materials such as deactivated fused silica wool or beads, CarboFrit™ packing, and other packings are used to increase sample vaporization.

The most common liner for split analysis is the *4mm straight liner with deactivated wool*. This offers the analyst a wide variety of options. The wool has a high surface area for more sample evaporation to occur, and promotes a uniform vapor cloud to enter the split point. This liner is the most economical of the split liners; the drawback is that the wool increases breakdown of highly active compounds. Extensive upkeep is required to maintain analysis reproducibility when using this liner, as the wool needs to be changed frequently and its position and quantity inside the liner is critical.

Cup splitter liners offer a more homogenous vaporization through increased sample residence time in the liner. The sample passes through a series of tortuous flow paths, which aids in sample vaporization. First, the sample travels around an elongated cup and is trapped at the base of the liner, where vaporization occurs. Then it travels back up the liner and onto the column. These liners are best suited for high molecular weight compounds.
The *Cyclosplitter® liners* incorporate a cylindrical glass screw in the sample pathway. The screw helps to mix and vaporize the sample. The increased surface area in the cylindrical glass screw also helps to trap non-vaporized sample, therefore making it ideal for dirty samples.

Figure 2

Split liners are designed to use mixing chambers and tortuous flow paths to help vaporize the sample before it enters the column.



Does Deactivation Make a Difference?

Deactivation chemistry has come a long way since acid and dimethyldichlorosilane (DMDCS) deactivation. With more choices available, how do you choose one deactivation chemistry over another? Deactivation of the inlet liner is critical in the introduction of the sample to the column, because the liner is the first point of contact for the sample in the inlet system. If the liner is not properly deactivated, adsorption or breakdown of the sample can occur and result in poor quantitation or misidentification of compounds (Figure 3).
For the majority of analyses, liner deactivation is

necessary to ensure complete, sample transfer to the capillary column. Deactivation is especially critical for analysis of certain pesticides, herbicides, amines, acids, and drugs.

Not all deactivations are alike—different types of chemicals and processes are used to deactivate the surface of the glass. There are several types of liner deactivations available:

Pinpoint Deactivation

This is the most widely used deactivation technique for liners and typically uses DMDCS deactivation. It is good for most non-critical analyses, analysis of polyaromatic hydrocarbons (PAHs), highly concentrated samples, and non-active sample matrices. This deactivation has very low resistance to sample degradation before re-deactivation or liner replacement is needed.

Polymeric Deactivation

A polymeric deactivation provides total surface coverage. There are no exposed active sites as there are with pinpoint deactivation. Polymeric deactivation has a high resistance to sample degradation and shows increased response for low concentration samples and highly active samples, such as endrin, DDT, and drugs. Endrin breakdown of less than 2% is standard when using a polymeric deactivation. Longer liner lifetimes are provided because the deactivation is bonded to the surface of the glass and is more durable than pinpoint deactivation.

Base or Amine Deactivation

This is a special deactivation for the analysis of bases and amines. This deactivation provides superior response and sample repeatability for the analysis of trace amine compounds (Figure 4).

When choosing a liner for your analysis, match the liner geometry and deactivation to the analysis you will be performing. The liner geometry and deactivation are as important as the choice of column for the analysis of special compounds. Increased performance and more accurate analysis will be the results of a thoughtful decision.

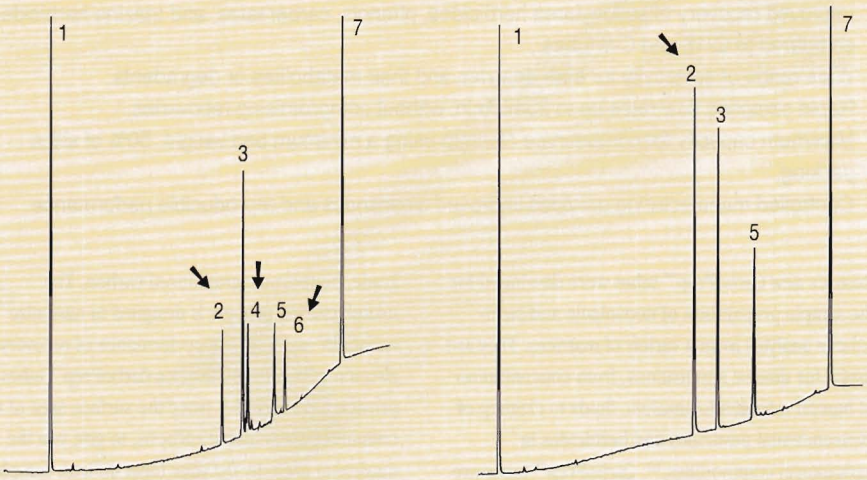
Request Restek's handy pocket reference guide ***Inlet Supplies*** (lit. cat. #59980), or visit our web site at www.restekcorp.com. The Inlet Supplies pocket guide is an at-your-fingertips reference packed with liner selection information, inlet supplies, and a complete product listing.

Figure 3

Deactivated Liners Significantly Decrease Endrin Breakdown

The untreated inlet liner exhibits 62% endrin breakdown.

The deactivated inlet liner exhibits 1% endrin breakdown.



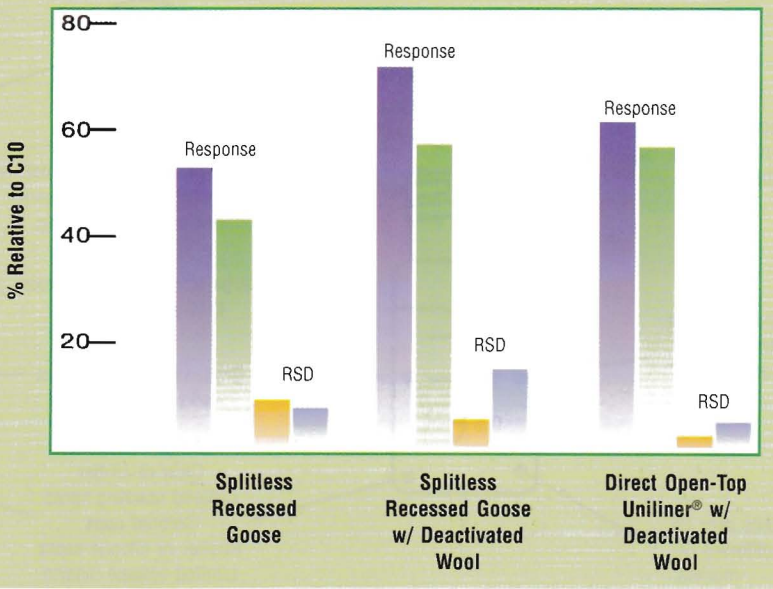
1. 2,4,5,6-tetrachloro-*m*-xylene
2. endrin
3. 4,4'-DDT
4. endrin aldehyde
5. methoxychlor
6. endrin ketone
7. decachlorobiphenyl

Figure 4

Response Comparison of Amine Deactivation vs. Polymeric Deactivation.

Amine Deactivation Polymeric Deactivation

Column: 30m, 0.53mm ID, 3.0µm, Rtx®-5 Amine; **Diethanolamine on-column concentration:** 15ng; **Injections/sleeve:** 5; **Inj./det. temp:** 250°C/285°C Each sleeve conditioned @ 285°C for 1 hour prior to injections. HP 5890II Plus with HP 7673 Autosampler.



New CarboPrep™ SPE Tubes

RESPREP™

We are pleased to announce the addition of CarboPrep™ SPE tubes to our growing line of Respprep™ products!

by Lydia Nolan, Sample Preparation Product Line Manager

- Improved recovery of sulfonylurea herbicides, phenols, carbamates, and triazine herbicides compared to C-18 and C-8 tubes.
- Wide range of selectivity for both analytes and their metabolites or degradents.
- Rapid sampling flow rates up to 20mL/min. without compromising recoveries.
- Maximum capacity for contaminate cleanup using a minimum bed weight, 50% of silica packing.
- Controlled manufacturing provides improved cleanliness and reproducible performance.

Restek's new CarboPrep™ tubes were developed for the sample preparation of nonvolatile and semi-volatile analytes from a variety of matrices. They are useful for diverse applications, from concentration of human estrogen in amniotic fluids to cleanup of environmental pesticides and herbicides in agricultural commodities.[1,2] CarboPrep™ tubes are commercially manufactured from chromatographic-grade, nonporous, graphitized carbon. The manufacturing process is designed to minimize variability and improve performance of recovery and cleanup procedures. Restek's CarboPrep™ tubes provide a carbon with twice the surface area of most commercially available carbons, to give you the maximum capacity for your most difficult samples.

To experience the many benefits of CarboPrep™ tubes for yourself, call 800-356-1688, ext. 3, or contact your local Restek representative for a sample pack and *Get Prepped* today!

References
1. F. Andreolini, C. Borra, F. Caccamo, A. DiCorcia, and R. Samperi. *Analytical Chemistry*. 1987, 59, pp 1720-1725.
2. J. Fillion and L. Nolan, *Today's Chemist*. 1996, pp. 14-24.

cat. #	
CarboPrep™ 3mL, 250mg	
26088	(50-pk.)
CarboPrep™ 6mL, 500mg	
26087	(30-pk.)

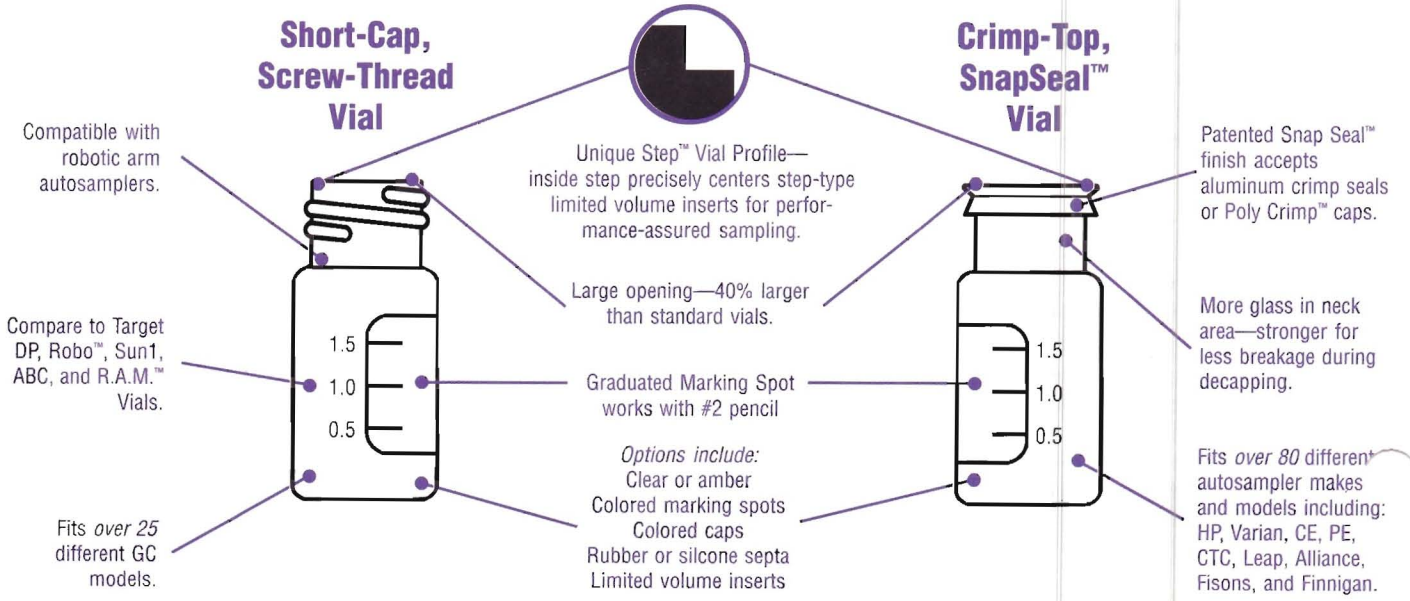
Custom sizes available, please call for details.

The Ultimate Autosampler Vials

Restek has assembled what may be the ultimate line of autosampler vial products. These vials offer more features—such as large openings, step insert profiles, and graduated marking spots—all at the same low price as other manufacturers' standard plain vials. For a complete listing of Restek's extensive line of vials for any instrument or application, see the *1999 Product Guide*.

For a free sample vial kit, call 800-356-1688, ext. 3, or contact your local Restek representative.

- ✔ ***Type I, 33 expansion borosilicate glass offers the least pH shift (lowest leaching characteristics) of any glass type.***
- ✔ ***Manufactured in compliance with ISO 9002 guidelines for assured, consistent quality.***
- ✔ ***Restek's exclusive Silcote™ CL7, silicon polymer-based silanization ensures sample integrity.***



Peak Performers

by Doug Elliott, GC Accessories Product Line Manager

Flexible Graphite Ferrules

New Technology Means Longer Ferrule Life

Our fused silica quality assurance (QA) analysts are reporting twice the number of re-use cycles with the new Restek Flexible Graphite Ferrules. In addition to having lower bleed and improved sealing performance, they can be re-used 4 or 5 times! Our analysts are very happy with this high level of performance. Let us know how well they work in your applications.

For a complete listing of ferrules, please see the Restek 1999 Product Guide.

Graphite Capillary Ferrules

(for 1/16" compression-type fittings)

Ferrule ID	Fits Column ID	10-pack
0.3mm	0.20mm	20233
0.4mm	0.25mm	20200
0.5mm	0.32mm	20201
0.8mm	0.53mm	20202
		50-pack
0.4mm	0.25mm	20227
0.5mm	0.32mm	20228
0.8mm	0.53mm	20224

Compact Graphite Ferrules for HP GCs

(for capillary injection ports)

Ferrule ID	Fits Column ID	10-pk.
0.4/0.5mm	0.25–0.32mm	20250
0.8mm	0.53mm	20252
		50-pk.
0.4/0.5mm	0.25–0.32mm	20251
0.8mm	0.53mm	20253

Cross Disk Inlet Seal

Replacement 0.8mm ID Inlet Seal, for HP 6890 GCs

Restek now has the 0.8mm ID cross-type inlet seals for HP 6890 injectors. Deactivations include our gold-plating process, which has been optimized to be stable and uniform. The legendary Silcosteel®-treated seals are as inert as gold, but more rugged. Try them for yourself!

Washers included!

0.8mm ID Cross Disk Inlet Seal for HP GCs

(Similar to HP part #5182-9652)

Inlet Seal Type	2-pk.	10-pk.
Gold-Plated	20477	20476
Silcosteel®-Treated	20475	20474

***For a complete listing of inlet seals, please see the* 1999 Product Guide.**

Air Diverter

for 5890/6890 GCs

- Diverts GC exhaust heat away from the lab bench.
- Reduces oven cycle time.
- Improves retention time precision.
- Easy to install—no tools required.

(Similar to HP part # 19247-60510)
Cat.# 22076, (ea.)

Restek Leak Detective™

Electronic Leak Detector

- Compact, lightweight, hand-held design.
- Contamination-free leak detection.
- Detects helium or hydrogen trace leaks at $\geq 3 \times 10^{-4}$ cc/sec or ≥200ppm.
- Pays for itself in the first leak found.
- Battery or AC line adaptor

110v: Cat. #21607, ***220v:*** Cat.# 21609, (ea.)

Clean & Green

Instrument-Grade Tubing

We know how important clean tubing for plumbing is to the successful delivery of pure gas to your instrument. We also know that we have a responsibility to protect the environment and our employees. For those reasons, Restek has developed a new cleaning process that uses biodegradable detergents, solubilizers, penetrants, organic acids, and non-toxic solvents for superior surface cleaning—leaving no film or residue.

Now even better!

When you want the cleanest tubing for plumbing your analytical instrument system, and you also are concerned about the quality of the environment and worker safety, choose Restek Instrument-Grade Tubing. See the *1999 Product Guide* for details.

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