

the **RESTEK** Advantage

Innovators of High Resolution Chromatography Products

Rtx[®]-VMS Columns

Fast Volatile Organic Compounds GC/MS Analysis

by Christopher English, Applications Chemist

- ✓ Improved selectivity, lower bleed, and faster analysis time than traditional "624/1301" phases for more efficient VOC analyses.
- ✓ Tuned phase resolves EPA Method 8260B compounds that share common quantitation ions, giving you more accurate identifications.
- ✓ Excellent gas resolution, even with a 50°C starting temperature, which results in faster oven cycle time and increased sample throughput.

Volatile organic compounds (VOCs), consisting of a wide range of solvents and chemicals, continue to be a common cause of environmental pollution. SW-846 contains the analytical and test methods that the Environmental Protection Agency (EPA) has

evaluated as acceptable under the Resource Conservation and Recovery Act (RCRA) of 1976. SW-846 outlines testing procedures for hazardous waste, sludge or other discarded material. US EPA Method 8260B is found in SW-846 and is used as a guideline to setting acceptable, although not required, standards

for determination of VOCs by gas chromatography/mass spectrometry (GC/MS).

A challenge to environmental laboratories is the accurate and rapid analysis of almost 100 VOCs in a wide range of matrices. With the aid of computer modeling, the stationary phase for the Rtx[®]-VMS column has been tuned specifically to resolve the compounds in EPA Method 8260B that share common quantitation ions. This has allowed for better selectivity, lower bleed and faster analysis time than the traditional "624/1301" phases. The design of this stationary phase takes into account unconventional contaminants that are not target compounds in the usual test methods, such as ethers and acetates. These compounds were added to the

design criteria for the Rtx[®]-VMS phase because many of them have been discovered in ground water and are added to calibration mixes by environmental laboratories.

Excellent resolution of early-eluting gases was achieved on the Rtx[®]-VMS column, even with a 50°C starting temperature using a 20m, 0.18mm ID, 1.00µm column. This allows a fast oven cycle time (Fig. 1, pg. 2). The 10-minute analysis time is less than the cycle time of the purge and trap, so the cycle time is not being held up by column requirements.

A 60m, 0.25mm, 1.40µm ID Rtx[®]-VMS column allows better column capacity and longer lifetime than the 0.18mm ID column. The 60 meter column also allows a 60°C starting temperature without a significant loss of gas resolution, and provides an overall analysis time of 18 minutes. This is the same as the cycle time for the purge and trap system under optimum conditions (Fig. 2, pg. 3).

Both analyses were optimized to resolve the most difficult pair of compounds—chlorobenzene-d5 and 1,1,1,2-tetrachloroethane, which share ion 117 (Fig. 3, pg. 3). The factor that limits analysis time for the Rtx[®]-VMS column is the use of chlorobenzene-d5 for the internal standard (IS). Faster analyses can be achieved by using an alternate IS that will still satisfy the requirements of SW-846.

For more information on the Rtx[®]-VMS column, request the "Rtx[®]-VMS Benefits Brochure" (lit. cat# 59209).

See pages 2–3 for figures and product listing.

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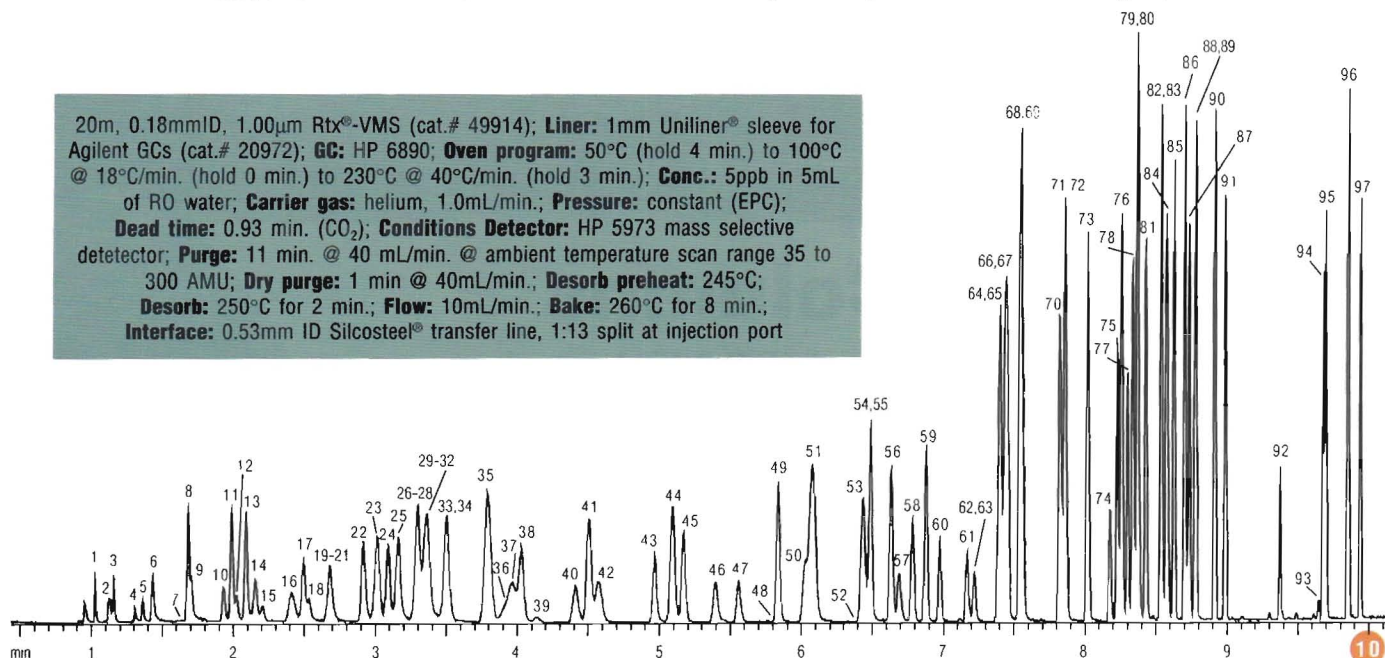
Winter 2001

INTERNATIONAL

Figure 1

A 20m, 0.18mm ID Rtx®-VMS column resolves 97 volatile organic compounds in 10 minutes using GC/MS.

20m, 0.18mmID, 1.00µm Rtx®-VMS (cat.# 49914); **Liner:** 1mm Uniliner® sleeve for Agilent GCs (cat.# 20972); **GC:** HP 6890; **Oven program:** 50°C (hold 4 min.) to 100°C @ 18°C/min. (hold 0 min.) to 230°C @ 40°C/min. (hold 3 min.); **Conc.:** 5ppb in 5mL of RO water; **Carrier gas:** helium, 1.0mL/min.; **Pressure:** constant (EPC); **Dead time:** 0.93 min. (CO₂); **Conditions Detector:** HP 5973 mass selective detector; **Purge:** 11 min. @ 40 mL/min. @ ambient temperature scan range 35 to 300 AMU; **Dry purge:** 1 min @ 40mL/min.; **Desorb preheat:** 245°C; **Desorb:** 250°C for 2 min.; **Flow:** 10mL/min.; **Bake:** 260°C for 8 min.; **Interface:** 0.53mm ID Silcosteel® transfer line, 1:13 split at injection port



- | | | |
|--------------------------------------|---------------------------------------|--|
| 1. dichlorodifluoromethane | 34. 1,1-dichloropropene | 67. 1,1,1,2-tetrachloroethane |
| 2. chloromethane | 35. benzene | 68. <i>m</i> -xylene |
| 3. vinyl chloride | 36. pentafluorobenzene (IS) | 69. <i>p</i> -xylene |
| 4. bromomethane | 37. <i>tert</i> -amyl-methyl ether | 70. <i>o</i> -xylene |
| 5. chloroethane | 38. 1,2-dichloroethane | 71. styrene |
| 6. trichlorofluoromethane | 39. isobutyl alcohol | 72. bromoform |
| 7. ethanol | 40. isopropyl acetate | 73. isopropylbenzene |
| 8. 1,1-dichloroethene | 41. trichloroethene | 74. 4-bromo-1-fluorobenzene (SMC) |
| 9. carbon disulfide | 42. 1,4-difluorobenzene (SMC) | 75. bromobenzene |
| 10. allyl chloride | 43. dibromomethane | 76. <i>n</i> -propylbenzene |
| 11. methylene chloride | 44. 1,2-dichloropropane | 77. 1,1,2,2-tetrachloroethane |
| 12. acetone | 45. bromodichloromethane | 78. 2-chlorotoluene |
| 13. <i>trans</i> -1,2-dichloroethene | 46. methyl methacrylate | 79. 1,3,5-trimethylbenzene |
| 14. methyl- <i>tert</i> -butyl-ether | 47. <i>n</i> -propyl acetate | 80. 1,2,3-trichloropropane |
| 15. <i>tert</i> -butyl alcohol | 48. 2-chloroethanol | 81. 4-chlorotoluene |
| 16. diisopropyl ether | 49. <i>cis</i> -1,3-dichloropropene | 82. <i>tert</i> -butylbenzene |
| 17. 1,1-dichloroethane | 50. toluene- <i>d</i> 8 (SMC) | 83. pentachloroethane |
| 18. acrylonitrile | 51. toluene | 84. 1,2,4-trimethylbenzene |
| 19. vinyl acetate | 52. pyridine | 85. <i>sec</i> -butylbenzene |
| 20. allyl alcohol | 53. tetrachloroethene | 86. <i>p</i> -isopropyltoluene |
| 21. ethyl- <i>tert</i> -butyl ether | 54. 4-methyl-2-pentanone | 87. 1,3-dichlorobenzene |
| 22. <i>cis</i> -1,2-dichloroethene | 55. <i>trans</i> -1,3-dichloropropene | 88. 1,4-dichlorobenzene- <i>d</i> 4 (IS) |
| 23. 2,2-dichloropropane | 56. 1,1,2-trichloroethane | 89. 1,4-dichlorobenzene |
| 24. bromochloromethane | 57. ethyl methacrylate | 90. <i>n</i> -butylbenzene |
| 25. chloroform | 58. dibromochloromethane | 91. 1,2-dichlorobenzene |
| 26. ethyl acetate | 59. 1,3-dichloropropane | 92. 1,2-dibromo-3-chloropropane |
| 27. carbon tetrachloride | 60. 1,2-dibromoethane | 93. nitrobenzene |
| 28. methyl acrylate | 61. <i>n</i> -butyl acetate | 94. hexachlorobutadiene |
| 29. propargyl alcohol | 62. 2-hexanone | 95. 1,2,3-trichlorobenzene |
| 30. dibromofluoromethane (SMC) | 63. 2-picoline | 96. naphthalene |
| 31. tetrahydrofuran | 64. chlorobenzene- <i>D</i> 5 (IS) | 97. 1,2,4-trichlorobenzene |
| 32. 1,1,1-trichloroethane | 65. chlorobenzene | |
| 33. 2-butanone | 66. ethylbenzene | |

Important Tech Tip!

Gas management is of critical importance when setting up a purge and trap system. Leaks in the chromatographic system can introduce oxygen, which can damage the stationary phase at higher temperatures and result in elevated bleed. Always make sure oxygen, hydrocarbon, and water traps are replaced on a regular basis, and carefully leak-check the entire system from the oxygen trap to the injection port.

These simple steps will reduce bleed for any column and result in enhanced sensitivity and performance. For more information, contact our technical service team at support@restekcorp.com or call 800-356-1688 or 814-353-1300, ext. 4.

Also, you can review our column installation instructions online at www.restekcorp.com/capillary/guide.htm

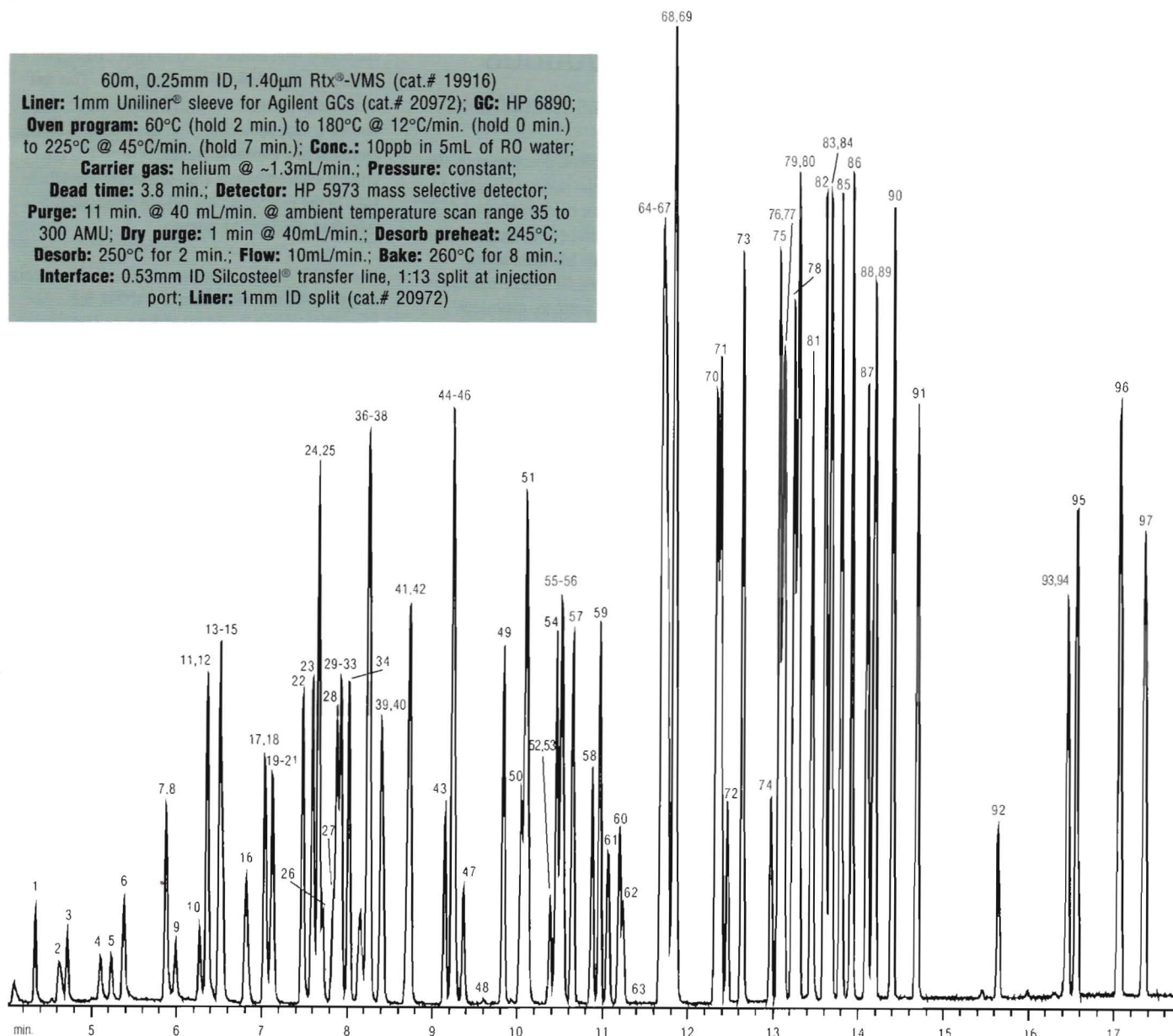
Rtx®-VMS (Fused Silica)

ID	df (µm)	Temp. Limits	30-Meter	60-Meter
0.25mm	1.40	-40 to 240/260°C	19915	19916
0.32mm	1.80	-40 to 240/260°C	19919	19920
0.45mm	2.55	-40 to 240/260°C	19908	19909
ID	df (µm)	Temp. Limits	20-Meter	40-Meter
0.18mm	1.00	-40 to 240/260°C	49914	49915

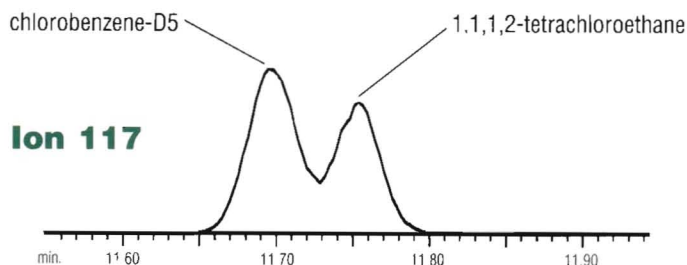
Figure 2

A 60m, 0.25mm ID Rtx®-VMS column can operate with a 60°C starting temperature to match the purge and trap cycle time while still achieving good gas resolution.

60m, 0.25mm ID, 1.40µm Rtx®-VMS (cat.# 19916)
Liner: 1mm Uniliner® sleeve for Agilent GCs (cat.# 20972); **GC:** HP 6890;
Oven program: 60°C (hold 2 min.) to 180°C @ 12°C/min. (hold 0 min.) to 225°C @ 45°C/min. (hold 7 min.); **Conc.:** 10ppb in 5mL of RO water;
Carrier gas: helium @ ~1.3mL/min.; **Pressure:** constant;
Dead time: 3.8 min.; **Detector:** HP 5973 mass selective detector;
Purge: 11 min. @ 40 mL/min. @ ambient temperature scan range 35 to 300 AMU; **Dry purge:** 1 min @ 40mL/min.; **Desorb preheat:** 245°C;
Desorb: 250°C for 2 min.; **Flow:** 10mL/min.; **Bake:** 260°C for 8 min.;
Interface: 0.53mm ID Silcosteel® transfer line, 1:13 split at injection port; **Liner:** 1mm ID split (cat.# 20972)

**Figure 3**

The Rtx®-VMS column is optimized to resolve chlorobenzene-D5 and 1,1,1,2-tetrachloroethane (peaks 64 and 67); these difficult-to-resolve compounds share quantitation ion 117.



**Contact Restek's
 Technical Service Team at
support@restekcorp.com to
 determine the right volatiles
 column for your
 analysis.**

Siltek™ Deactivation

Improved Analysis of Samples Containing Acidic, Basic, and Neutral Compounds

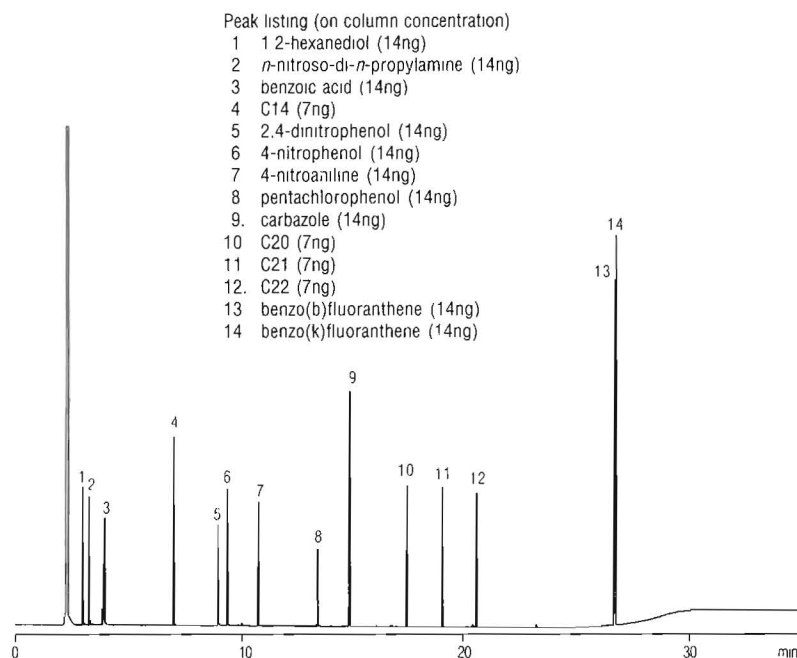
by Dr. David Smith, R&D Chemist, and Kristi Sellers, Innovations Team Chemist

Siltek™ deactivation (patent pending) offers the widest range of surface inertness for basic, acidic, and neutral compounds. This unique surface deactivation also displays high thermal stability, virtually zero bleed, and resistance to caustic degradation. The Siltek™ surface is not susceptible to cleavage, which forms acidic silanols on traditional deactivated surfaces. This resistance to silanol formation greatly reduces bleed, breakdown and adsorption of active compounds.

Another major benefit of Siltek deactivation is the potential to "regenerate" the surface by solvent rinsing. Most contamination can be removed from the Siltek™-deactivated surface with simple solvent rinsing. The Siltek™ surface has been tested without adverse effects after 10 minute sonication in solvents such as water, acetone, methylene chloride, methanol, and hexane.

Figure 1

Siltek™ liner, Press-Tight® connector, and guard column show excellent inertness even with low concentration (ng) of active compounds.



Improved analyses of samples containing acidic, basic, and neutral compounds (even through a 30m guard column!)

To illustrate the highly inert Siltek™ surface, a mixture of compounds was analyzed using a Siltek™ inlet liner and a 30m Siltek™ guard column connected with a Siltek™ Press-Tight® connector to an XTI®-5 analytical column (Figure 1). This test mix contains a wide variety of acidic, basic, and neutral compounds, and is an excellent test of the inertness of a chromatographic system. The on-column concentrations (ng) are noted parenthetically in the compound list. All the compounds exhibited excellent response.

The Siltek™ surface offers an unsurpassed inertness range, combined with thermal stability, the prevention of compound breakdown and adsorption, and the economy of potential solvent regeneration.

30m, 0.25mm ID, 0.25µm XTI®-5 (cat.# 12223) connected to a 30m, 0.25mm ID Siltek™ guard column* with a Siltek™ Universal Press-Tight® connector (cat.# 20480)

Liner: Siltek™ single gooseneck for Agilent GCs (cat.# 20798-214.1)

Sample: 0.5µL split injection on XTI® mix; 10:1 split ratio; split vent flow: 80mL/min.; **GC:** HP 6890; **Oven program:** 100°C to 235°C @ 6°C/min. to 330°C @ 15°C/min. (hold 10 min.); **Carrier gas:** hydrogen;

Flow rate: 5mL/min.; **Pressure:** constant; **Injector temp.:** 330°C; **FID temp.:** 330°C

* Restek does not offer 30-meter Siltek™ guard columns as a stock item. It was used in this analysis to illustrate inertness at unconventional guard column lengths

for **moreinfo**

For more information and a complete product listing, request the **Siltek™ Deactivation Benefits Brochure** (lit. cat.# 59803A).

For Siltek™ inlet liners, add the corresponding suffix number to your Restek liner catalog number. See the Annual Chromatography Products Guide for a complete inlet liner listing.

Siltek™ Inlet Liners					
qty.	Siltek™		Siltek™ with Siltek™ wool		Siltek™ with CarboFrit™
each	-214.1	addl. cost	-213.1	addl. cost	-216.1 addl. cost
5-pk.	-214.5	addl. cost	-213.5	addl. cost	-216.5 addl. cost
25-pk.	-214.25	addl. cost	-213.25	addl. cost	-216.25 addl. cost

Siltek™ Guard Columns			
nominal ID	nominal OD	5-meter	10-meter
0.25mm	0.37 ± 0.04mm	10026	10036
0.32mm	0.45 ± 0.04mm	10027	10037
0.53mm	0.69 ± 0.04mm	10028	10038

Chiral GC Column Kits



Economical Method Development

by Christine Vargo, GC Columns Marketing Manager

- ✓ Application-specific chiral column kits simplify column selection for the analysis of environmental pollutants, pharmaceuticals, essential oils, & juices
- ✓ Broad range of columns and selectivities
- ✓ Convenience and cost savings with kit purchase

Any carbon atom that is bonded to four different functional groups is termed a chiral carbon. Molecules containing one or more of these carbon centers are considered chiral molecules. Chiral centers can exist in two forms called enantiomers. These two forms are non-superimposable mirror images of each other, but have similar properties. However, they generally possess different aroma and flavor characteristics; and more importantly, possess differences in toxicity and biological activity.

Chiral chromatography is the separation of enantiomeric compounds. The common liquid stationary phases used in gas chromatography (GC) resolve components from one another, but they do not possess adequate selectivity for enantiomeric separation. Stationary phases incorporating modified cyclodextrins have the greatest ability to separate chiral compounds using GC. By adding cyclodextrins to the bonded cyanopropyl-dimethyl polysiloxane

(Rtx®-1701 column) stationary phase, the lifetime and overall performance of these columns is greatly enhanced over columns made from pure cyclodextrins. Restek has developed seven unique, modified cyclodextrin phases for the separation of chiral compounds.

If you are unsure which chiral column to use for your analysis, try one of Restek's new application-specific chiral column kits. We have pre-selected our most recommended chiral columns for your specific applications. Whether you are analyzing essential oils, flavors, juices, pharmaceuticals, or environmental pollutants, we have a kit for you!

for more info

For more information on these kits and to see applications, request the brochure "**Chiral Capillary Columns & Kits**" (lit. cat.# 59242).

Pharmaceutical Chiral Column Kits

Dimensions & Columns	cat.#
30m, 0.25mm ID, 0.25µm: Rt-BDEXcst™ & Rt-BDEXsm™ columns (one each)	13190
30m, 0.32mm ID, 0.25µm: Rt-BDEXcst™ & Rt-BDEXsm™ columns (one each)	13191

Environmental Chiral Column Kits

Dimensions & Columns	cat.#
30m, 0.25mm ID, 0.25µm: Rt-BDEXcst™ & Rt-BDEXsm™ columns (one each)	13192
30m, 0.32mm ID, 0.25µm: Rt-BDEXcst™ & Rt-BDEXsm™ columns (one each)	13193

Juices Chiral Column Kits

Dimensions & Columns	cat.#
30m, 0.25mm ID, 0.25µm: Rt-BDEXse™, Rt-BDEXsm™, & Rt-γDEXsa™ columns (one each)	13194
30m, 0.32mm ID, 0.25µm: Rt-BDEXse™, Rt-BDEXsm™, & Rt-γDEXsa™ columns (one each)	13195

Essential Oils Chiral Column Kits

Dimensions & Columns	cat.#
30m, 0.25mm ID, 0.25µm: Rt-BDEXsm™, Rt-BDEXse™, Rt-BDEXsa™, & Rt-BDEXsp™ columns (one each)	13196
30m, 0.32mm ID, 0.25µm: Rt-BDEXsm™, Rt-BDEXse™, Rt-BDEXsa™, & Rt-BDEXsp™ columns (one each)	13197

August 1, 2000 Update to USP <467> Organic Volatile Impurities Analysis

by Christopher Cox, Director of R&D

Several revisions to USP <467> were published in Supplement Two of USP-24/NF-19, effective August 1st, 2000. USP has officially removed the limit test requirements for benzene from any article specified to be tested by <467> for organic volatile impurities, except where a specific limit for benzene is in the individual monograph. The new mixtures, shown at right, can be used for <467> when no benzene limit is specified in the monograph.

Restek also offers several USP <467> calibration mixtures with benzene, and an individual component ethylene oxide mixture for monographs requiring those limit tests. Restek will continue to follow the revisions to <467> and develop new calibration mixtures to meet the latest requirements. Please call technical service for the latest available mixtures.

USP <467> Calibration Mix #6

chloroform	60µg/mL
1,4-dioxane	380 µg/mL
methylene chloride	600µg/mL
trichloroethylene	80µg/mL

prepared in methanol, 1mL per ampul

each	cat. # 36008
10-pack	cat. # 36108

USP <467> Calibration Mix #7

chloroform	60µg/mL
1,4-dioxane	380µg/mL
methylene chloride	600µg/mL
trichloroethylene	80µg/mL

prepared in dimethylsulfoxide, 1mL per ampul

each	cat. # 36009
10-pack	cat. # 36109

New Books!

Information on GC, HPLC, and LC/MS Analysis

by Jack Crissman, Technical Training and Education Manager

Chromatographic Analysis of Environmental and Food Toxicants

T. Shibamoto, Marcel Dekker, 1998, 331pp., ISBN 0-8247-0145-3

cat.# 21085

Covering such topics as conventional chromatographic methods, accommodating clean up, and preparing substances for further instrumental analysis, this practical resource is the only available book showing how to choose the most effective techniques for assessing the toxicity of chemicals in both food and the environment.

Chromatographic Detectors. Design, Function, and Operation

R. P. W. Scott, Marcel Dekker, 1996, 514pp., ISBN 0-8247-9779-5

cat.# 21090

Written by a seasoned chromatographer with over 40 years experience in the field, this reference comprehensively covers the design, construction, and operation of gas chromatography, liquid chromatography, and thin-layer chromatography detectors—all in one convenient, up-to-date source.

Chromatographic Analysis of Pharmaceuticals. 2nd Edition

J. A. Adamovics, Marcel Dekker, 1997, 527pp., ISBN 0-8247-9776-0

cat.# 21089

Revised and updated throughout, the second edition of this highly-lauded reference explores the chromatographic methods used for the measurement of drugs, impurities, and excipients in pharmaceutical preparations, such as tablets, ointments, and injectables. It contains a 148-page listing of chromatographic data of over 1300 drugs and related substances, including matrix analysis, sample handling procedures, column packings, mobile phase, mode of detection, and more.

Introduction to Analytical Gas Chromatography. 2nd Edition

R. P. W. Scott, Marcel Dekker, 1998, 397pp., ISBN 0-8247-0016-3

cat.# 21084

Completely updated to maintain relevancy in a rapidly changing field, the second edition of this text furnishes comprehensive information requisite to the successful practice of gas chromatography; covering the principles of chromatographic separation, the chromatographic process from a physical chemistry perspective, instrumentation for performing analyses, and various operational procedures.

Techniques and Practice of Chromatography

R. P. W. Scott, Marcel Dekker, 1995, 395pp., ISBN 0-8247-9460-5

cat.# 21088

Introducing scientists of all disciplines to the chromatographic process and how it functions, this unique, easy-to-read volume treats chromatography as a single subject discussing the basic principles and theory of chromatographic separation, as well as specific chromatographic procedures, including gas, liquid, and thin-layer chromatography.

Liquid Chromatography-Mass Spectrometry. 2nd Edition

W. M. A. Niessen, Marcel Dekker, 1999, 634pp., ISBN 0-8247-1936-0

cat.# 21086

This extensively updated reference provides a comprehensive account of the latest developments, principles, and applications of combined liquid chromatography-mass spectrometry and related techniques.

Handbook of HPLC

E. Katz, R. Eksteen, P. Schoenmakers, and N. Miller, Eds., Marcel Dekker, 1998, 989pp., ISBN 0-8247-9444-3

cat.# 21087

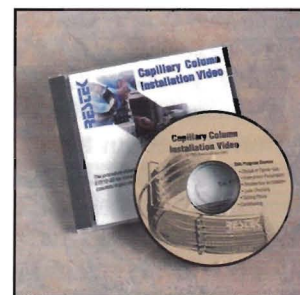
This book discusses the principles, techniques, and instrumentation involving HPLC within a detailed comprehensible framework, delineating its usage in separation, purification, and detection processes across a wide variety of disciplines from industry to applied research.



Restek is Your Technical Literature Source!

"I just received the Fast Facts on pesticide reference materials. I love it! It's great to see all of the items that I need on one sheet. I will keep it handy. I really appreciate the work that it takes to put out such a pamphlet, but I also do appreciate the amount of time that it saves me while tracking down item numbers and prices."

Jeff Westerlund, Sound Analytical



Capillary Column Installation Video

The technical wizards at Restek produced an instructional video that takes the mystery and frustration out of capillary column installation. This essential resource covers critical points in the proper installation of a capillary column such as: choice of carrier gas, instrument preparation, trouble-free installation, leak checking, setting carrier and detector gas flows, and column conditioning. The installation sequence allows you to install your capillary column correctly, minimize downtime, and get your GC up and running quickly. The video follows ASTM Method E1510-93 for installing fused silica open tubular capillary columns in gas chromatographs to ensure your lab conforms with industry standards.

VHS: cat.# 20490 (ea.)

PAL: cat.# 20491 (ea.)

CD-ROM: cat.# 20499 (ea.)

Visit www.restekcorp.com
for a complete list of over **50** books
available from Restek.

HPLC Analysis of Fat-Soluble Vitamins

by Greg France, HPLC Product Marketing Manager

- ✓ Excellent peak shape of fat-soluble vitamins.
- ✓ Minimize silanol interactions.

Vitamins encompass a wide range of organic compounds. They are required by the human body in only minute quantities. Yet, they have significant impact at the cellular level. Vitamins play a vital role in converting fat and carbohydrates to energy, regulating metabolism, and other bodily functions.

The market for nutritional and dietary supplements is growing tremendously. And, as these nutraceutical and dietary supplement markets grow, the need for simple, rugged, and accurate methods to analyze vitamins becomes increasingly important.

Vitamins can be broadly classified into two groups, water-soluble vitamins and fat-soluble vitamins. Fat-soluble vitamins include constituents such as Vitamin A (retinol) and Vitamin D3 (cholecalciferol). Vitamin A has been found to play an important role in proper growth and eye function, while Vitamin D is required for proper bone and tooth growth.

The Restek Ultra C18 column is an ideal first choice to separate a wide range of fat-soluble vitamins. Its very retentive, high-purity packing exhibits excellent peak shape. The silica has a

carbon load of 20% and is fully end-capped, which eliminates any unwanted silanol interactions and improves column-to-column reproducibility.

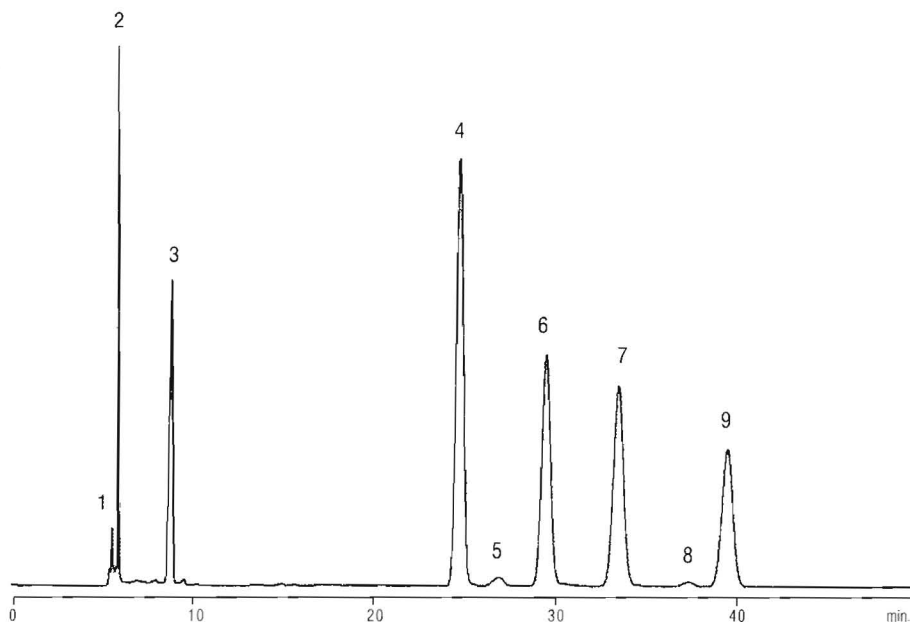
The fat-soluble vitamins are extremely hydrophobic and must be dissolved in an organic solvent. A mixture of six fat-soluble vitamins were separated using an Ultra C18 column (Figure 1). The analytes were dissolved in diethyl ether. The sample solution was then analyzed using a simple isocratic mobile phase (acetonitrile:methanol [90:10]) with a stepped flow rate (1mL/min. from 1 minute to 5 minutes, then 2mL/min. from 5 minutes to 50 minutes). All six constituents are easily resolved and show excellent peak shape

formoreinfo

For more information, request the applications note "HPLC Analysis of Vitamins Using an Ultra C18 Column" (lit. cat.# 59181).

Figure 1

The Ultra C18 column provides excellent peak shape and resolution of fat-soluble vitamins.



Peak List:	Conc.: (mg/mL)
1. solvent front	
2. menadiene (vitamin K3)	0.45
3. all-trans-retinol (vitamin A)	0.34
4. vitamin D3	0.4
5. unknown	
6. alpha tocopherol (vitamin E)	2.4
7. alpha tocopherol acetate (vitamin E Acetate)	2.4
8. unknown	
9. phyloquinone (vitamin K1)	0.84

Sample:

All analytes dissolved in HPLC-grade diethyl ether

Column:	Ultra C18
Catalog #:	9174575
Dimensions:	250 x 4.6mm
Particle size:	5µm
Pore size:	100Å
Conditions:	
Mobile phase:	acetonitrile:methanol (90:10)
Time (min.):	Flow (mL/min.):
1.00	1.00
5.00	1.00
5.01	2.00
50.0	2.00
Temp.:	30°C
Det.:	UV @ 280nm



Welcome Greg France!

Greg joins the Restek family as the HPLC Product Marketing Manager. He holds a B.A. in Chemistry from the College of the Holy Cross in Massachusetts

and an M.B.A. from St. Joseph's University in Pennsylvania. Bringing over 12 years experience in Analytical Chemistry, Greg is a valuable asset to the HPLC Team. Join us in welcoming him!

Particle Size: 5µm	1.0mm ID cat.#	2.1mm ID cat.#	3.2mm ID cat.#	4.6mm ID cat.#
30mm length	9174531	9174532	9174533	9174535
50mm length	9174551	9174552	9174553	9174555
100mm length	9174511	9174512	9174513	9174515
150mm length	9174561	9174562	9174563	9174565
200mm length	9174521	9174522	9174523	9174525
250mm length	9174571	9174572	9174573	9174575

Rtx[®]-VGC Columns

Fast, Accurate Volatile Compounds Analysis

by Christopher English, Applications Chemist

- ✓ 23-minute analysis time with Rtx[®]-502.2 confirmational column.
- ✓ Faster oven cycle times.
- ✓ Extremely low bleed—ideal for PID and ELCD detectors.

The determination of purgable halocarbons and aromatics in solid waste and waste water by photoionization detector (PID) in series with an electrolytic conductivity detector (ELCD) is detailed in US Environmental Protection Agency (EPA) Methods 601/602. The PID responds to aromatic and unsaturated compounds, while the ELCD responds to halogenated compounds.

There are several columns that have been used for the analysis of EPA Method 601/602 compounds, however Restek's new Rtx[®]-VGC column offers improved resolution and faster analysis time. In addition to the 32 compounds listed in Method 601/602, many labs also include methyl-tert-butyl ether (MTBE) and several surrogate standards as part of the analysis. We have also found that many labs use the shorter list of compounds (8021/8020, 601/602) by 8021 criteria.

A 75m, 0.45mm ID, 2.55µm Rtx[®]-VGC column resolves the Method 601/602 compounds, MTBE, and the 5 most commonly used surrogate standards in 20 minutes (Figure 1). In addition, the early eluting gases can be separated at a starting temperature of 40°C. This higher starting temperature allows a faster oven cycle time which results in more usable instrument time. The low bleed characteristic of the Rtx[®]-VGC is critical for both the PID and ELCD. Excess column bleed can degrade sensitivity and increase instrument downtime.

Confirmational analysis can be performed using a 75m, 0.45mm ID, 2.55µm Rtx[®]-502.2 column (Figure 1). This column offers a different phase selectivity compared to the Rtx[®]-VGC which produces significant differences in retention time as well as retention time order (see peaks 12

through 15 in the ELCD chromatograms). Simultaneous confirmation is possible using dual PID/ELCDs, since both columns can be operated using the same temperature program conditions and result in similar analysis times.

The analysis of EPA Method 601 and 602 can be improved with the use of the Rtx[®]-VGC column. The optimized selectivity of this new stationary phase results in better resolution of these volatile compounds and reduces analysis time to 20 minutes. Due to its different selectivity, the Rtx[®]-502.2 makes an excellent confirmation column to the Rtx[®]-VGC and can be operated simultaneously using dual PID/ELCDs.

Conditions for Figure 1

Primary column: 75m, 0.45mm ID, 2.55µm Rtx[®]-VGC (cat.# 19409); **Confirmation column:** 75m, 0.45mm ID, 2.55µm Rtx[®]-502.2 (cat.# 10986);

Concentration of analytes: 10ppb in 5mL of RO water; **Concentrator:** Tekmar LSC3100 Purge and Trap **Trap:** Vocab3000; **Purge:** 11 min. @ 40mL/min. **Dry purge:** 1 min. @ 40mL/min.; **Desorb:** 2 min. @ 250°C for 2 min.;

Bake: 260°C for 8 min.; **Interface:** direct connection from concentrator to column;

Transfer line: Silcosteel[®]-treated tubing **GC:** Finnigan 9001; **Carrier gas:** helium @ ~10 mL/min.;

Pressure: constant; **Dead time:** 2.04 min.

Adjust dichlorodifluoromethane to a retention time of 2.47 minutes @ 40°C **Oven program:** 40°C (hold 2 min.) to 58°C @ 4°C/min. (hold 0 min.) to 90°C @ 10°C/min. (hold 5 min.) to 220°C @ 40°C/min. (hold 5 min.);

Det.: µGold Tandem PID/Hall; **PID makeup:** 7mL/min, purge 7mL/min, set @ 0.35mV, base temp 200°C.;

Hall 2000: RxnGas 25mL/min., RxnTemp.940°C, propanol flow 470)µL/min.

Standard	cat.#
502.2 mix#1	30042
624 cal mix #2	30021
624 cal mix #3	30022
MTBE	30402
1,4-dichlorobutane	30227
fluorobenzene	30030
1-chloro-2-fluorobenzene	30040
4-bromo-1-chlorobenzene	30230
bromochloromethane	30225

Peak List for Figure 1

1. dichlorodifluoromethane	14. carbon tetrachloride	27. 1,1,2-trichloroethane
2. chloromethane	15. 1,1,1-trichloroethane	28. dibromochloromethane
3. vinyl chloride	16. benzene	29. chlorobenzene
4. bromomethane	17. 1,2-dichloroethane	30. ethylbenzene
5. chloroethane	18. fluorobenzene	31. 1-chloro-2-fluorobenzene
6. trichlorofluoromethane	19. trichloroethene	32. bromoform
7. 1,1-dichloroethene	20. 1,2-dichloropropane	33. 1,4-dichlorobutane
8. methylene chloride	21. bromodichloromethane	34. 1,1,2,2-tetrachloroethane
9. <i>trans</i> -1,2-dichloroethene	22. 2-chloroethyl-vinyl-ether	35. 1,3-dichlorobenzene
10. methyl- <i>tert</i> -butyl-ether	23. <i>cis</i> -1,3-dichloropropene	36. 1,4-dichlorobenzene
11. 1,1-dichloroethane	24. toluene	37. 1,2-dichlorobenzene
12. bromochloromethane	25. tetrachloroethene	38. 4-bromo-1-chlorobenzene
13. chloroform	26. <i>trans</i> -1,3-dichloropropene	

Rtx[®]-VGC (Fused Silica)

Temperature limits: -40 to 240/260°C

ID	df (µm)	30-Meter	60-Meter	75-Meter
0.25mm	1.40	19415	19416	
0.32mm	1.80	19419	19420	
0.45mm	2.55	19408		19409

Rtx[®]-502.2 (Fused Silica)

Temperature limits: -20 to 250/270°C

ID	df (µm)	30-Meter	60-Meter	105-Meter
0.25mm	1.40	10915	10916	
0.32mm	1.80	10919	10920	10921
0.53mm	3.00	10908	10909	10910

ID	df (µm)	75-Meter
0.45mm	2.55	10986

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

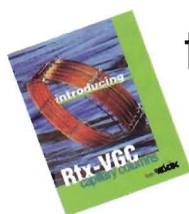
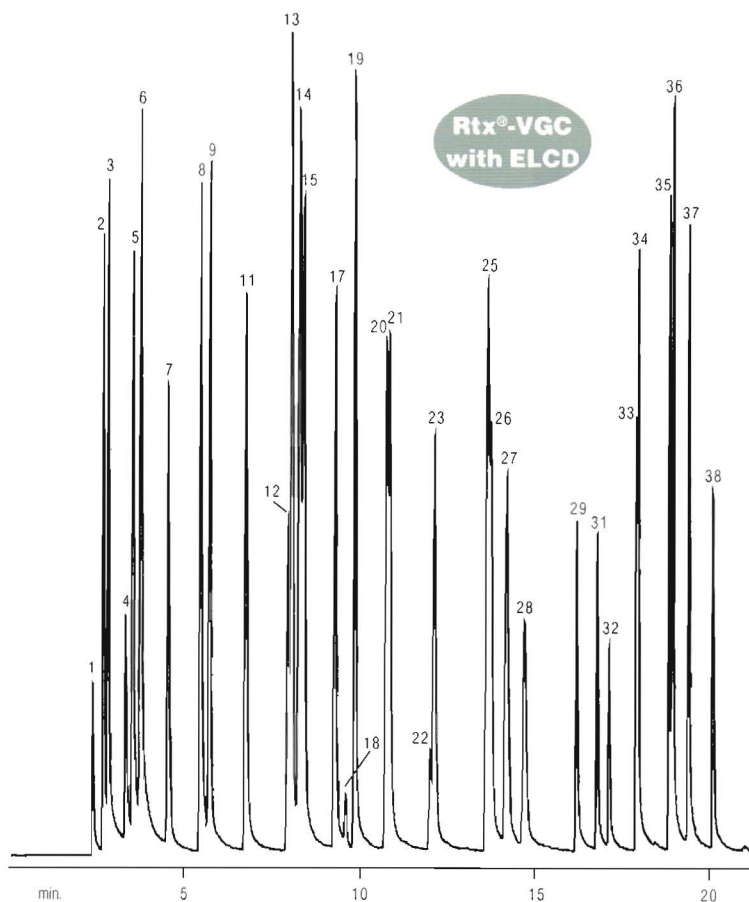
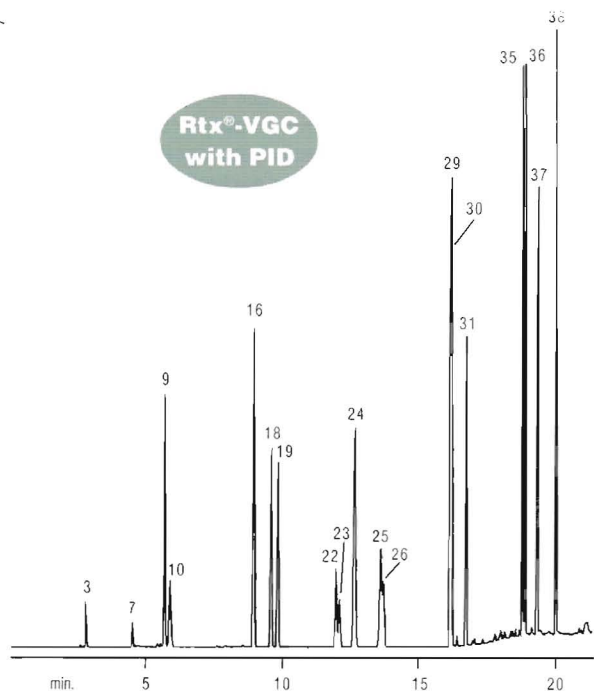


Having trouble choosing the right GC column for your volatile compounds analysis?

See the helpful chart on page 447 of the *2001 Chromatography Products Guide* for details (lit. cat.# 59960). Also, Restek's knowledgeable Tech Service Team is ready to answer your toughest analytical questions.

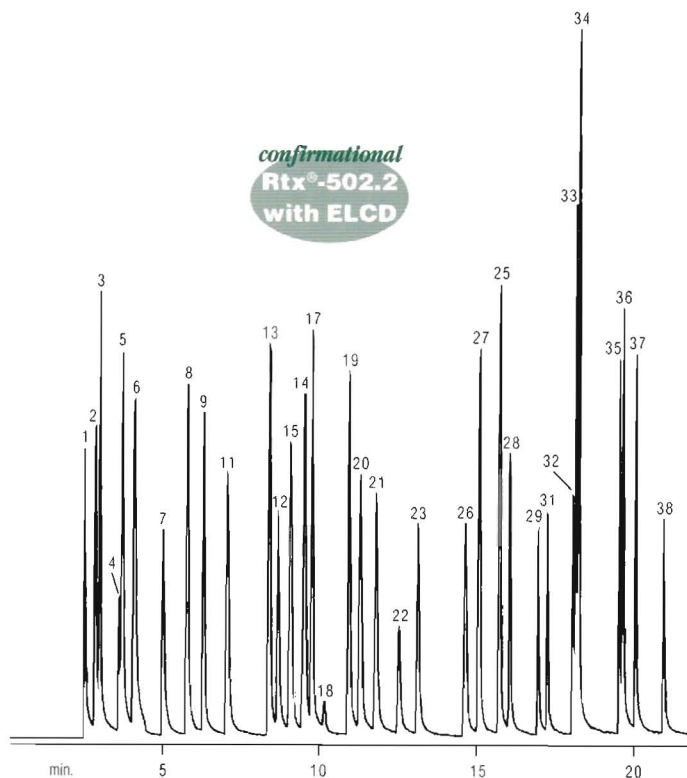
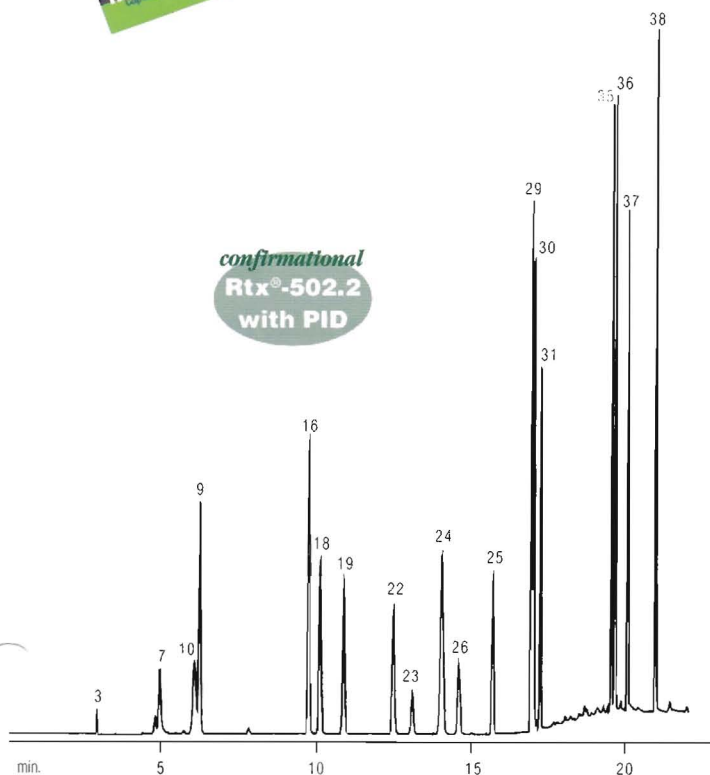
Figure 1

Using the Rtx®-VGC and Rtx®-502.2 columns with dual PID/ELCD resolves volatile organic compounds in 23 minutes.



for more info

Request the
"Rtx®-VGC Columns Brochure"
(lit. cat.# 59217).



Koni's Korner

Food Technology: Are we as safe as we think?

by Dr. Konrad Grob



Food technology is developing at an ever-faster rate. Clever treatments can make meat look more attractive or convert by-products to a well-paid food component; modifications or additions enable

sales-stimulating health claims and new packaging materials allow longer storage. To the shareholders, the strategy of pushed innovation is presented as a means to increase profitability. To the consumers, it is presented as a means to make "improved" products. Innovation to find cheaper technology (e.g., the addition in wine of extracts from oak chips instead of storing the wine in oak barrels) is almost inevitable in a competitive economy.

I do not want to discuss the benefits of such innovation, but instead draw attention to the risks. Those responsible for the innovation say they conduct thorough "scientific" research and "rigorous control" of all risks, but when food control authorities ask specific questions, they usually discover the research was not done because it would have been too expensive or there was no time because of the intense competition. An example: it is about two cents (US) cheaper to remove the free fatty acids from a liter of raw edible oil by steam treatment compared to the older method of extraction with alkali. As steam treatment requires heating the oil at 240-260°C for a few hours, it seems obvious that the effects of such extremely high temperatures should be investigated. For instance, up to 60% of the essential linolenic acid is isomerized to various trans components. The industry never checked whether this can be tolerated. I hope the consumer profits from the two-cent savings—he carries the risks anyway.

By eating we expose our bodies, perhaps our most valuable asset, to these products. Often only in the hospital (e.g., when cancer ruins our organism) do we start seriously thinking about such risks. Nobody knows how many cases of cancer, heart disease, obesity, and other diseases are caused by unwanted side-effects of food technology, but it might be many more than we think. During the last 15 years, Europe has dealt with the threat of bovine

spongiform encephalopathy (BSE), also called mad cow disease. If BSE had been transferred from animals to humans equally as well as between certain animals, most of the British and a good portion of the continental Europeans could have died already or would die during the next ten years, resulting in hundreds of millions of deaths. It is amazing that we have seen only 90 victims so far. This case should teach us a lesson. It was caused by the use of "valuable by-products" (i.e., wastes from animal bodies) and a more "efficient" treatment using the knowledge of the time that heating to 120°C should rule out infection. However, this was wrong; one of these errors caused by our incomplete knowledge.

Every new process or component added to our foods introduces risks. Most risks are small, but there are thousands of them. For a food control laboratory, such as the one where I work, this is frightening. For example, in 1995 we were testing olive oil for adulteration with specially-treated sunflower oil when we were disturbed by an extra peak. It turned out to be bisphenol-A diglycidyl ether (BADGE) released from the internal coating of the food can, present at a concentration of 80ppm. Considered as a suspect carcinogen, the Swiss legal limit was 20ppb. 80ppm for a possible carcinogen was astounding! Billions of food cans contained BADGE at levels exceeding 1ppm. Millions were removed from the Swiss market and authorities in the EU became active. Later BADGE was shown to be non-carcinogenic. Hence, we were lucky once again. However, it only took a few months to realize that the migrant from the food containers to the food usually forms a forest of peaks, with dozens of giant trees whose identities—not to mention their toxicity—nobody has investigated.

Many analysts know that food packaging materials often release forests of peaks, rendering their search for a given compound difficult. Usually they have no time to identify these peaks, or are even told not to do so. Many producers do not want to know about them because then they would carry the responsibility. It is a horror to any manager that a migrant from his product could be a potent carcinogen, and to have journalists or lawyers show him documents that prove he knew about it for years. Ignorance may be bliss to them.

With the fast changes in food technology safety is a delicate issue. We analysts have the most powerful

tools in our hands to know that food safety does not meet the standards communicated to consumers. Some of us daily see the forests of unknown peaks, the artifacts from technological processes, and contaminants. The probability is high that among the ten thousands of compounds, a few are highly toxic. Many of the untested compounds we ingest in amounts that exceed those required to prevent pregnancy or to change our thinking (e.g., LSD or psychopharmaca). They might make us more beautiful, but, unfortunately, many more chemicals cause damage than help us. The problem is that we cannot spit them out once we notice that they are bad.

We analysts carry the burden of knowledge and responsibility to inform; even if it is to people who do not want to know. This is a difficult position. The analyst working for producers is the spoilsport of the technologists who are enthusiasts of their new product and of those who want to see the money coming in soon. The others working for the public usually get to hear that there is no regulation on the subject, that there is no sufficient proof of a harmful effect and, hence, that nothing can be done, or that there is no longer anybody around who could investigate the subject. In fact, mechanisms of economy get stronger, whereas authorities defending the public interests shrink.

I believe and hope that, in the end, we Europeans will escape BSE with less than 1000 human deaths. We have no idea how many diseases and deaths are due to minor risks caused by food technology, but we should take notice that the probability of being severely hit constantly increases. This makes one wonder if the game about higher profitability plays with our safety?



Selection Guide for Polar WAX GC Column Phases

- Information on six polyethylene glycol (PEG) columns.
- PEG stationary phase selection guide.
- Applications for each PEG column.

Call Restek at 800-356-1688 or 814-353-1300, ext. 5, or contact your local Restek representative for your copy.
lit. cat.# 59890 (domestic), lit. cat.# 59891 (international).

Analytical Reference Materials

ASTM Method D6042-96 for Plastic Container Testing

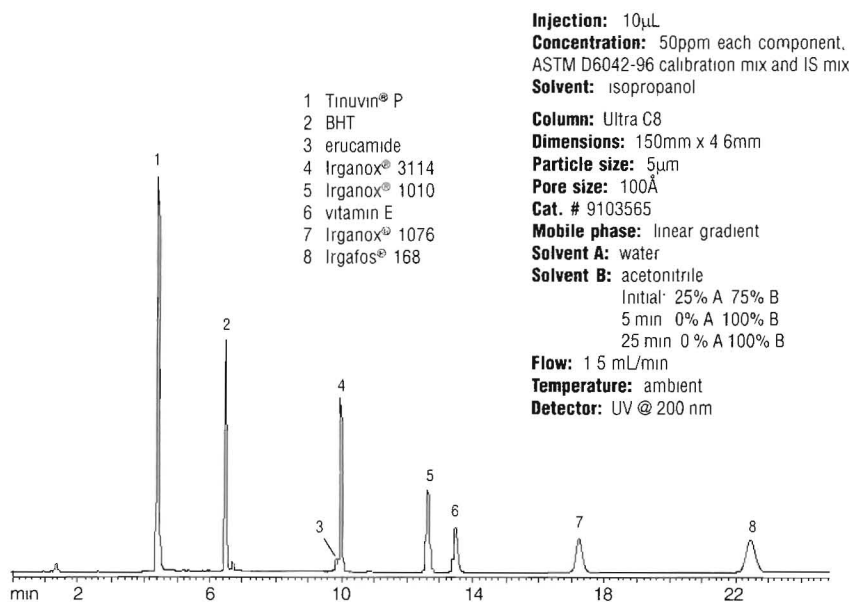
by Christopher Cox, Director of R&D

American Society for Testing and Materials (ASTM) Method D6042-96—*Standard Test Method for Determination of Phenolic Antioxidants and Erucamide Slip Additives in Polypropylene Homopolymer Formulations Using Liquid Chromatography*—is a “consensus” or “referee” method used between plastic manufacturers and the pharmaceutical companies that purchase plastic containers. Plastic container manufacturers use this test to ensure the quality of their product to their pharmaceutical customers. Pharmaceutical companies also specify this test and provide their own lists of target compounds and concentration limits in purchase agreements.

This test uses isopropanol extraction, HPLC separation, and UV detection. Restek offers a variety of reverse phase HPLC columns suitable for these types of separations. Figure 1 shows the separation of the test validation mixture on a Restek Ultra C8 column. Restek also has designed a new analytical reference material to validate this method. This mixture contains the common antioxidants and slips listed in ASTM D6042-96, along with BHT.

Figure 1

Restek's new standards are used to ensure container quality as outlined in ASTM Method D6042-96.



ASTM D6042-96 Calibration Mix

contains:

BHT
erucamide slip
vitamin E
Irgafos® 168
Irganox® 3114
Irganox® 1010
Irganox® 1076

50µg/mL each in isopropanol, 1mL per ampul
supplied with a certificate of analysis

cat. # 31628	(ea.)
cat. # 31628-510	(5-pk.)
cat. # 31728	(10-pk.)

ASTM D6042-96 Internal Standard Mix

contains:

Tinuvin® P

51.8µg/mL in isopropanol, 1mL per ampul
supplied with a certificate of analysis

cat. # 31629	(ea.)
cat. # 31629-510	(5-pk.)
cat. # 31729	(10-pk.)

Other Additives Available From Restek on a Custom Basis

Similar methods for extractables in plastic pharmaceutical containers are cited in the United States Pharmacopoeia (USP), British Pharmacopoeia (BP), European Pharmacopoeia (EP), and Japanese Pharmacopoeia (JP). Customers may also have formulation-specific or product-specific test mixtures. Please contact us for a custom mixture. Our current inventory of raw materials includes these popular antioxidants. We have many more not listed and can obtain most compounds you may need.

- Ultrinox® 626
- Ethanox 330
- Santanox R
- Ethanox 323
- Ethanox 702
- Ethanox 703
- Irganox® L06
- Irganox® L57
- Irganox® L64
- Irganox® L109
- Irganox® L134
- Irganox® L135
- Irganox® 1035
- Vanlube® 81
- Vanlube® 848
- Vanlube® 7723
- Vanlube® AZ
- Vanlube® NA
- Vanlube® PCX
- Vanlube® SL
- Vanlube® SS



Reach for Restek!

Restek offers technical information and reference materials for United States Pharmacopoeia (USP) and European Pharmacopoeia (EP) Methods. Visit our website at www.restekcorp.com, email us at support@restekcorp.com, or call us at 800-356-1688 or 814-353-1300, ext. 4.

Rtx[®]-CLPesticides

Column Pair

Shortens Analysis Time & Decreases Breakdown

by Gary Stidsen, Innovations Team Manager

- ✓ Baseline resolution using same conditions.
- ✓ 22 Components in less than 24 minutes.

Laboratories have been analyzing chlorinated pesticides for many years. Throughout this time, method requirements have become more stringent, specifically the CLP pesticide method and SW-846 method 8081B. Changes in methods require tighter linearity, lower percent differences on continuing calibration checks, and a more inert system. Proper column selection, system set-up, and instrument maintenance are keys to meeting these more stringent requirements. Following the listed recommendations will reduce your laboratory's downtime and increase sample throughput.

Resolution

Typically, the first step of a chlorinated pesticides analysis is choosing a pair of columns to provide the best resolution of the compounds of interest. The most common list is the 20 components and two surrogates listed in Table 1. There are many traditional columns to choose for this analysis, such as Rtx[®]-1, Rtx[®]-5, Rtx[®]-35, Rtx[®]-50 and Rtx[®]-1701 columns. If the columns are used individually with control of the temperature program and flow rate, resolution may be obtained for all 22 compounds. However, a problem occurs when these columns are

run in parallel for quantitative and confirmational analysis. The flow rates and temperature program for one column are not optimal for the other.

The 22 chlorinated pesticides and surrogates can be resolved with the Rtx[®]-CLPesticides and Rtx[®]-CLPesticides2 columns run in parallel, which results in baseline resolution using the same conditions (Figure 1). This column pair exceeds the performance of other column pairs because the columns result in greater than 95% resolution of all components and can be baked out up to 330°C to reduce sample matrix interferences.

Linearity

Linearity of each component is one of the requirements for the analysis of chlorinated pesticides. Once a GC is set up, barring injection port contamination, linearity of the compounds will pass the required quality control limits of published methods. Typical calibration for chlorinated pesticides covers a 16-100 fold range. One factor affecting linearity is the flow rate of the make-up gas, whether nitrogen or argon/methane (P5) is used. The make-up gas flow rate should be set to optimize the linearity of α -BHC and methoxychlor. Generally, a higher make-up flow will result in better linearity for α -BHC and a lower make-up flow will result in better linearity for methoxychlor. Adjust the make-up gas so the linearity (percent relative standard deviation) of the two compounds is equal. See Table 1 for typical linearity of the chlorinated pesticides over a 16-fold range on the Rtx[®]-CLPesticides and Rtx[®]-CLPesticides2 columns.

System Inertness

The third criterion for initial calibration is checking the inertness of the inlet system by calculating the percent breakdown of 4,4'-DDT and endrin.

Figure 1

Rtx[®]-CLPesticides and Rtx[®]-CLPesticides2 achieve baseline resolution of the 22 chlorinated pesticides using the same conditions.

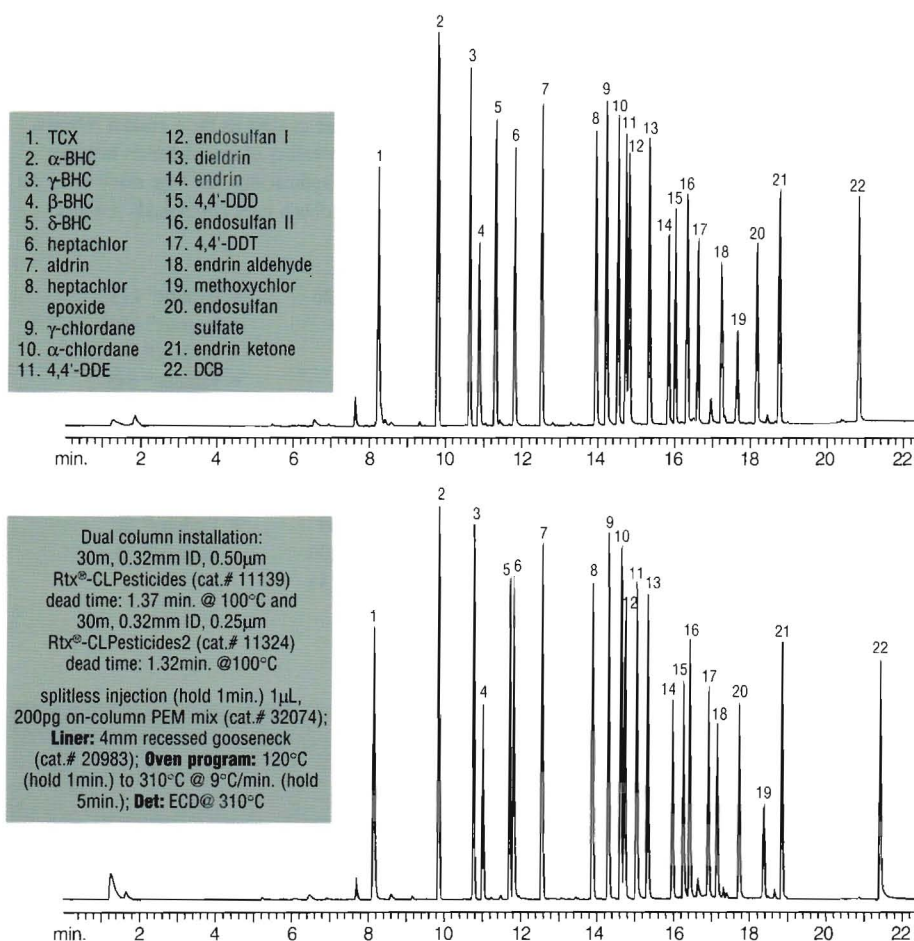


Table I

The most common list of chlorinated pesticides and their linearity.

Compound	Rtx [®] -CLP %RSD	Rtx [®] -CLP2 %RSD
TCX	7.7	2.0
α -BHC	12.0	12.8
γ -BHC	9.1	9.4
β -BHC	4.7	4.4
δ -BHC	13.9	13.1
heptachlor	3.2	3.8
aldrin	2.1	3.9
heptachlor epoxide	6.1	3.8
γ -chlordane	5.2	2.7
α -chlordane	5.7	2.0
p,p'-DDE	3.4	6.2
endosulfan I	4.8	2.3
dieldrin	0.8	2.7
endrin	4.0	4.0
p,p'-DDD	2.5	3.9
endosulfan II	3.8	2.0
p,p'-DDT	5.3	2.5
endrin aldehyde	6.1	6.7
methoxychlor	7.4	12.0
endosulfan sulfate	4.2	3.5
endrin ketone	3.1	4.7
DCB	8.8	11.0

The causes of 4,4'-DDT and endrin breakdown are numerous and happen in the injection port or in the first 0.5 meters of the capillary column. Generally, 4,4'-DDT breakdown is caused by dirty or oil-laden injection ports. Endrin breakdown is caused by active sites in the system, which may be from improper deactivation of the inlet sleeve or column, or cored septum particles in the sleeve. To prevent breakdown, use a Siltek™ sleeve. Siltek™ glass deactivation treatment is resistant to chemical attack and maximizes the inertness of the sample pathway (see pg. 4). Table 2 shows typical breakdown of endrin and 4,4'-DDT prior to calibration on the Rtx®-CLPesticides and Rtx®-CLPesticides2 columns.

Continuing Calibration

Once the analytical system is calibrated, samples are analyzed and calibration checks are acquired to verify system stability. Generally, a system will stay

within calibration as long as the samples being analyzed are "clean." When dirty samples are analyzed and calibration checks fail, maintenance is necessary. Removing the first 6-10 inches of column and replacing the septum and the liner is usually sufficient. The system then is checked for inertness and re-calibrated.

Conclusion

Chlorinated pesticide analysis is very demanding on laboratories. An instrument problem can cause a backlog of customer results and waste valuable lab time. One way to combat this is to reduce instrument cycle times so more analyses can be acquired per day. Use of the Rtx®-CLPesticides and Rtx®-CLPesticides2 column pair and Siltek™ sleeves, along with performing routine instrument maintenance, are a few ways to help your laboratory successfully analyze chlorinated pesticides.

Table II

Typical breakdown on a GC system prior to calibration and sample analysis using the Rtx®-CLPesticides and Rtx®-CLPesticides2 columns.

analyte	% breakdown	
	Rtx®-CLP	Rtx®-CLP2
Endrin	6.8	5.5
DDT	0.9	0.5

Rtx®-CLPesticides Column Kits

Kits include a universal angled "Y" Siltek™ Press-Tight® connector, 5m Siltek™ guard column, and columns listed.

nominal ID	cat.#
30m, 0.53mm ID, 0.50µm Rtx®-CLPesticides column and 30m, 0.53mm ID, 0.42µm Rtx®-CLPesticides2 column	11197
30m, 0.32mm ID, 0.50µm Rtx®-CLPesticides column and 30m, 0.32mm ID, 0.25µm Rtx®-CLPesticides2 column	11198
30m, 0.25mm ID, 0.25µm Rtx®-CLPesticides column and 30m, 0.25mm ID, 0.20µm Rtx®-CLPesticides2 column	11199

Siltek™ Inlet Liners

For Siltek™ inlet liners, add the corresponding suffix number to your Restek liner catalog number.

qty.	Siltek™		Siltek™ with Siltek™ wool		Siltek™ with CarboFrit™	
each	-214.1,	addl. cost	-213.1,	addl. cost	-216.1,	addl. cost
5-pk.	-214.5,	addl. cost	-213.5,	addl. cost	-216.5,	addl. cost
25-pk.	-214.25,	addl. cost	-213.5,	addl. cost	-216.25,	addl. cost

for more info

For detailed information on pesticide analysis, request "Guide to the Analysis of Chlorinated Pesticides" (lit. cat.# 59892).

Tech Tip from the Restek Wizards

Install the Right Jet in Your HP GC

by Rick Parmely, Chromatography Information Services Manager

When HP produced the FID jet for their 5890 GC, there was only one basic design. The jet is approximately 6.2cm long, and has a jet head that measures approximately 1.5cm. This jet was the only one usable for these FIDs for several years. With the introduction of the HP 6850/6890 GC, two additional FID configurations were designed. One is called "adaptable," which uses the same FID jet as the 5890 (part# 19244-80560). The second configuration is called "dedicated." The latter design is optimized for capillary use and requires a slightly shorter FID jet (part# G1531-80560). It measures roughly 4.3cm in length (Figure 1).

Which one do you select for your FID when using capillary columns? Physically, the two detector options look different in the GC oven. The "adaptable" detector configuration is much larger and allows for installation of 5890-style jets and packed columns. Being smaller, the "dedicated" detector configuration minimizes dead space; therefore it only accepts the shorter, "dedicated" FID jet. Restek offers both configurations. Please contact Restek technical service to select these and other jets for your GC.

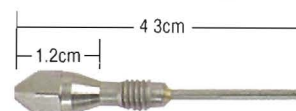
Figure 1

The Agilent 5890 and Agilent 6890 jets are configured differently.

5890/6890 adaptable jet configuration



6890 dedicated jet configuration



Jet	standard		High-Performance Silcosteel®	
5890 Standard	20670 (ea.)	20671 (3-pk.)	20672 (ea.)	20673 (3-pk.)
6890 Adaptable	20670 (ea.)	20671 (3-pk.)	20672 (ea.)	20673 (3-pk.)
6850/6890 Dedicated	21621 (ea.)	21682 (3-pk.)	21620 (ea.)	21683 (3-pk.)

Fast SimDist

Using a Thermo Orion-Rtx[®]-1 Capillary GC Column

by Christine Vargo, GC Columns Product Marketing Manager

Simulated distillation (SimDist) is a temperature-programmed analysis that determines the boiling point range distribution of petroleum samples using gas chromatography (GC). The American Society for Testing and Materials (ASTM) Method 2887 is a commonly used SimDist method for petroleum products having a final boiling point below 1000°F (540°C) and distillation ranges of at least 100°F (40°C).

Although the technique has been used with packed columns for more than 25 years, ASTM-2887 permits a choice of analytical conditions and column dimensions. Consequently, there is a wide variety of operating conditions used in different laboratories. Generally, a fast GC oven temperature program rate (e.g., 10–15°C/min.) and a high final oven temperature (e.g., 250–360°C) are employed to elute the wide range of molecular weights for hydrocarbons in SimDist analysis. Analysis times usually range from 24 to 60 minutes, not including the additional time needed to cool the column for the next run. Total cycle time of a SimDist analysis can range from 20 to 70 minutes. Figure 1 shows a typical chromatogram of SimDist analysis by capillary GC.

The wide range of boiling points in a petroleum sample and the long GC oven cycle times make SimDist analysis a perfect candidate for fast GC. Figure 2 shows similar SimDist analysis to Figure 1, but performed with an EZ Flash[®] system by Thermo Orion (formerly Orion Research, Inc.). The EZ Flash[®] system uses resistive heating

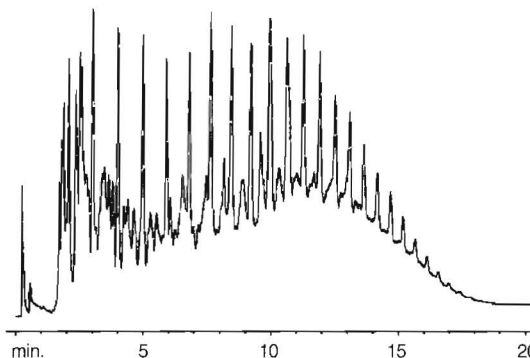
techniques and fast temperature programming to achieve increased productivity. The EZ Flash[®] attachment was installed on an HP (Agilent) 6890 GC equipped with an FID and a 5m x 0.32mm x 0.1µm Thermo Orion-Rtx[®]-1 column. (The EZ

Flash[®] system is compatible with 0.53mm ID, 0.32mm ID, and smaller [0.25–0.1mm ID] columns). This system enables a conventional GC to analyze a SimDist sample in under 2 minutes. Total cycle time with cool down is less than 7 minutes, which equates to almost 9 samples per hour!

Every minute counts when improving laboratory throughput and efficiency. Fast GC technology can let you out-run your competition—whether it's by optimizing your GC column dimensions, phases, program rates, or systems. For more information on how you can turn your everyday analysis into a fast GC analysis, contact Restek's Technical Service Team at support@restekcorp.com or call 800-356-1688 or 814-353-1300, ext. 4.

Figure 1

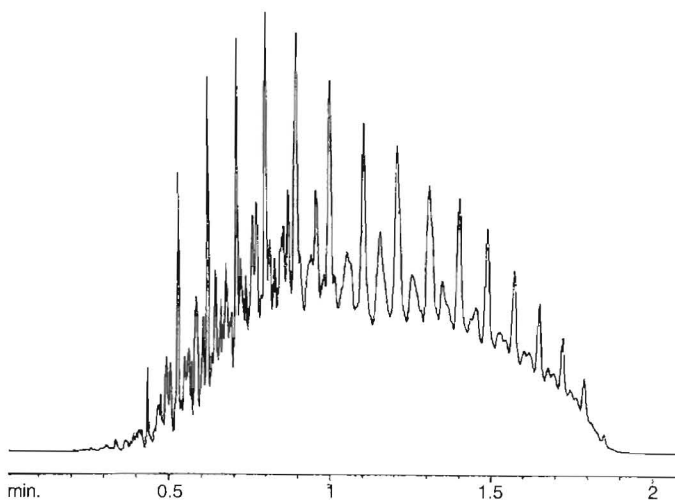
Traditional ASTM-2887 SimDist analysis results in a long GC cycle time.



10m, 0.53mm ID, 2.65µm Rtx[®]-2887 (cat.# 10199); 1.0µL direct injection of a 0.1 to 0.01 wt % of ASTM D2887 reference gas oil #1 (C6–C44) hydrocarbon standard in carbon disulfide;
Oven temp.: 35°C to 360°C @ 15°C/min. (hold 5 min.); Inj. & det. temp.: 360°C;
Carrier gas: nitrogen; Linear velocity: 112cm/sec. (15mL/min.)

Figure 2

2-Minute SimDist analysis is possible using the EZ Flash[®] system with Restek's 5m, 0.32mm ID, 0.1µm Thermo Orion-Rtx[®]-1 column.



Contact Thermo Orion for this column: 5m, 0.32mm ID, 0.1µm Thermo-Orion-Rtx[®]-1
Inj.: split at 35:1; Oven temp.: 45°C to 325°C @ 105°C/min. (hold 20 sec.); Inj. temp.: 310°C;
FID temp.: 350°C; Carrier gas: helium; Linear velocity: 66cm/sec;
Standard: ASTM D-2887 reference gas oil#1

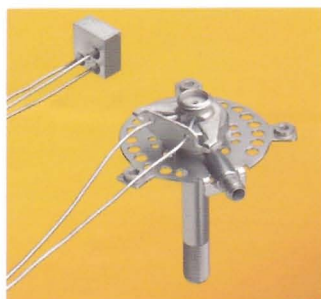
for **moreinfo**



For more information on the EZ Flash[®] system, contact Thermo Orion at 1-888-EZFLASH or www.ezflash.com

Peak Performers

by Brad Righthour, Instrument Innovations Team Manager



Split/splitless injection port for Agilent 6890/6850 GCs

- ✓ Constructed of high-quality stainless steel.
- ✓ Meets or exceeds original equipment specifications.

Replacement split/splitless weldment for 6890/6850 GCs with EPC: cat.# 22674 (ea.)

Replacement split/splitless weldment for 6890/6850 GCs with manual flow: cat.# 20265 (ea.)

Replacement shell weldment for 6890/6850 GCs: cat.# 22673 (ea.)



FID collector assembly for Agilent 6890/6850 GCs

- ✓ Constructed of high-quality stainless steel.
 - ✓ Meets or exceeds original equipment specifications.
- (Similar to Agilent part# G1531-60690)
cat.# 21699 (kit)



Injector & detector maintenance/start-up kits for Agilent GCs

- ✓ No more trying to find the parts you need.
- ✓ All parts meet or exceed original equipment specifications.
- ✓ Easy to re-order parts list so you never run out of supplies.

Injector maintenance/start-up kit for 5890/6890 GCs: cat.# 21069 (kit)

Detector maintenance/start-up kit for 5890 GCs: cat.# 21070 (kit)

Detector maintenance/start-up kit for 6890 GCs: cat.# 21071 (kit)

New inlet liners for APEX ProSep™ 800 & ProSep™ 800 plus

- ✓ 100% polymeric deactivation increases inertness to prevent sample adsorption.
- ✓ Meet or exceed original equipment specifications.

APEX Liner (1.0mm ID)

(similar to APEX# L-00110)

Uses: injections <5µL

ID*/OD & Length (mm): 1.0 ID x 6.0 OD x 240
cat.# 21073 (ea.)

APEX Liner (2.0mm ID)

(similar to APEX# L-00210)

Uses: injections <25µL

ID*/OD & Length (mm): 2.0 ID x 6.0 OD x 240
cat.# 21074 (ea.)

APEX Liner (4.0mm ID)

(similar to APEX# L-00410)

Uses: injections <125µL

ID*/OD & Length (mm): 4.0 ID x 6.0 OD x 240
cat.# 21075 (ea.)

New inlet liners for Varian 1177 injector

- ✓ 100% polymeric deactivation increases inertness to prevent sample adsorption.
- ✓ Meet or exceed original equipment specifications.

4mm Split w/glass frit

Uses: dirty samples, non-active compounds

ID*/OD & Length (mm): 4.0 ID x 6.5 OD x 78.5

Similar to Varian Part # 39-26119-36

cat.# 21045 (ea.) cat.# 21046 (5-pk.)

2mm Splitless w/wool

Uses: trace samples <2µL

ID*/OD & Length (mm): 2.0 ID x 6.5 OD x 78.5

Similar to Varian Part # 39-26119-38

cat.# 21077 (5-pk.)

4mm Split w/wool

Uses: trace samples >2µL

ID*/OD & Length (mm): 4.0 ID x 6.5 OD x 78.5

Similar to Varian Part # 39-26119-37

cat.# 21079 (5-pk.)

*Nominal ID at syringe needle expulsion point.

Plus 1™ Restek's Customer Commitment

You will be seeing the Plus 1™ symbol throughout our catalog, on our packaging, on our website, and even on our 15th anniversary coffee mug. Plus 1™ Service means we will surpass your expectations every time you contact us! You'll get Plus 1™ service when you ask our experienced Technical Service Team to help solve a difficult analytical problem. Our helpful, efficient Customer Service Team provides Plus 1™ service even when you place a late-day order.

Plus 1™ customer service is what has made Restek unique. If special attention was paid to your requests or if our employees went out of their way to fulfill your needs, we would like to recognize and reward them. Contact us with your Restek success stories today!



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Please direct your comments on this publication to Kristin Dick, Editor, at kristind@restekcorp.com or call Restek, ext. 2313.

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