

mize residual silanols and metal ions on the packing particles, which could interact with the analytes and cause tailing and unwanted (and sometimes unpredictable) retention.

The reagent solution we use in the mobile phase, Ultra Quat Reagent Solution (cat.# 32441), alters the chemical nature of the analytes as perceived by the column and mobile phase. It reduces the ability of water to solvate the analytes and hydrogen bond with them, forcing the charged complexes into the stationary phase and improving retention.

Unlike ion pairing techniques, our new approach requires only water, Ultra Quat Reagent Solution, and acetonitrile (which cannot form hydrogen bonds) to accomplish the separation. For highest sensitivity, we monitor for paraquat at 257nm and for diquat at 308nm. Using the new column, mobile phase, and conditions, the detection limit for either herbicide is 6ppb in the final sample extract—a detectable amount of 0.12 nanograms on column. Data are summarized in Table 1. Using the solid phase extraction procedure in Table 2, which concentrates samples 200-

fold (1L to 5mL), the detection limit is 0.03ppb—a significant improvement over current methodology. Analyte concentrations can be increased by modifying the solid phase extraction procedure or by increasing the injection volume, to improve quantification and detection limits.

Figure 2 overlays chromatograms of paraquat and diquat reference standards at a range of concentrations (20 μ g/mL–100 μ g/mL); resolution, retention times, and peak symmetry are highly consistent. Concentrations up to 100 μ g/mL are consistent with linear detector responses.

Note that glassware used to prepare and analyze samples and reference materials for this analysis must be deactivated (e.g., with dimethyldichlorosilane—DMDCS, cat.# 31840). EPA Method 549.2 requires retesting of all samples if the response for the reference standards changes by more than 20% over the time of the analysis. We found all reference standards showed degradation after only 1 hour in untreated glassware, with the lowest concentrations being the most affected. 30% losses in

response were not uncommon; a diquat reference standard of 6ppb in water became undetectable.

When you perform the challenging paraquat/diquat analysis, our new Ultra Quat column, Ultra Quat Reagent Solution and Paraquat/Diquat Calibration Mix, and extraction procedure will give you the edge you need to obtain the most accurate and consistent information.

In Summary

Highly polar paraquat and diquat can't be separated on a reversed phase HPLC column without adding ion pair modifier to the mobile phase, but the ion pair reagent in current methodology does not provide optimum resolution and does not permit detection below 0.7 μ g/mL. We have developed a column and a mobile phase modifier for rapid, complete resolution of paraquat and diquat, with detection to concentrations as low as 0.5 μ g/mL—an improvement of 30%.

Table 1

Approximate detection/quantification limits for paraquat and diquat, using an Ultra Quat column.

On column limit of detection (LOD): 0.12ng
On column limit of quantification (LOQ): 1.2ng

Sample Volume (mL)	Injection Volume (μ L)	Limit of Detection (ppb)	Limit of Quantification (ppb)
1	20	6	20
100	20	0.06	0.2
250	20	0.024	0.08
1000	20	0.006	0.02
1	100	1.2	4
100	100	0.012	0.04
250	100	0.0048	0.016
1000	100	0.0012	0.004
1	200	0.6	2
100	200	0.006	0.02
250	200	0.0024	0.008
1000	200	0.0006	0.002

Table 2

Solid phase extraction of diquat and paraquat from aqueous samples.

Sample Extraction

SPE Tubes:	Restek WCX, weak cation exchanger, 3mL/500mg, cat.# 26062.
Samples:	1 liter deionized water containing 50 μ g each of diquat and paraquat. Samples spiked with 20 μ L 549.2 Calibration Mix, cat.# 32437, diluted with HPLC grade water.
Conditioning:	3mL acetonitrile, then 3mL deionized water, applied sequentially. Do not allow adsorbent bed to dry before applying sample.
Extraction:	Pass 1 liter water samples through SPE tubes at a rate of 5-10mL/min. Arrange 5mL collection vessels under extraction tubes. Place 1mL acidic elution solution* in each tube, draw into bed, allow to stand for up to 1 min. Pass solution at a slow (drop-wise) rate through SPE tubes into collection vessels. Repeat with 2 x 2mL acidic elution solution. Correct final volume in collection vessels to 5mL with acidic elution solution.
Analysis:	Neutralize eluates with approximately 20 μ L concentrated ammonium hydroxide, then analyze by HPLC. Adjust amount of ammonium hydroxide used to assure each sample is neutral (test with pH indicating paper).

*1mL 85% H₃PO₄ diluted to 1 liter with deionized HPLC grade water (0.1%).

Results

Analyte	Recovery (%)	RSD (%)	
diquat	99.0	0.89 (n=5)	Extracted samples stored and analyzed in Silcote™ CL7 deactivated autosampler vials (cat.# 24671). Polypropylene vials and inserts (e.g., cat.# 24651) also may be used.
paraquat	96.3	1.59 (n=5)	

Ultra Quat HPLC Column

Physical Characteristics:

particle size: 5 μ m, spherical
pH range: 2.5 to 7.5
temperature limit: 80°C



5 μ m Column, 4.6mm ID	cat.#
150mm	9181565

Ultra Quat Guard Cartridges

Length	4.0mm ID	cat.#
10mm		918150210
20mm		918150220

Ultra Quat Reagent Solution

Each	10-pk.
In water, 20mL/ampul	
32441	32541

Paraquat & Diquat Calibration Mix

diquat dibromide	paraquat dichloride
Each	
1,000 μ g/mL each in water, 1mL/ampul	
32437	
w/data pack	
32437-500	

Dimethyldichlorosilane (DMDCS)

Each	5-pk.
Neat, 20mL/ampul	
31840	31840-510

WCX Solid Phase Extraction Tubes



3mL/500mg, 50-pk., cat.# 26062,

GC/MS Analysis of Phthalate and Adipate Esters in Drinking Water

Using New Restek Reference Mixes and a Low-Bleed Column

by Katia May, Ph.D., Senior R&D Chemist, and Christopher English, Environmental Innovations Chemist

- New calibration and quality control check mixes save preparation time and effort.
- Stable baseline with Rtx®-5Sil MS column—no interference with sensitive detection.
- Rapid analysis, excellent resolution.

Phthalate esters are of considerable interest because their extensive use in consumer products, mainly as plasticizers, leads to widespread human exposure and potential for environmental contamination. In the United States, the Environmental Protection Agency (EPA) established strict drinking water standards for two of these semivolatile compounds, bis(2-ethylhexyl)phthalate and bis(2-ethylhexyl)adipate, as potential carcinogenic agents. Because even trace amounts of these esters can have a harmful effect on drinking water quality, the goal is to extract the compounds efficiently and identify them accurately. EPA Method 506 offers a procedure for extracting, identifying, and quantifying seven phthalate and adipate esters in drinking water, using liquid/liquid extraction (methylene chloride / hexane) or liquid/solid extraction (octadecyl (C18) disk, e.g., Restek cat.# 24004), extract concentration to 1mL, and analysis by gas chromatography/mass spectrometry.

We have developed two new reference materials for analyses of the phthalate and adipate esters targeted by Method 506. We prepare 506 Calibration Mix in isooctane at 1000µg/mL, per method recommendation, and 506 Laboratory Performance Check Mix in purge-and-trap grade methanol at x10⁵ the method detection limit (MDL) for each analyte.

Rtx®-5Sil MS Column (fused silica)

(Selectivity equivalent to Crossbond® 5% diphenyl / 95% dimethyl polysiloxane) (temp. limits -60°C to 330°C)

30-Meter, 0.25mm ID, 0.25µm df
cat.# 12723



Resprep™-C18 & Resprep™-C8 SPE Disks

- 47mm glass fiber disks embedded with C18 or C8 bonded silica.
- Extract semivolatile organic compounds.
- Deep-pore design reduces clogging and allows faster flow rates.
- Meet requirements for US EPA Methods 525.1, 506, 550.1, 549.1.
- Lower cost than Teflon® disks.

Description	qty.	cat.#
Resprep™-C18		
47mm SPE Disks	20-pk.	24004
Resprep™-C8		
47mm SPE Disks	24-pk.	24048

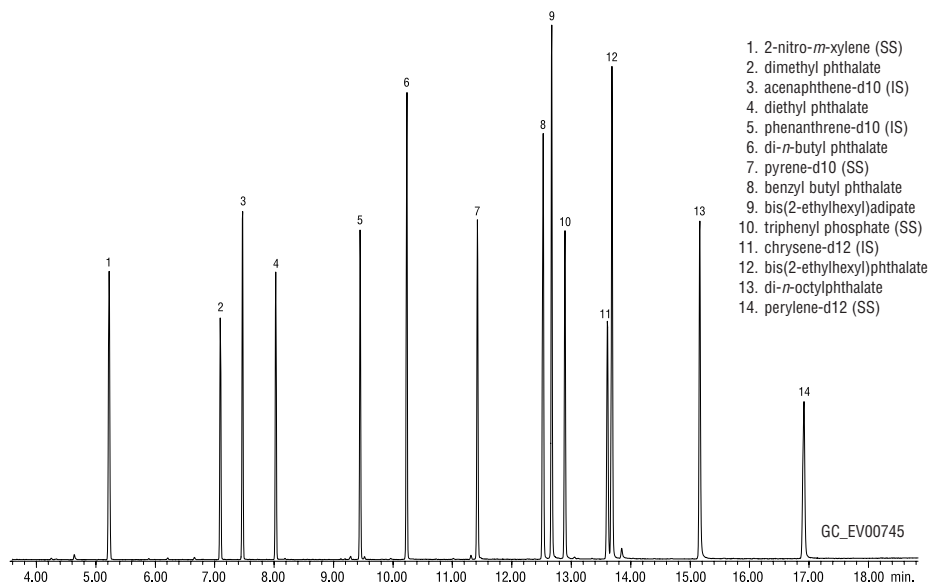
In our quest for superior chromatography and improved detection limits for this and other analyses, we have developed a series of low bleed polymeric stationary phases, using Crossbond® bonding technology. By providing stable baselines at higher temperatures, these phases allow higher signal-to-noise ratios, and thus greater sensitivity.

EPA Method 506 suggests low levels of phthalate and adipate esters be evaluated using a

photoionization detector. The method allows other approaches for detection, however, if equivalent performance can be demonstrated. Figure 1 shows a GC/MS analysis of the phthalates and adipates, using an Rtx®-5Sil MS column. Column bleed is low, even at the 310°C oven temperature needed to elute the phthalate esters with the highest boiling points. At this temperature, column bleed from an unstable column could have a significant effect on detection limits. The 80°C starting temperature and 18°C/min. temperature program ensure a fast analysis, without inhibiting resolution. US EPA 525.2 internal standards and surrogates were used since the method does not list specific monitoring compounds.

In analyses for phthalate and adipate esters, a low-bleed Rtx®-5Sil MS column can extend detection limits and help ensure you of reliable data from your samples.

Figure 1 Rapid analysis of phthalates, with excellent resolution, using an Rtx®-5Sil MS column.



506 Laboratory Performance Check Mix

benzyl butyl phthalate	250µg/mL	di- <i>n</i> -octyl phthalate	650
bis(2-ethylhexyl)adipate	1200	diethyl phthalate	100
bis(2-ethylhexyl)phthalate	250	dimethyl phthalate	100
di- <i>n</i> -butyl phthalate	100		

Each	5-pk.	10-pk.
In P&T methanol, 1mL/ampul		
31844	31844-510	—
w/data pack		
31844-500	31844-520	31944

506 Calibration Mix

benzyl butyl phthalate	di- <i>n</i> -octyl phthalate
bis(2-ethylhexyl)adipate	diethyl phthalate
bis(2-ethylhexyl)phthalate	dimethyl phthalate
di- <i>n</i> -butyl phthalate	

Each	5-pk.	10-pk.
1,000µg/mL each in isooctane, 1mL/ampul		
31845	31845-510	—
w/data pack		
31845-500	31845-520	31945

Column: Rtx®-5Sil MS, 30m, 0.25mm ID, 0.25µm (cat.# 12723)
 Sample: 506 Calibration Mix, 1000µg/mL each analyte (cat.# 31845)
 Method 525.2 Internal Standard Mix (cat.# 31825)
 Method 525.2 Surrogate Standard Mix (cat.# 31826)
 Inj.: 1.0µL, 20ppm each analyte using a 4mm splitless single gooseneck inlet liner (cat.# 20799) splitless hold time 0.40 min., 0.45 min. pressure pulse @ 50psi
 GC: Agilent 6890
 Inj. temp.: 270°C
 Carrier gas: helium, constant flow
 Flow rate: 1.0mL/min.
 Oven temp.: 80°C (hold 0.5 min.) to 260°C @ 18°C/min., to 310°C @ 6°C/min. (hold 1 min.)
 Det.: Agilent 5973 GC/MS
 Transfer line temp.: 280°C
 Scan range: 35–550 amu
 Solvent delay: 3 min.
 Tune: DFTPP

Fast Dual-Column Analysis of Pesticides or PAHs

new!
★

Using an Rtx®-440 Capillary GC Column

By Greg France, Innovations Chemist, and Gary Stidsen, Innovations Team Manager

- Analyze 20 organochlorine pesticides in less than 9 minutes.
- Analyze 16 PAHs in 22 minutes.
- New low-bleed, high-resolution column is ideal for dual-column analyses.

Assessments for organochlorine pesticides or polynuclear aromatic hydrocarbons (PAHs) are critical, frequently performed GC analyses—and they often are among the most challenging. Issues that can arise include analyte breakdown and poor linearity, and calibration times can be long. In addition to the problems inherent to the analysis, analysts must be concerned

with column reactivity and bleed, which affect sensitivity and reproducibility. In analyses of PAHs, there are critical pairs to resolve and, because the samples often include interfering hydrocarbons, a confirmation column typically is required. Compounding these problems is constant pressure to process more samples in less time.

Figure 1 Separate 20 organochlorine pesticides in 9 minutes, using an Rtx®-440 column.

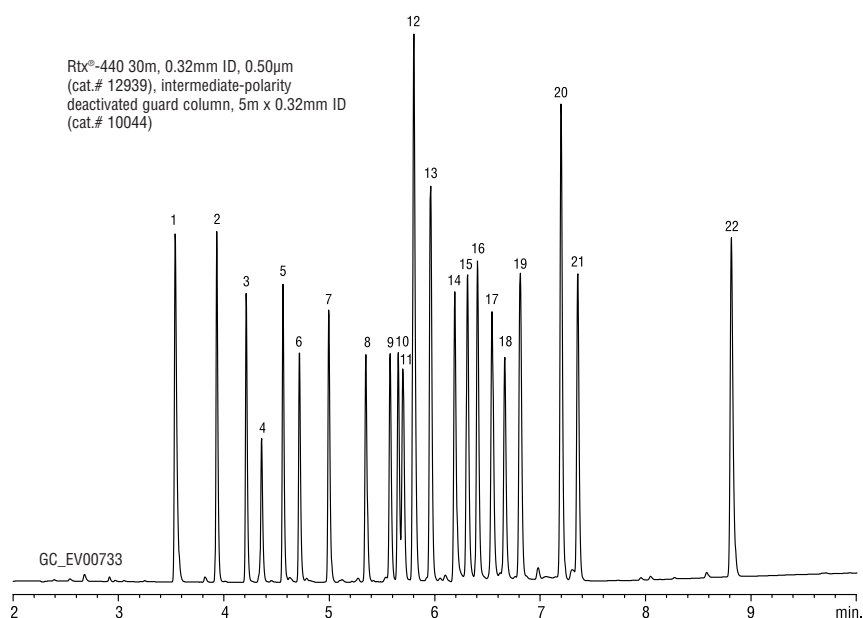
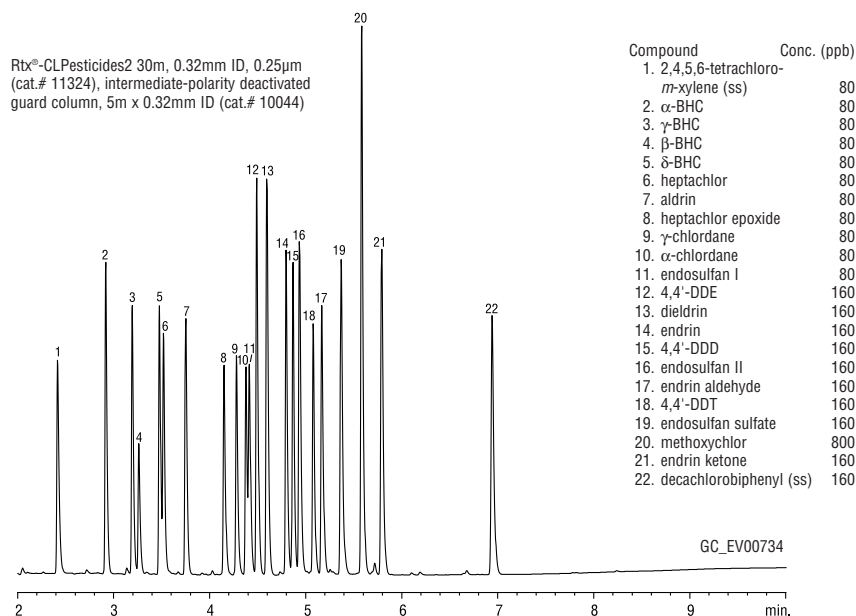


Figure 2 An Rtx®-CLPesticides2 column complements the Rtx®-440 column in dual-column analysis of organochlorine pesticides.



With the new Rtx®-440 column, Restek makes available an excellent choice for both of these demanding applications.

Organochlorine Pesticides: Sub-10-Minute Analyses

Figure 1 shows a separation of 20 commonly analyzed organochlorine pesticides, obtained in less than 10 minutes by using an Rtx®-440 column. Only α-chlordane and endosulfan I (peaks 10 & 11) are not separated to the baseline. The column's excellent thermal stability is indicated by a virtually flat baseline between the initial temperature and the maximum temperature of the program, 330°C. In a dual-column approach to this application, an Rtx®-440 column can be paired with an Rtx®-CLPesticides2 column. The latter column will provide an equally fast separation (Figure 2) and near-equivalent resolution, with a reverse in elution order for endrin aldehyde and 4,4'-DDT (peaks 17 & 18). By connecting the two columns to a "Y" connector and making the sample injection onto a 5-meter intermediate-polarity deactivated guard column, the two analyses can be conducted simultaneously.

Polynuclear Aromatic Hydrocarbons: Baseline Resolution of Critical Pairs

In Figure 3, 16 commonly encountered PAHs have eluted from an Rtx®-440 column in less than 18 minutes. Two critical pairs, phenanthrene/anthracene (peaks 5 and 6) and benzo(a)anthracene/chrysene (peaks 9 and 10), are resolved to baseline, and benzo(b)fluoranthene and benzo(k)fluoranthene (peaks 11 and 12) and indeno(1,2,3-cd)pyrene and dibenzo(a,h)anthracene (peaks 14 and 15) are almost completely separated. Also notice the excellent thermal stability—baseline rise is negligible even at 320°C. Similar results can be obtained by using an Rtx®-5Sil MS column or an Rtx®-CLPesticides2 column and constant flow, as shown in the Applications section of our general catalog. An Rtx®-440 column can be paired with either of these other columns, for a rapid, dual-column/FID analysis of commonly encountered PAHs.

Conditions for Figures 1 and 2

Sample: Organochlorine Pesticide Mix AB #2 (cat.# 32292), 2,4,5,6-tetrachloro-*m*-xylene (ss) (cat.# 32027), decachlorobiphenyl (ss) (cat.# 32029), diluted in hexane, on-column amounts listed on figure

Inj.: 1.0µL splitless (hold 0.75 min.), 4mm Drilled Uniliner® inlet liner (cat.# 21055)

Inj. temp.: 225°C

Carrier gas: hydrogen, constant pressure

Linear velocity: 73cm/sec. (Rtx®-440) or 77cm/sec. @ 140°C (Rtx®-CLPesticides2)

Oven temp.: 140°C (hold 0.5 min.) to 268°C @ 30°C/min., to 290°C @ 11°C/min., to 330°C @ 25°C/min. (hold 5 min.)

Det.: ECD @ 320°C

Conclusion

The new Rtx®-440 column is an excellent addition to the selection of innovative columns available from Restek. The column exhibits high thermal stability and, for organochlorine pesticides, a selectivity alternative to the Rtx®-CLPesticides2 column. An Rtx®-440 column can be paired with an Rtx®-CLPesticides2 column to ensure sub-10-minute analysis times for organochlorine pesticides, or can be used as a confirmation column, with an Rtx®-5Sil MS or an Rtx®-CLPesticides2 column, for GC/FID analysis of PAHs.

Organochlorine Pesticide Mix AB #2

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

Each	5-pk.	10-pk.
In hexane:toluene (1:1), 1mL/ampul		
32292	32292-510	—
	w/data pack	
32292-500	32292-520	32392

2,4,5,6-Tetrachloro-m-xylene

Each	5-pk.	10-pk.
200µg/mL in acetone, 1mL/ampul		
32027	32027-510	—
	w/data pack	
32027-500	32027-520	32127
200µg/mL in acetone, 5mL/ampul		
32028	32028-510	—
	w/data pack	
32028-500	32028-520	32128

Decachlorobiphenyl (BZ #209)

Each	5-pk.	10-pk.
10µg/mL in isooctane, 1L/ampul		
32289	32289-510	—
	w/data pack	
32289-500	32289-520	32389
200µg/mL in acetone, 1mL/ampul		
32029	32029-510	—
	w/data pack	
32029-500	32029-520	32129
200µg/mL in acetone, 5mL/ampul		
32030	32030-510	—
	w/data pack	
32030-500	32030-520	32130

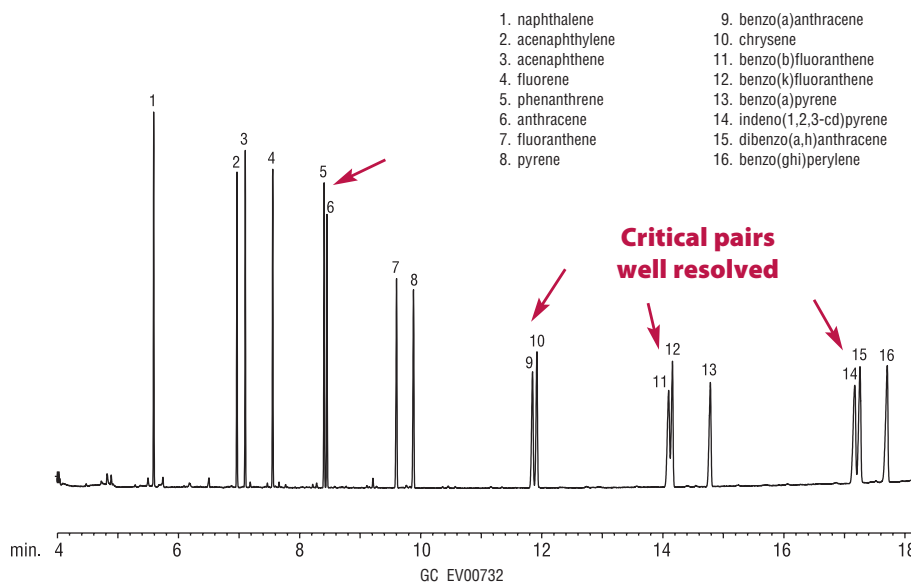
Rtx®-440 Columns (fused silica)

ID	df (µm)	temp. limits	30-Meter
0.25mm	0.25	20°C to 320/340°C	12923
	0.50	20°C to 320/340°C	12938
0.32mm	0.25	20°C to 320/340°C	12924
	0.50	20°C to 320/340°C	12939
0.53mm	0.50	20°C to 320/340°C	12940
	1.00	20°C to 320/340°C	12955

Rtx®-CLPesticides2 Columns (fused silica)

ID	df (µm)	temp. limits	10-Meter	15-Meter	20-Meter	30-Meter	60-Meter
0.10mm	0.10	-60 to 310/330°C	43301		43302		
0.18mm	0.14	-60 to 310/330°C	42301		42302		
0.25mm	0.20	-60 to 320/340°C		11320		11323	11326
0.32mm	0.25	-60 to 320/340°C		11321		11324	
0.53mm	0.42	-60 to 300/320°C		11337		11340	

Figure 3 Analyze 16 PAHs in 22 minutes, and resolve critical pairs, with an Rtx®-440 column.



Column: Rtx®-440 30m, 0.25mm ID, 0.25µm (cat.# 12923)
 Sample: 610 PAH Mix (cat.# 31011) diluted to 20ppm each compound in methylene chloride
 Inj.: 1.0µL splitless (hold 0.4 min.), 4mm splitless liner (cat.# 20772)
 Inj. temp.: 320°C
 Carrier gas: hydrogen, constant flow
 Flow: 3.6mL/min.
 Oven temp.: 40°C (hold 2 min.) to 240°C @ 30°C/min., to 320°C @ 8°C/min. (hold 5 min.)
 Det.: FID @ 320°C

Searching for a chromatogram?
www.restek.com

SV Calibration Mix #5 / 610 PAH Mix

Each	5-pk.	10-pk.
acenaphthene	chrysene	
acenaphthylene	dibenzo(a,h)anthracene	
anthracene	fluoranthene	
benzo(a)anthracene	fluorene	
benzo(a)pyrene	indeno(1,2,3-cd)pyrene	
benzo(b)fluoranthene	naphthalene	
benzo(k)fluoranthene	phenanthrene	
benzo(ghi)perylene	pyrene	

Each	5-pk.	10-pk.
2,000µg/mL each in methylene chloride, 1mL/ampul		
31011	31011-510	—
	w/data pack	
31011-500	31011-520	31111

SeCure™ "Y" Connectors

- Use standard "Y" Press-Tight® connectors and 1/16" graphite ferrules.
- Reliable seal integrity, will not unexpectedly disconnect during temperature-programmed analyses.
- Open design allows visual confirmation of the seal for added confidence in the connection.



Restek
 Innovation!

Kits include: SeCure™ "Y" connector body, 3 knurled nuts, 1 "Y" Universal Press-Tight® union, 3 ferrules

Description	Ferrules Fit Column ID (mm)	qty.	cat.#
SeCure™ "Y" Connector Kit	0.25/0.28	kit	20276
SeCure™ "Y" Connector Kit	0.28/0.32	kit	20277
SeCure™ "Y" Connector Kit	0.45/0.53	kit	20278
Knurled nut		3-pk.	20279

Rapid Analysis of Residual Solvents in Pharmaceuticals

Restek
Innovation!

Using Static Headspace Sampling and Stop-Flow GC

by Christopher English, Environmental Innovations Chemist, Rebecca Wittrig, Ph.D., HPLC Product Marketing Manager, and Frank Dorman, Ph.D., Director of Technical Development



Kit is easily attached to Agilent 6890 GC!

- Resolve 35 residual solvents in 18 minutes.
- Simplify inventory—use one pair of chromatography columns and one set of conditions for all ICH Class I and Class II solvents.
- Complete, easy to install system.

The International Conference on Harmonization (ICH) makes recommendations concerning amounts of residual solvents considered safe in pharmaceutical finished goods. The ICH has published guidelines and daily exposure limits for 61 solvents, classified in three groups, according to their toxicity. Class I solvents are known carcinogens or environmental hazards, to be avoided if at all possible. Class II solvents are less toxic, but their use should be limited. Class III solvents have low toxicity or no health-related exposure limit.¹ All pharmaceutical products must be analyzed for residual solvents, regardless of the matrix, and an enormous number of methods potentially can be required to address the total list of solvents. The complexity and high cost of compliance are major hurdles in drug manufacture.

In February 2004, Teledyne Tekmar developed a universal analytical method for extracting and determining 32 ICH Class II and Class III residual solvents, using static headspace sampling.² Simultaneously, Restek chemists were developing an approach for resolving the Class I and Class II solvents, using a new technology known as Stop-Flow GC, but lacked a sample preparation method suitable for achieving the detection limits required by the ICH.³ By using a Teledyne Tekmar 7000HT headspace autosampler unit in conjunction with Stop-Flow GC technology, it is possible to achieve resolution, sensitivity, and rapid sample turn-around times for the Class I and Class II residual solvents. In Stop-Flow GC the solvents are separated by passing the sample through a two-column ensemble consisting of a Stabilwax[®] column and an Rtx[®]-200 column coupled in series. Carrier gas flow through the

second (Rtx[®]-200) column is interrupted briefly (stop-flow pulses) to tune the separation at the outlet of the column ensemble.

In an analysis on two GC columns in series there are four possible outcomes for two sample components: 1) the two compounds are resolved at the column junction and remain resolved at the end of the ensemble; 2) the two compounds coelute at the junction, but are resolved on the second column; 3) the two compounds are resolved at the junction, but coelute at the end of the column ensemble; 4) the two compounds coelute at the column junction and at the end of the ensemble. For 1) and 2) no adjustment is necessary. For 4) other stationary phase combinations should be investigated to ensure separation on at least one of the two columns. For 3) Stop-Flow GC is appropriate. Carrier gas flow into the second column is interrupted briefly, immediately after one of the two compounds has crossed the junction, but while the other compound is still in the first column. The timing and duration of the stop-flow pulse are set to ensure that the two components remain separated when they reach the end of the column ensemble. The key to choosing a column ensemble for a specific application is to make separate analyses on each column, to ensure that no two compounds coelute on both stationary phases.

Figure 1 is the product of applying three stop-flow pulses at the junction point of the column ensemble, to pull apart three analytes: trichloroethene, acetonitrile, and chloroform. The other analytes are resolved by adjusting the carrier gas flow and temperature program, and

do not require pulses. The chromatogram includes all ICH Class I and Class II solvents, except ethylene glycol (which was not detected at 200ppm), at 200ppm each in 5mL of 1,3-dimethyl-2-imidazolidinone (DMI) solvent. By resolving closely eluting component pairs, Stop-Flow GC enables pharmaceutical laboratories to monitor all ICH Class I and Class II solvents with one pair of chromatography columns and a single set of conditions.

This analysis for 35 residual Class I and Class II solvents is rapid, sensitive, and reliable. If you are required to monitor solvents in pharmaceutical products, we welcome the opportunity to discuss Stop-Flow GC with you.

References

1. ICH Guidance for Industry, Q3A Impurities: Residual Solvents US Dept. of Health and Human Services, Food and Drug Administration, Center for Drug Evaluation and Research, Center for Biologics Evaluation and Research (CBER). International Conference on Harmonization, Dec. 1997.
2. Wallace, B. and J. Kancler. *One Universal Method for Residual Solvents in Pharmaceuticals Using a High Temperature Static Headspace Sample Introduction System* Application Note 7000-021b.doc, Teledyne Tekmar Instruments, Feb. 2004.
3. Wittrig, R.E.; F.L. Dorman, C.M. English, R.D. Sachs, *J.Chromatogr. A* 1027: 75-82 (2004).

Acknowledgement

Special thanks to Brian Wallace of Teledyne Tekmar for the use of the 7000HT headspace autosampler.

Stop-Flow GC for Agilent 6890 GCs

Description	qty.	cat.#
Stop-Flow System for use with Cool On-Column EPC (includes: Stop-Flow enclosure, top mounting plate, 1-line weldment, and interface cable)	kit	21168
Stop-Flow System for use with Split/Splitless EPC (includes: Stop-Flow enclosure, top mounting plate, 2-line weldment, and interface cable)	kit	21169

Stabilwax[®] Column

15-Meter, 0.25mm, ID 0.5 μ m df, cat.# 10635

Rtx[®]-200 Column

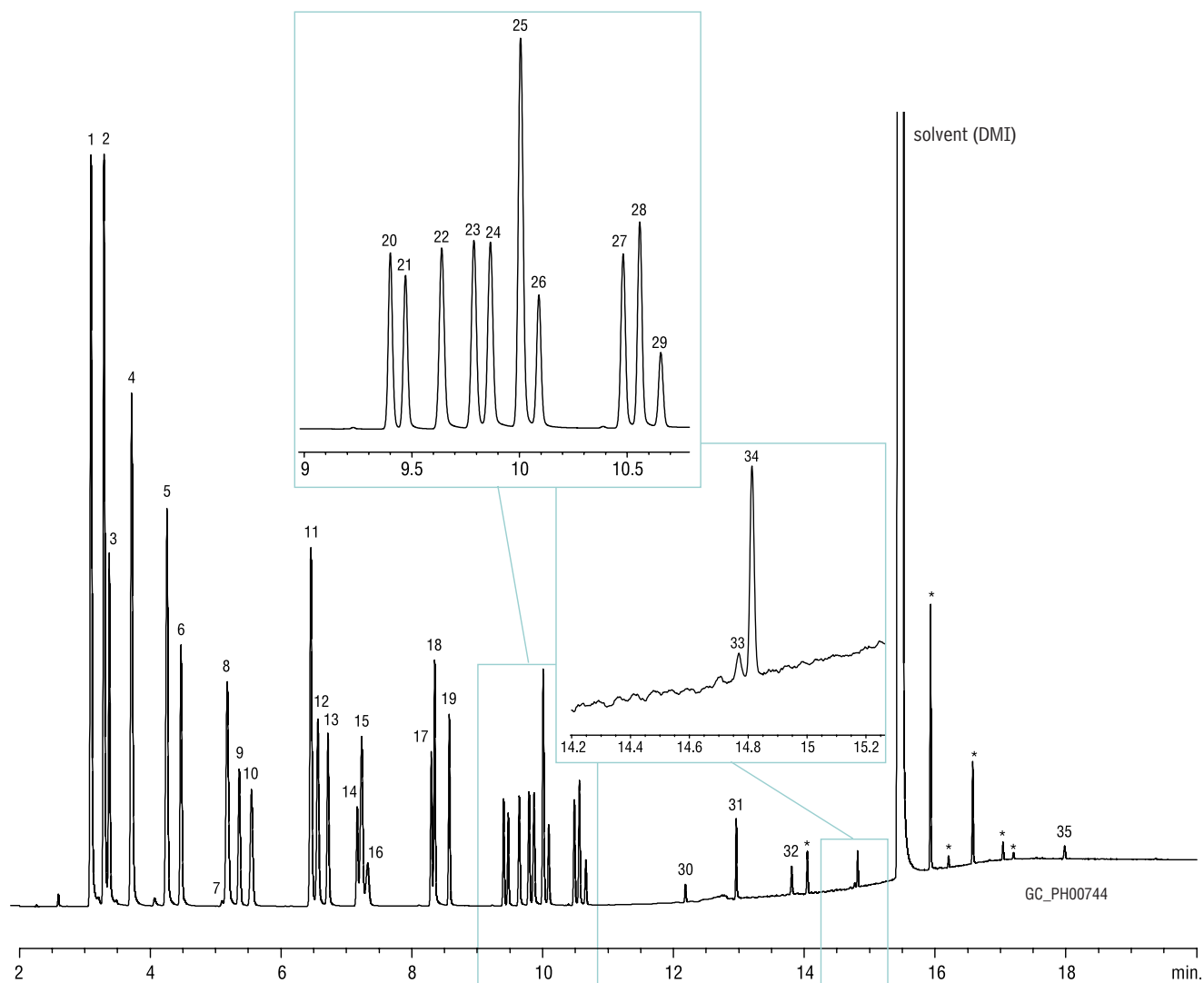
30-Meter, 0.25mm ID, 1.0 μ m df, cat.# 15053

Did you
know?



We offer many reference mixes of residual solvents for EP and USP methods. For descriptions, please refer to our chromatography supplies catalog, or visit our website.

Figure 1 Stop-Flow GC provides a rapid, sensitive analysis for ICH Class I and Class II residual solvents.



1. 2-methylpentane
2. hexane
3. 1,1-dichloroethene
4. methyl cyclopentane
5. methanol
6. *trans*-1,2-dichloroethene
7. carbon tetrachloride
8. methyl cyclohexane
9. methylene chloride

10. 1,1,1-trichloroethane
11. benzene
12. 1,2-dimethoxyethane
13. *cis*-1,2-dichloroethene
14. trichloroethene
15. acetonitrile
16. chloroform
17. 1,2-dichloroethane
18. toluene

19. 1,4-dioxane
20. nitromethane
21. 2-methoxyethanol
22. 2-hexanone (MBK)
23. *p*-xylene
24. *m*-xylene
25. pyridine
26. 2-ethoxyethanol
27. *o*-xylene

28. chlorobenzene
29. 1,1,2-trichloroethane
30. dimethyl formamide (DMF)
31. N,N-dimethylacetamide
32. 1,2,3,4-tetrahydronaphthalene
33. formamide
34. 1-methyl-2-pyrrolidinone
35. sulfolane
- * impurities in solvent

Headspace Conditions

Instrument: Teledyne Tekmar 7000HT high temperature static headspace unit

Platen temp.: 140°C

Sample equilibration: 5 min.

Mixing time: 10 min.

Mixing power: 2

Mixture stabilization: 1 min.

Pressure time: 0.2 min.

Pressure equilibration: 0.3 min.

Vial vol.: 22mL (high temperature vials)

Sample loop vol.: 1mL (standard size, Silcosteel® treated)

Loop/line temp.: 250°C

Loop fill time: 0.1 min.

Loop equilibration: 0.05 min.

Inj. time: 1.0 min.

Static vial press.: 3.5psi helium

Vial press.: 8psi helium

Variable inj. press. (VIPR): 5psi helium

Interface: plumbed through injection port, 1:20 split

GC Conditions

Column #1: Stabilwax®, 15m x 0.25mm x 0.5µm (cat. # 10635)

Column #2: Rtx®-200, 30m x 0.25mm x 1.0µm (cat. # 15053)

Sample: 200ppm each component in 1,3-dimethyl-2-imidazolidinone (DMI)

Instrument: Agilent 6890

Inj. port temp.: 250°C

Carrier gas: helium, constant flow

Flow rate: 1.9mL/min., 25.6psi @ 40°C

Oven temp.: 40°C (hold 2 min.) to 55°C @ 4°C/min., to 110°C @ 25°C/min. (hold 2 min.) to 250°C @ 25°C/min. (hold 5 min.)

Det.: FID #1 at column junction, FID #2 at sample outlet (equal settings)

Det. temp.: 250°C

Reaction gas: hydrogen, 40mL/min.

Air flow: 400mL/min.

Makeup: helium, 40mL/min.

Data collection rate: 100Hz

Stop-Flow Conditions

Instrument: Restek Stop-Flow System for Agilent 6890 GC with cool on-column injector (cat. #21168)

Inj. port connection: cool on-column injector

Pressure: 31.0psi, constant pressure

Pulses: valve opened 3.00 - 3.15 min., 4.65 - 5.02 min., 5.10 - 5.40 min.

Total analysis time: 20.55 min.

Improving Detailed Hydrocarbon Analysis

Using an Rtx®-1PONA Capillary GC Column

by Barry Burger, Petroleum Applications Chemist,
and Neil Mosesman, GC Columns Product Marketing Manager

- Column meets or exceeds all ASTM D-6730-01 and Canadian General Standards Board method requirements.
- 30% faster analysis (C13 retention = 97 minutes), using helium.
- Excellent responses and peak symmetry for polar oxygenates.
- Guaranteed column-to-column reproducibility for retention, efficiency, selectivity, peak skewness, resolution, low bleed.

Gasolines are complex mixtures of hundreds of compounds. Information about concentrations of the individual components is important for evaluating raw materials and controlling refinery processes. A high-resolution GC method for detailed hydrocarbon analysis (DHA) of gasolines is outlined in American Society of Testing and Materials (ASTM) Method D-6730-01—often referred to as the PONA (paraffins, olefins, naphthenes, aromatics) or PIANO (paraffins, isoparaffins, aromatics, naphthenes, olefins) analysis.* ASTM D-6730-01 is specific for the analysis of these hydrocarbon components, plus oxygenated additives such as methanol, ethanol, *tert*-butanol, methyl *tert*-butyl ether (MTBE), and *tert*-amyl methyl ether (TAME) in spark-ignition engine fuels.

To maximize resolution of these complex mixtures, the ASTM method recommends a 100-meter x 0.25mm ID capillary column coated with 0.5 μ m of 100% dimethyl polysiloxane stationary phase, and sets minimum resolution criteria for several critical pairs of closely eluting com-

pounds. To retain the aromatics, and accomplish the separations, a short tuning column, approximately 2-3 meters long, coated with 5% diphenyl/95% dimethyl polysiloxane polymer, is connected to the inlet of the 100-meter analytical column. Through a series of trial analyses, the length of the tuning column is adjusted to ensure the critical resolutions are achieved.

Analytical columns used for this application must exhibit high efficiency and exceptional inertness, especially for polar oxygenates in gasoline. Figure 1 illustrates a column efficiency of 613,596 total theoretical plates, measured on C5, and shows excellent peak symmetry for the oxygenated additives, including ethanol and *t*-butanol (*t*-butanol skewness = 1.25). We test every Rtx®-1PONA column for retention (*k*), efficiency (*n*), stationary phase selectivity (*RI*), and bleed, and guarantee reproducible column-to-column performance.

An Rtx®-1PONA column meets all ASTM D-6730-01 requirements for critical pair resolution, as

demonstrated by Figure 2. A 2.6-meter tuning column was used to achieve the highlighted resolutions, based on retention of the aromatics (e.g., resolution for 1-methylcyclopentene / benzene = 1.28).

In addition to qualifying for the ASTM D-6730-01 analysis, Rtx®-1PONA columns meet the similarly stringent requirements of Canadian General Standards Board (CGSB) methodology. For additional detailed hydrocarbon analysis chromatograms and more information about these high-performance columns, please request a free copy of Applications Note 59568, or review the applications note and chromatography on our website.

Rtx®-1 PONA Column (fused silica)

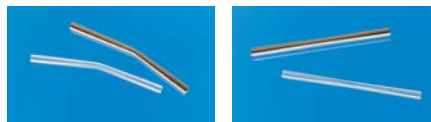
(Crossbond® 100% dimethyl polysiloxane phase optimized for hydrocarbon analysis) (temp. limits: -60 to 300/340°C) 100m, 0.25mm ID, 0.50 μ m df, cat.# 10195,

Rtx®-5 PONA Tuning Column

(Crossbond® 5% diphenyl/95% dimethyl polysiloxane phase) 5m, 0.25mm ID, 1.0 μ m df, cat.# 554206,

Press-Tight® Connectors

- Made from inert fused silica.
- Fit column ODs from 0.33–0.74mm (Restek 0.1mm–0.53mm ID).
- Angled connector reduces strain on connection.



Description	5-pk.
Universal Press-Tight® Connectors	20400
Siltek™-treated Universal Press-Tight® Connectors	20480
Universal Angled Press-Tight® Connectors	20446
Siltek™-treated Universal Angled Press-Tight® Connectors	20482

Vu2 Union™ Connector

A Vu2 Union™ connector combines the simplicity of a Press-Tight® union with the strength of a metal union. The columns cannot unexpectedly disconnect, even at temperatures as high as 400°C.

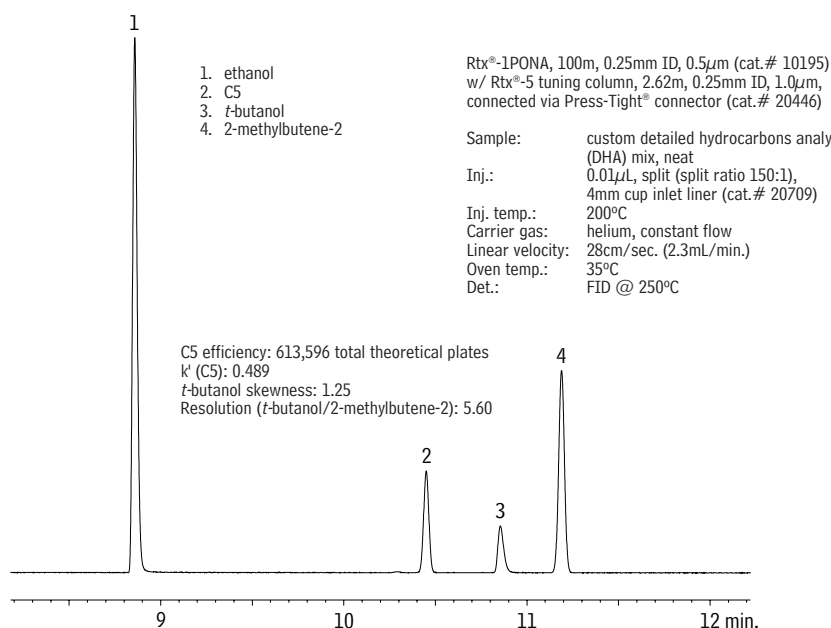


**Secure, reliable
column-to-column
connections!**

Kits include: Vu2 Union™ body, 2 knurled nuts, 2 Press-Tight® unions, and 4 ferrules

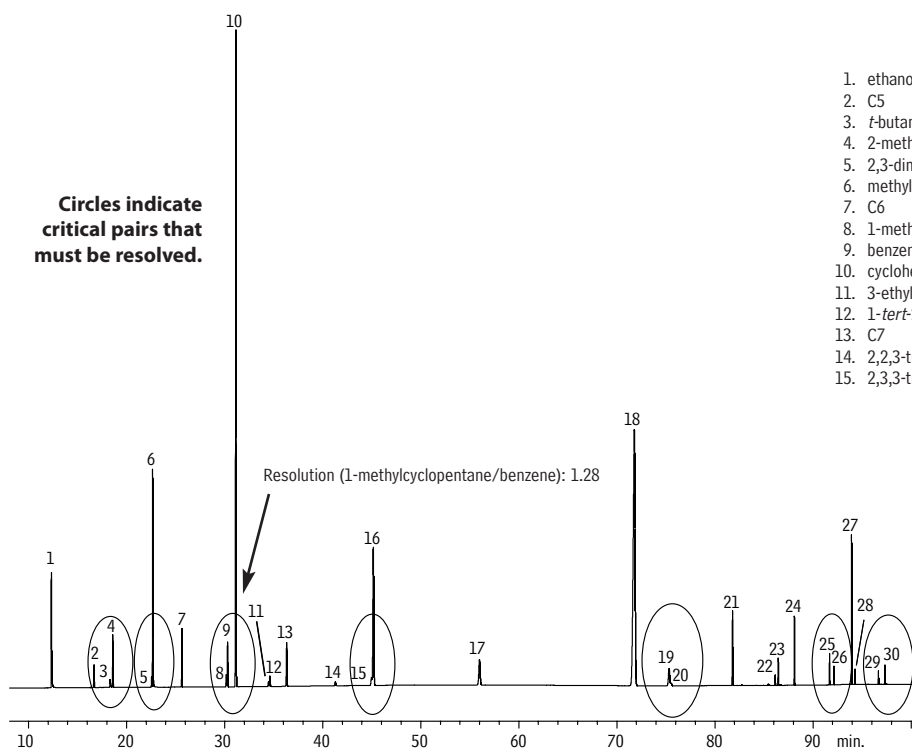
Connector Kit (Ferrules Fit Restek Column ID)	cat.#
Vu2 Union™ Connector Kit (0.15–0.25mm)	21105
Vu2 Union™ Connector Kit (0.28/0.32mm)	21106
Vu2 Union™ Connector Kit (0.45/0.50 & 0.53mm)	21107

Figure 1 Sharp, symmetric peak for ethanol (gasoline oxygenate), using an Rtx®-1PONA column.



*In alternate terminology: paraffins & isoparaffins = alkanes; naphthenes = cyclic alkanes; olefins = alkenes.

Figure 2 Critical pairs of gasoline components resolved per ASTM specifications, using an Rtx®-1PONA column.



- | | |
|--|--------------------------------|
| 1. ethanol | 16. toluene |
| 2. C5 | 17. C8 |
| 3. <i>t</i> -butanol | 18. ethylbenzene |
| 4. 2-methylbutene-2 | 19. <i>p</i> -xylene |
| 5. 2,3-dimethylbutane | 20. 2,3-dimethylheptane |
| 6. methyl <i>tert</i> -butyl ether (MTBE) | 21. C9 |
| 7. C6 | 22. 5-methylnonane |
| 8. 1-methylcyclopentane | 23. 1,2-methylethylbenzene |
| 9. benzene | 24. C10 |
| 10. cyclohexane | 25. C11 (undecane) |
| 11. 3-ethylpentane | 26. 1,2,3,5-tetramethylbenzene |
| 12. 1- <i>tert</i> -2-dimethylcyclopentane | 27. naphthalene |
| 13. C7 | 28. C12 (dodecane) |
| 14. 2,2,3-trimethylpentane | 29. 1-methylnaphthalene |
| 15. 2,3,3-trimethylpentane | 30. C13 (tridecane) |

Rtx®-1PONA, 100m, 0.25mm ID, 0.5µm (cat.# 10195)
 w/ Rtx®-5 tuning column, 2.62m, 0.25mm ID, 1.0µm,
 connected via Press-Tight® connector (cat.# 20446)
 Sample: custom detailed hydrocarbons analysis
 (DHA) mix, neat
 Inj.: 0.01µL, split (split ratio 150:1), 4mm cup
 inlet liner (cat.# 20709)
 Inj. temp.: 200°C
 Carrier gas: helium, constant flow
 Linear velocity: 28cm/sec. (2.3mL/min.)
 Oven temp.: 5°C (hold 15 min.) to 50°C @ 5°C/min.
 (hold 50 min.) to 200°C @ 8°C/min. (hold
 10 min.)
 Det.: FID @ 250°C

Restek on the Road

Autumn Seminars in Europe and the US

by Rick Parmely, Technical Training & Education Manager

Our GC and HPLC seminars are well known and well received across the USA and around the world. Our autumn seminars include those listed here. For details about these seminars, please contact the Restek representative listed. For the most current schedule of US seminars, please visit our website (www.restek.com/seminar). If you're interested in a seminar in your city, or country, please call and talk with us.



US Seminars

Comprehensive Capillary GC

Date	Location
Sept. 13	Boulder, CO
Sept. 13	Blue Ash, OH
Sept. 15	Columbus, OH
Sept. 16	Salt Lake City, UT
Sept. 17	Buffalo, NY
Oct. 14	RTP, NC
Oct. 21	Buena Park, CA

GC/MS

Nov. 1	Pleasanton, CA
Nov. 3	Seattle, WA

Comprehensive HPLC

Oct. 5	Rockville, MD
Oct. 6	Princeton, NJ
Oct. 8	Plymouth Meeting/ King of Prussia, PA
Oct. 18	La Jolla/San Diego, CA

HPLC Method Development (2 days)

Sept. 23/24	Indianapolis, IN
Oct. 21/22	Foster City, CA

International Seminars

GC/MS Seminar

Date	Location	Contact
September 14	Lisbon, Portugal	Dias De Sousa (phone: +351 21 953 31 20 or e-mail: DS@dias-de-sousa.pt)
September 15	Barcelona, Spain	Teknokroma (phone: +34 936 748 800 or e-mail: comercial@teknokroma.es)
September 17	Milan, Italy	CPS Analytics (phone: +39 (0)2 8954201 or e-mail: cps@cps.it)
September 20	Stockholm, Sweden	Coricon AB (phone: +46 18 34 90 34 or email: info@coricon.se)
September 21	Oslo, Norway	Instrument Teknikk (phone: (+47) 67 16 41 00 or e-mail: firmapost@instrument-teknikk.no)
October 11	Cork, Ireland	Restek Ireland (phone: +44-28-90-814576 or e-mail: restekteurope@aol.com)
October 12	Dublin, Ireland	Restek Ireland (phone: +44-28-90-814576 or e-mail: restekteurope@aol.com)
October 14	Cork, Ireland	Restek Ireland (phone: +44-28-90-814576 or e-mail: restekteurope@aol.com)
October 20	Moscow, Russia	Laverna Group (phone: +7-095-482-2001 or e-mail: rodchenkov@lab.comcor.ru)

Comprehensive GC Seminar

Date	Location	Contact
October 18	Budapest, Hungary	Lab Comp (phone: +36 1 347 6090 or e-mail: labcomp@lab-comp.hu)

Convenient Calibration, Faster GC/MS Analysis for Volatile Organics in Water

Using a New Restek Calibration Mix and an Rtx®-VMS Column

by Katia May, Ph.D., Senior R&D Chemist, and Jack Crissman, Ph.D., Analytical Reference Materials Marketing Manager

- 60-component MegaMix™ includes six target gases—eliminates mixing errors.
- 0.18mm Rtx®-VMS column offers fast cycles, excellent resolution of gases.
- Monitor drinking water - wastewater - hazardous waste.

Volatile organic analytes (VOAs) are a common source of environmental pollution, and are among the most difficult and expensive contaminants to monitor in water. Analysis and quantification of VOAs in drinking water are detailed in US EPA methods 502 and 524, and in many other methods worldwide.

Until now, Restek has offered two complex calibration mixes of volatile compounds for drinking water analysis: a mix containing 54 target compounds (502.2 MegaMix™, cat.# 30432), and one containing 73 compounds (Drinking Water VOA MegaMix™ 524.2 Rev. 4.2, cat.# 30601). The only target compounds in the EPA methods that we do not include in these mixes are the highly volatile gases, and, for Method 524.2, the reactive ketones. To prevent acetal formation, we offer the five ketones as a separate mix (cat.# 30602). We also offer the six gases separately, as 502.2 Calibration Mix #1 (cat.# 30042 or cat.# 30439). Analysts monitoring samples for the gases combine the gases mix with either the 54-component mix or the 73-component mix—this takes time and can introduce variation or mixing errors.

For the convenience of our customers, we have developed a new 60-component calibration mix (Volatiles MegaMix™ with Gases, cat.# 30603) that contains the 54 target compounds in 502.2 MegaMix™ mix, plus the six gases in 502.2 Calibration Mix #1, at 200 ppm each in purge and trap methanol. The new mix is suitable for Method 502, Method 524, or other methods followed in monitoring these compounds. The new mix brings a choice. The 60-component mix is very convenient to use, and eliminates both variation and the potential for errors (associated with measuring and mixing from multiple ampuls). An unopened ampul of this mix has a 24-month shelf life, but once the ampul is opened, the gases can begin to escape from the solution, and opened ampuls of the new mix should be replaced more frequently than ampuls of the 54-component mix. (This also is true of the 6-component gas mix.) Analysts choosing to work with the 54-component mix and the six gases mix must contend with the potential for mixing errors, but can see longer lifetimes from ampuls of the opened 54-component mix—if they are stored properly. We recommend storing

all VOAs reference mixes in a freezer, especially those containing the gases.

Chemists monitoring VOAs in water require fast and accurate analyses. A chromatography column with a cyanopropylphenyl/dimethyl polysiloxane stationary phase (e.g., a “624” column) or a diphenyl/dimethyl polysiloxane phase (e.g., a “502.2” column) can provide a fast analysis, but some compounds are likely to coelute, creating quantification problems. In contrast, Rtx®-VMS columns are designed specifically for

Rtx®-VMS Columns (fused silica)

(temp. limits -40°C to 240/260°C)

0.18mm ID, 1.00µm df

20-Meter, cat.# 49914,

40-Meter, cat.# 49915,



Volatiles MegaMix™ with Gases (60 Components)

benzene	2,2-dichloropropane
bromobenzene	1,1-dichloropropene
bromochloromethane	<i>trans</i> -1,3-dichloropropene
bromodichloromethane	<i>cis</i> -1,3-dichloropropylene
bromoform	ethylbenzene
bromomethane (methyl bromide)	hexachloro-1,3-butadiene
<i>n</i> -butylbenzene	(hexachlorobutadiene)
<i>sec</i> -butylbenzene	isopropylbenzene (cumene)
<i>tert</i> -butylbenzene	4-isopropyltoluene (<i>p</i> -cymene)
carbon tetrachloride	methylene chloride
chlorobenzene	(dichloromethane)
chloroethane (ethyl chloride)	naphthalene
chloroform	<i>n</i> -propylbenzene
chloromethane (methyl chloride)	styrene
2-chlorotoluene	toluene
4-chlorotoluene	1,1,1,2-tetrachloroethane
dibromochloromethane	1,1,2,2-tetrachloroethane
1,2-dibromo-3-chloropropane	tetrachloroethylene
1,2-dibromoethane (EDB)	1,2,4-trichlorobenzene
dibromomethane	1,2,3-trichlorobenzene
1,2-dichlorobenzene	1,1,1-trichloroethane
1,3-dichlorobenzene	1,1,2-trichloroethane
1,4-dichlorobenzene	trichloroethylene
dichlorodifluoromethane (CFC-12)	trichlorofluoromethane (CFC-11)
1,1-dichloroethane	1,2,3-trichloropropane
1,2-dichloroethane	1,3,5-trimethylbenzene
1,1-dichloroethylene	1,2,4-trimethylbenzene
<i>cis</i> -1,2-dichloroethylene	vinyl chloride
<i>trans</i> -1,2-dichloroethylene	<i>m</i> -xylene
1,2-dichloropropane	<i>o</i> -xylene
1,3-dichloropropane	<i>p</i> -xylene

Each	5-pk.	10-pk.
200µg/mL each in P&T methanol, 1mL/ampul		
30603	30603-510	—
w/data pack		
30603-500	30603-520	30703

analyses of volatiles by GC/MS, and circumvent such problems. Analysis on a 20m, 0.18mm ID, 1.0µm Rtx®-VMS column (cat.# 49914), using a 45°C initial oven temperature, will provide good resolution of the early eluting gases and ensure faster oven cycles. Under optimized analytical conditions and using a dual purge and trap system, as shown in Reference 1, Figure 47, the narrow bore column can reduce the analysis time to approximately 10 minutes, without sacrificing resolution.

If you are testing for volatiles in drinking water, wastewater, or hazardous waste, an Rtx®-VMS column and our new 60-component volatiles MegaMix™ with gases will help you meet the requirements for most analytical methods.

Reference

1. *Optimizing the Analysis of Volatile Organic Compounds*
Restek technical guide,
lit. cat.# 59887A,
free on request.
Also available on our website.



502.2 MegaMix™ (54 Components)

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

Each	5-pk.	10-pk.
200µg/mL each in P&T methanol, 1mL/ampul		
30432	30432-510	—
w/data pack		
30432-500	30432-520	30532
2,000µg/mL each in P&T methanol, 1mL/ampul		
30431	30431-510	—
w/data pack		
30431-500	30431-520	30531

502.2 Calibration Mix #1 (gases)

bromomethane	dichlorodifluoromethane
chloroethane	trichlorofluoromethane
chloromethane	vinyl chloride

Each	5-pk.	10-pk.
200µg/mL each in P&T methanol, 1mL/ampul		
30439	30439-510	—
w/data pack		
30439-500	30439-520	30539
2,000µg/mL each in P&T methanol, 1mL/ampul		
30042	30042-510	—
w/data pack		
30042-500	30042-520	30142

Additional Reference Materials for GC/MS Analysis of Volatile Organics in Water

Drinking Water VOA MegaMix™, 524.2 Rev. 4.1 (73 Components)

acrylonitrile	<i>trans</i> -1,3-dichloropropene
allyl chloride	diethyl ether (ethyl ether)
benzene	ethylbenzene
bromobenzene	ethyl methacrylate
bromochloromethane	hexachlorobutadiene
bromodichloromethane	hexachloroethane
bromoform	iodomethane (methyl iodide)
<i>n</i> -butylbenzene	isopropylbenzene (cumene)
<i>sec</i> -butylbenzene	4-isopropyltoluene (<i>p</i> -cymene)
<i>tert</i> -butylbenzene	methacrylonitrile
carbon disulfide	methyl acrylate
carbon tetrachloride	methylene chloride
chloroacetonitrile	(dichloromethane)
chlorobenzene	methyl methacrylate
1-chlorobutane	methyl <i>tert</i> -butyl ether
chlorodibromomethane	(MTBE)
(dibromochloromethane)	naphthalene
chloroform	nitrobenzene
2-chlorotoluene	2-nitropropane
4-chlorotoluene	pentachloroethane
1,2-dibromo-3-chloropropane	propionitrile (ethylcyanide)
(DBCP)	<i>n</i> -propylbenzene
1,2-dibromoethane	styrene
(ethylene dibromide)	1,1,1,2-tetrachloroethane
dibromomethane	1,1,2,2-tetrachloroethane
1,2-dichlorobenzene	tetrachloroethene
1,3-dichlorobenzene	tetrahydrofuran
1,4-dichlorobenzene	1,2,3-trichlorobenzene
<i>trans</i> -1,4-dichloro-2-butene	1,2,4-trichlorobenzene
1,1-dichloroethane	1,1,1-trichloroethane
1,2-dichloroethane	1,1,2-trichloroethane
1,1-dichloroethene	trichloroethene
<i>cis</i> -1,2-dichloroethane	1,2,3-trichloropropane
<i>trans</i> -1,2-dichloroethane	1,2,4-trimethylbenzene
1,2-dichloropropane	1,3,5-trimethylbenzene
1,3-dichloropropane	toluene
2,2-dichloropropane	<i>m</i> -xylene
1,1-dichloropropene	<i>o</i> -xylene
<i>cis</i> -1,3-dichloropropene	<i>p</i> -xylene

Each	5-pk.	10-pk.
2,000µg/mL each in P&T methanol, 1mL/ampul		
30601	30601-510	—
	w/data pack	
30601-500	30601-520	30701

Ketones Mix, 524.2 Rev. 4.1

acetone	2-hexanone
2-butanone (MEK)	4-methyl-2-pentanone (MIBK)
1,1-dichloro-2-propanone	

Each	5-pk.	10-pk.
5,000µg/mL each in 90% P&T methanol:10% water, 1mL/ampul		
30602	30602-510	—
	w/data pack	
30602-500	30602-520	30702

Antifoam Agent for Purge & Trap Samples

- Efficiently controls foam over a wide pH range.
- Effective at less than 0.1% of sample volume.
- Will not conflict with chromatography of target analytes.

Foam generated when purge gas passes through a sample can enter the analytical trap, and possibly into the GC column. Our non-hazardous silica-containing antifoam agent is of great help in volatile organics analyses.

Each	5-pk.
Neat, 1mL/ampul	
31822	31822-510

Food for Thought:

New Selections from the Restek Bookshelf

by Jack Crissman, Educational Products Marketing Manager

Many other titles available - see our website or catalog.

Chiral Separations by Liquid Chromatography and Related Technologies
Types, structures, and properties of chiral stationary phases and their preparation, application, and future. Technologies include sub- and super-critical fluid chromatography, capillary electrochromatography, and thin layer chromatography.
H.Y. Aboul-Enein and I. Ali, Marcel Dekker, Inc., 2003, 400pp., ISBN 0-8247-4014-9
cat.# 21449 (ea.) \$165

Current Practice of Gas Chromatography-Mass Spectrometry
Principles, instrumentation, and a wide range of applications. 40 contributors, worldwide; more than 1200 references, equations, tables, and graphics. A superb reference for operators, managers, and students.
W.M.A. Niessen, Marcel Dekker, Inc., 2001, 528pp., ISBN 0-8247-0473-8
cat.# 21489 (ea.) \$175

Handbook of GC/MS. Fundamentals and Applications
Sample preparation through data evaluation, including MS library searches and a substance index. Applications include environmental, food, pharmaceutical, and clinical analysis.
Hans-Joachim Hübschmann, Wiley-VCH, 2001, 608pp., ISBN 3-527-30170-4
cat.# 21490 (ea.) \$230

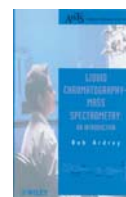
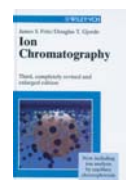
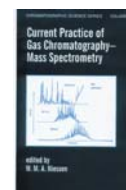
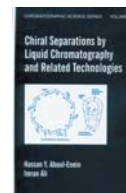
Handbook of Size Exclusion Chromatography and Related Techniques. 2nd Ed.
High-speed SEC, SEC of low molecular weight materials, and the extended family of techniques from two-dimensional liquid chromatography to high osmotic pressure chromatography.
C. Wu, Marcel Dekker, Inc., 2003, 716pp., ISBN 0-8247-4710-0
cat.# 21448 (ea.) \$195

The HPLC Solvent Guide. 2nd Ed.
Even experienced analysts tend to select from three familiar solvents. This guide describes many solvents suitable for HPLC separations.
P.C. Sadek, John Wiley & Sons, Inc., 2002, 664pp., ISBN 0-471-41138-8
cat.# 21979 (ea.) \$105

Ion Chromatography. 3rd Ed.
Materials, principles, and methods, including capillary electrophoresis and chemical speciation. Excellent introduction for novices or guide for experienced analysts.
J.S. Fritz and D.T. Gjerde, Wiley-VCH, 2000, 267pp., ISBN 3-527-29914-9
cat.# 21789 (ea.) \$135

Mass Spectrometry. Principles and Applications. 2nd Ed.
Principles, theories, and key applications, focused on recent developments. Expanded coverage of ESI and MALDI, and of biological and pharmaceutical applications. For students, and for researchers reviewing the latest techniques and developments.
E. de Hoffmann and V. Stroobant, John Wiley & Sons, Inc., 2001, 420pp., ISBN 0-471-48566-7
cat.# 21978 (ea.) \$65

Liquid Chromatography-Mass Spectrometry: An Introduction (softcover)
An indispensable reference for anyone wishing to use this increasingly important tandem technique.
R.E. Ardrey, John Wiley & Sons Ltd., 2003, 296pp., ISBN 0-471-49801-7
cat.# 20176 (ea.) \$50



New Reference Materials for Environmental Analyses

by Katia May, Ph.D., Senior R&D Chemist



US EPA Method 8270 Mixes in 100% Methylene Chloride

- Better peak shape for early eluting semivolatiles, compared to methylene chloride/benzene solvent.
- Methanol-free methylene chloride enhances stability.
- Calibration mix and matrix spike mix.

Most complex mixes for US EPA Method 8270 are prepared in combinations of methylene chloride/benzene solvent. The primary reason for using benzene, a high boiling solvent, is the belief that polyaromatic hydrocarbons are not readily soluble in methylene chloride. Benzene, however, can contribute to poor peak shape and low responses for early eluting compounds such as pyridine, N-nitrosomethylamine, N-nitrosomethylethylamine, 1,4-dioxane, and 2-picoline. Restek chemists have studied the solubility of polyaromatic hydrocarbons and determined that our unique method of preparing our Method 8270 MegaMix™ calibration mix (cat.# 31850) allows us to exclude benzene as a solvent.

US EPA Method 524 Surrogates Standard

- Separate mixes for surrogates and internal standard.
- Fortification solution combines surrogates and internal standard.
- Calibration mixes and all other quality control mixes also available.

US EPA Method 524 requires a surrogates standard, an internal standard, and the surrogates and internal standard combined in a fortification solution. We have offered the fortification solution (cat.# 30201) and the internal standard (cat.# 30030); we now offer the surrogates mix, described here. Use the new mix to monitor method performance by combining it with the sample before extraction. Together with Drinking Water VOA MegaMix™ 524.2 Rev. 4.2 calibration mix (cat.# 30601, see page 11), Ketones Mix 524.2 Rev. 4.2 (cat.# 30602, see

Methylene chloride alone is an effective solvent for these analytes; methanol-free methylene chloride enhances the stability of the product. Our new 8270 MegaMix™ in methylene chloride is a direct replacement for the older mix, but allows better chromatography.

Similarly, we offer new 8270 Matrix Spike Mix (cat.# 31851), 8270 Benzidines Mix (cat.# 31852), and 1,4-Dioxane (cat.# 31853) in 100% methylene chloride. Equivalents of these three mixes are available in methanol/methylene chloride/benzene (cat.# 31687) or methanol (cat.# 31688, cat.# 30287), respectively.

page 11), and additional calibration and quality control mixes listed in our catalog, the new mix completes our set of reference materials for Method 524.

524.2 Surrogate Standard

Each	5-pk.	10-pk.
1-bromo-4-fluorobenzene		
1,2-dichlorobenzene-d4		
2,000µg/mL each in P&T methanol, 1mL/ampul		
30607	30607-510	—
w/data pack		
30607-500	30607-520	30707

Drinking Water Odor Standard

- New reference mix of the two most common odor-causing compounds.
- Convenient concentration for purge and trap analysis: 100µg/mL in methanol.

Unpleasant odor in drinking water is associated with the growth and decay of microorganisms. Blue-green algae, green algae, diatoms, and flagellates are the four groups responsible for most common odor problems. Geosmin, produced by blue-green algae, has an earthy, musty smell. Actinomyces, mold-like bacteria also present in surface water, produce another common odor compound: 2-methylisoborneol.

The threshold value for these compounds is low (10ppt) and purge and trap analyses usually are used to quantify them. To help monitor the quality of drinking water, Restek's researchers have developed this convenient new reference mix.

Drinking Water Odor Standard

Each	5-pk.
(±)-geosmin	
2-methylisoborneol	
100µg/mL each in P&T methanol, 1mL/ampul	
30608	30608-510

new!

new!

new!

8270 MegaMix™ (76 components)

acenaphthene	2,4-dinitrophenol
acenaphthylene	2,4-dinitrotoluene
aniline	2,6-dinitrotoluene
anthracene	di- <i>n</i> -butyl phthalate
azobenzene ¹	di- <i>n</i> -octyl phthalate
benzo(a)anthracene	diphenylamine ²
benzo(a)pyrene	fluorene
benzo(b)fluoranthene	fluoranthene
benzo(g)helioperylene	hexachlorobenzene
benzo(k)fluoranthene	hexachlorobutadiene
benzyl alcohol	hexachlorocyclopentadiene
benzyl butyl phthalate	hexachloroethane
bis 2-ethylhexyl adipate	indeno(1,2,3- <i>cd</i>)pyrene
bis(2-chloroethoxy)methane	isophorone
bis(2-chloroethyl)ether	1-methylnaphthalene
bis(2-chloroisopropyl)ether	2-methylnaphthalene
bis(2-ethylhexyl)phthalate	2-methylphenol
4-bromophenyl phenyl ether	3-methylphenol*
carbazole	4-methylphenol*
4-chloroaniline	naphthalene
4-chloro-3-methylphenol	2-nitroaniline
2-chloronaphthalene	3-nitroaniline
2-chlorophenol	4-nitroaniline
4-chlorophenyl phenyl ether	nitrobenzene
chrysene	2-nitrophenol
dibenzo(a,h)anthracene	4-nitrophenol
dibenzofuran	N-nitrosodimethylamine
1,2-dichlorobenzene	N-nitroso-di- <i>n</i> -propylamine
1,3-dichlorobenzene	pentachlorophenol
1,4-dichlorobenzene	phenanthrene
2,4-dichlorophenol	phenol
diethyl phthalate	pyrene
dimethyl phthalate	pyridine
2,4-dimethylphenol	2,3,4,6-tetrachlorophenol
1,2-dinitrobenzene	2,3,5,6-tetrachlorophenol
1,3-dinitrobenzene	1,2,4-trichlorobenzene
1,4-dinitrobenzene	2,4,5-trichlorophenol
4,6-dinitro-2-methylphenol	2,4,6-trichlorophenol

Each	5-pk.	10-pk.
1,000µg/mL each in methylene chloride, 1mL/ampul*		
31850	31850-510	—
w/data pack		
31850-500	31850-520	31950

*3-methylphenol and 4-methylphenol at 500µg/mL.

¹1,2-diphenylhydrazine (8270-listed analyte) decomposes to azobenzene (mix component).

²N-nitrosodiphenylamine (8270-listed analyte) decomposes to diphenylamine (mix component).

8270 Matrix Spike Mix (76 components)

Same components as 8270 MegaMix™, but at lower concentration for spiking.

Each	5-pk.	10-pk.
200µg/mL each in methylene chloride, 5mL/ampul*		
31851	31851-510	—
w/data pack		
31851-500	31851-520	31951

*3-methylphenol and 4-methylphenol at 100µg/mL.

8270 Benzidines Mix

benzidine	3,3'-dimethylbenzidine
3,3'-dichlorobenzidine	

Each	5-pk.	10-pk.
2,000µg/mL in methylene chloride, 1mL/ampul		
31852	31852-510	—
w/data pack		
31852-500	31852-520	31952

1,4-Dioxane

Each	5-pk.	10-pk.
2,000µg/mL in methylene chloride, 1mL/ampul		
31853	31853-510	—
w/data pack		
31853-500	31853-520	31953

Searching for the Perfect Solution?

Let Restek create the perfect reference mixture—to your exact specifications. Contact the Technical Service Team, or your Restek representative, or visit us online at www.restek.com/solutions

Make GC/MS Column Changes in Minutes Without Venting

EZ No-Vent™ Connector Allows Fast Re-equilibration with No Loss of Data

by Brad Righthour, Instrument Innovations Manager,
and Christopher English, Environmental Innovations Chemist

- Cut downtime by 50% or more when changing columns.
- No special tools - no extra plumbing - low cost.
- Gold plated for inertness.
- 100µm ID transfer line keeps analytes focused.

Restek
Innovation!

A laser machined critical orifice in the EZ No-Vent™ connector minimizes the amount of air admitted into the MS source, eliminating the need for purge gas and for the lengthy vent and pump-down cycle otherwise required with a column change. This can save hours of downtime with each column change. The EZ No-Vent™ connector easily attaches to the MS source without special tools or extra plumbing: Figure 1 shows the connector installed and ready for use.

We tested the EZ No-Vent™ connector for dead volume, using highly volatile gases that are very susceptible to tailing. For reference, we operated the system as a purge-and-trap GC/MS system, with a split at the injection port and with the column inserted directly into the MS inter-

face. When we installed the EZ No-Vent™ connector in the MS interface, we anticipated that any dead volume in the connector would be revealed by tailing and broader peaks, relative to direct connection. Peak shape was excellent.

After establishing the connector had no effect on peak shape, we evaluated the ability of the MS to stabilize after a column change without venting. Again we used a purge-and-trap system, and halogenated volatiles as the analytes. We acquired Figure 2a, changed the column, and acquired Figure 2b 76 minutes later. Note the peak shapes and responses are unchanged. In the interval between the two analyses we verified MSD tuning, and the system passed bromofluorobenzene (BFB) criteria. Subsequent

detailed investigations confirmed the EZ No-Vent™ connector will allow several column changes in a single day, with no harm to the MS or loss of data.

If you're tired of waiting for your MS to stabilize after column changes, we highly recommend an EZ No-Vent™ connector. It will greatly reduce downtime and increase sample throughput.

Figure 1 MS transfer line installed in an EZ No-Vent™ connector. Connection can be made quickly, without special tools.

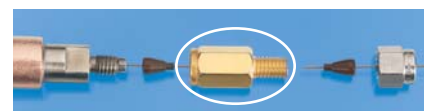
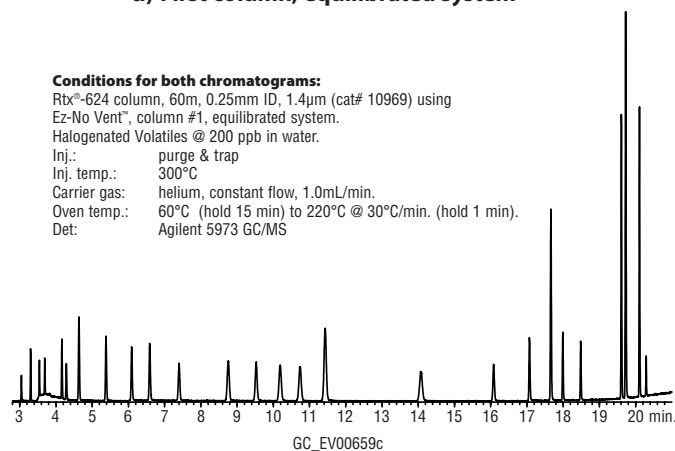
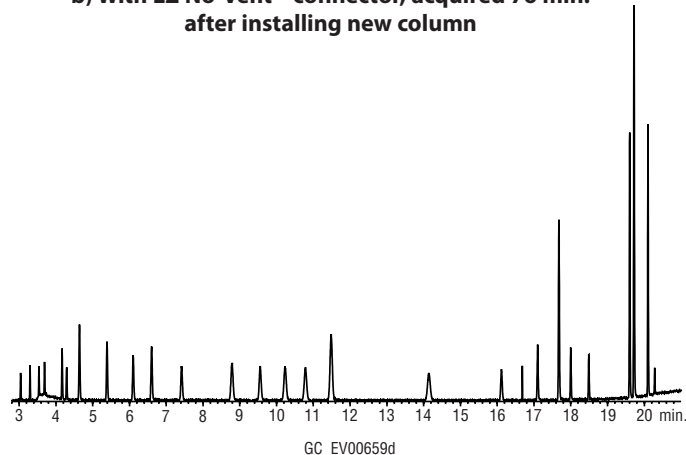


Figure 2 Sample acquired within an hour and a half after changing columns, using an EZ No-Vent™ connector.

a) First column, equilibrated system



b) With EZ No-Vent™ connector, acquired 76 min. after installing new column



EZ No-Vent™ GC Column-Mass Spectrometer Connector

Description	qty.	cat.#
EZ No-Vent™ Connector Kit for Agilent 5971/5972 and 5973 GC/MS Kit includes: EZ No-Vent™ Connector, two 0.4mm ID ferrules for capillary column, two 0.4mm ID ferrules for transfer line, 100µm deactivated transfer line (3 ft.), column plug, column nut.	kit	21323
Replacement ferrules for connecting capillary column to EZ No-Vent™:		
0.4mm ID	2-pk.	21015
0.5mm ID	2-pk.	21016
Replacement ferrules for connecting transfer line to EZ No-Vent™: 0.4mm ID	2-pk.	21043
Replacement 100µm deactivated transfer line	3 ft.	21018
Replacement EZ No-Vent™ Column Nut	5-pk.	21900
Replacement EZ No-Vent™ Plug	2-pk.	21915
Open-End Wrenches (1/4" x 3/16")	2-pk.	20110

Restek offers supplies and innovative tools for your MS. Refer to our general catalog.

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know?



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Simplify Life in Your Laboratory

by Donna Lidgett, GC Accessories Product Marketing Manager

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Injector Wrench for Shimadzu 17A and 2010 GCs

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- High-quality stainless steel construction.



Description	Similar to Shimadzu part #	qty.	cat.#
Injector Wrench for Shimadzu GCs	221-46977-00	ea.	21159

Septum Holder Kit for TRACE™ 2000 GCs

- Includes septum support and holder.
- Made from high quality stainless steel.



Description	Similar to TF part #	qty.	cat.#
Septum Holder Kit for TRACE™ 2000 GCs	23303015 350054335	ea.	21299

Silver PTV Seals for Agilent 6890 GCs



Description	Similar to Agilent part #	qty.	cat.#
Silver PTV Seals for Agilent 6890 GCs	5182-9763	5-pk.	21409


Injector Mounting Posts for Agilent Autosamplers

- Performance equivalent to OEM parts.



Description	Similar to Agilent part #	qty.	cat.#
A) Injector Mounting Post for Agilent 7673A & B Autosamplers	18597-60805	ea.	21236
B) Injector Mounting Post for Agilent 7683A Autosampler	07673-21140	ea.	21237
C) Injector Mounting Post for Agilent 7683N Autosampler	G2613-20500	ea.	21172

Splitless Liners for PerkinElmer GCs

Splitless Liners for PerkinElmer GCs	Benefits/Uses:	ID*/OD & Length (mm)	cat.#		
			ea.	5-pk.	25-pk.
	headspace & purge & trap	1.0 ID 6.2 OD x 92.1	21272	21273	21274

Auto SYS Splitless

*Nominal ID at syringe needle expulsion point.

Dual Vespel® Ring Inlet Seals

- Vespel® ring embedded in bottom surface eliminates need for washer.
- Vespel® ring embedded in top surface reduces operator variability by requiring minimal torque to seal.
- Prevents oxygen from permeating the carrier gas, increasing column lifetime.

Eliminate the washer!



Available in Siltek™-treated, gold-plated, or untreated stainless steel.

Our Dual Vespel® Ring Inlet Seal* greatly improves injection port performance—it stays sealed, even after repeated temperature cycles, without retightening the reducing nut! This new version of our popular Vespel® Ring Inlet Seal features two soft Vespel® rings, one embedded in its top surface and the other embedded in its bottom surface. The Vespel® rings eliminate the need for a washer, and ensure very little torque is needed to make a leak-tight seal. The rings will not harm the critical seal in the injector body, and are outside the sample flow path. Tests show Dual Vespel® Ring Inlet Seals will seal equally effectively at torques from 5 to 60 in. lb.

Why trust a metal-to-metal seal when you can make leak-tight seals quickly, easily, and more reliably—without a washer, with a Restek Dual Vespel® Ring Inlet Seal. Use an untreated stainless steel seal for analyses of unreactive compounds. To reduce breakdown and adsorption of active compounds, use a Siltek™-treated or gold-plated seal. Siltek™ treatment provides the highest level of inertness.

0.8mm ID Dual Vespel® Ring Inlet Seal

	2-pk.	10-pk.
Siltek™	21242	21243
Gold-Plated	21240	21241
Stainless Steel	21238	21239

1.2mm ID Dual Vespel® Ring Inlet Seal

	2-pk.	10-pk.
Siltek™	21248	21249
Gold-Plated	21246	21247
Stainless Steel	21244	21245

*Patent pending.

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FID-1000 Gas Station

Convenient, Safe Source of Zero Air and Pure Hydrogen

- Single unit produces UHP zero air from house compressed air and 99.9995% pure hydrogen from deionized water.
- Ideal for supplying 1 - 2 FIDs, FTDs, or FPDs.
- Eliminates inconvenient, dangerous cylinders.
- Silent operation, minimal operator attention required.

new!



Parker Balston's FID-1000 Gas Station provides both UHP grade hydrogen fuel gas and zero grade air (<0.1ppm THC) for flame ionization detectors on gas chromatographs. The system is specifically designed to supply fuel gas to either 1 or 2 FIDs and to support flame thermionic and flame photometric detectors.

The gas station produces up to 1000cc/min. of zero air by purifying compressed air to a total hydrocarbon concentration of less than 0.1ppm (measured as methane).

The hydrogen generator produces hydrogen gas from deionized water, using the principle of electrolytic dissociation of water and hydrogen proton conduction through a proton exchange membrane cell. Hydrogen is supplied at 90cc/min. at pressures up to 60 psig.

Built to International Standards

Produced and supported by an ISO 9001 registered organization, Parker Balston's hydrogen generators are built to meet the toughest laboratory standards - CSA, UL, CE, and IEC 1010.

When ordering an FID-100 Gas Station for use in countries other than the United States, add the appropriate international power cord suffix (see table at right) to the Gas Station cat.#.

Specifications: FID-1000 Gas Station

	Hydrogen	Zero Air
Product Purity	99.9995%	<0.1ppm total hydrocarbons
Flow Rate	90cc/min.	1000cc/min.
Delivery Pressure	60psig	40-125psig*
Inlet Connection	NA	1/4" NPT (female)
Outlet	1/8" compression	1/8" compression
Power Requirements	120VAC/amp	
Dimensions	16.5 x 10.5 x 17" (h x w x d) 42 x 27 x 43cm	
Weight Dry	46lbs / 21kg	

*Zero air inlet requires minimum of 40psig compressed air pressure.

Description	qty.	cat. #
FID-1000 Gas Station	ea.	20177

International Power Cord Sets

Location	qty.	cat.# suffix
United Kingdom (230VAC, 50/50Hz)	ea.	-550
Europe (230VAC, 50/60Hz)	ea.	-551
IEC Connector Only (230VAC, 50/60Hz)	ea.	-552
Japan (200VAC, 50/60Hz)	ea.	-556
Japan, for Zero Air (100VAC, 50/60Hz)	ea.	-553
Japan, for Hydrogen (100VAC, 50/60Hz)	ea.	-554
Japan, for Nitrogen (100VAC, 50/60Hz)	ea.	-555

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- 100% high-temperature polyimide.
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1/16"	0.4mm	10-pk.	22214	
1/16"	0.5mm	10-pk.	22215	
1/16"	0.8mm	10-pk.	22216	
1/16"	1.0mm	10-pk.	22217	
1/16"	1.2mm	10-pk.	22218	
1/16"	1/16"	10-pk.	22210	
1/8"	1/8"	10-pk.	22211	
1/4"	1/4"	10-pk.	22212	
1/4"	1/8"	10-pk.	22219	

Alumaseal™ Ferrules*

- Aluminum construction, will not crack or fragment.
- Eliminate out-gassing, make leak-tight seals, for less detector noise.
- No retightening after temperature cycles—excellent for GC/MS.
- Unique two-piece design permanently locks on fused silica tubing without causing breakage.
- Will not stick in fittings, like Vespel® or graphite.
- Use with any 1/16" compression-type fitting.



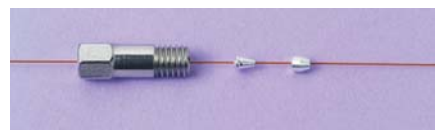
Ferrule ID	Fits Column ID	qty.	cat.#
0.4mm	0.25mm	10-pk.	21472
0.5mm	0.32mm	10-pk.	21473
0.8mm	0.53mm	10-pk.	21474

*Patent pending.



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by Rebecca Wittrig, Ph.D., HPLC Product Marketing Manager

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Description	Model #	Similar to OEM part #	qty.	cat.#
For Agilent HPLC Systems				
Detector Lamp, 1090 DA, 1050 VW/DA/MWD	1090, 1050	79883-60002	ea.	25260
Lamp, DAD G1315A, G1365A	1100	2140-0590	ea.	25261
Lamp, VWD G1314A	1100	G1314-60100	ea.	25262
8453 Deuterium Lamp	—	2140-0605	ea.	25263
G1321 Fluorescence Detector Flash Lamp	—	2140-0600	ea.	25264
For Beckman HPLC Systems				
Deuterium Lamp	DU60, 62, 64, 65	596791	ea.	25454
For Hitachi HPLC Systems				
Deuterium Lamp, Prealigned	L4000, L4200, L4250, L7400	885-3570	ea.	25465
For PerkinElmer Instruments				
Deuterium Lamp	PE Lambda--2, 5, 7, 8, 10, 11, 12, 14, 15, 16, 17, 18, 19, 20, 40, 800, 900	B0160917	ea.	25436
Deuterium Lamp	PE 200/785A	N2920149	ea.	25431
For Shimadzu HPLC Systems				
Deuterium Lamp	SPD-6A	062-65056-02	ea.	25283
Deuterium Lamp	SPD-10A, 10AV	228-34016-02	ea.	25284
For Waters™ Detectors				
Xenon Lamp (w/o holder or mirror)	470	—	ea.	25404
Xenon Lamp	474	—	ea.	25405
Deuterium Lamp (UV/Vis)	480, 481	99499	ea.	25403
Deuterium Lamp (UV/Vis)	484	80357	ea.	25406
Deuterium Lamp (UV/Vis)	486	80678	ea.	25407
Deuterium Lamp	996, 2996	WAT052586	ea.	25408
Deuterium Lamp	2487	WAS081142	ea.	25409
Deuterium Lamp, long life (2000 hours)	486	—	ea.	25410

Free Literature!



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(lit. cat.# 59012*)

Over time, detector lamps, check valves, pump piston seals, and other components wear out or become contaminated. Working with defective parts means poor chromatography and, possibly, shortened column lifetimes. This 4-page publication lists a wide selection of Restek parts for Agilent, Beckman, Hitachi, PerkinElmer, Shimadzu, and Waters instruments, to keep these systems running smoothly and chromatography sharp. Genuine Restek Replacement Parts equal or exceed the performance of original equipment components.



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*When requesting literature from outside the US, add "-INT" to the lit. cat.#.



Lit. Cat. # 59051
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