

THE RESTEK

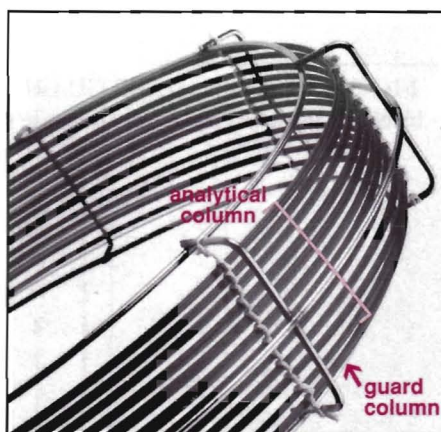
ADVANTAGE

Rtx[®]-5MS Guard: The Only Capillary Column with Built-in Protection

- Built-in 5-meter guard column eliminates difficult connection and leaks.
- Extended column life when analyzing dirty samples.
- Unsurpassed inertness and low bleed for GC/MS use.

In recent years, many capillary column manufacturers have introduced low bleed columns for use with today's more sensitive detection systems. While many of these columns offer low bleed and excellent inertness when first installed, their performance can deteriorate rapidly from nonvolatile contamination in the sample. After several injections of a dirty sample the column may no longer meet the requirements of the method. The analyst is faced with either removing the contaminated section of the column or trying to rinse out the contamination. The first option eventually results in a column that is too short to resolve the components of interest. The second option results in an increase in downtime.

Many labs have resorted to using guard columns to prevent contamination from reaching the analytical column. If the guard column becomes contaminated, a section of it can be removed without affecting the separation of



Restek's Rtx[®]-5MS column with built-in 5-meter guard column.

sample components. Although this technique has been used successfully by many labs, it has one major flaw: the connection of the guard column to the analytical column is difficult to make. Various connectors have been developed including butt, Press-Tight[®], and Vu-Union[®] connectors. However, all of these devices require some skill from the analyst, including the ability to make a good square cut on the column ends for a proper seal. The inability of many analysts to make a good connection has

resulted in reduced usage of guard columns in many labs. Also, many GC/MS users have avoided guard columns due to leakage problems caused by improper use of the connectors. Because of the high vacuum associated with the mass spectrometer, a small leak at the column connection can introduce more air into the system than a conventional atmospheric detector. A small air leak results in increased column bleed, stationary phase oxidation, compromised spectral quality, and shortened filament life.

GUARD COLUMNS—A MUST FOR DIRTY SAMPLES

Restek's chemists feel that a guard column is the most effective way to maintain column performance when analyzing dirty samples. In our efforts to promote guard column use, we wrote the *Guide to Analyzing Dirty Samples* and developed better and easier to use connection devices. However, many labs are still reluctant to use guard columns because of the difficulty in making a leak-free connection. The Restek Wizards will not be satisfied until every lab analyzing dirty samples is using a guard column. So, we are proud to introduce the world's first capillary column that contains a built-in guard column, the Rtx[®]-5 MS GUARD. By

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International Edition

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COLUMNS

only coating stationary phase on the last 30 meters of a 35 meter column, the front 5 meters are left as a guard column. Now you can have all the benefits of a guard column without the difficulties of making the connection or the worry of the connection leaking.

WHY A 5-METER BUILT-IN GUARD COLUMN?

Analysts currently using guard columns typically choose lengths of one to five meters. As the guard column becomes contaminated, the first loop (~0.5 meters) is removed to restore performance. If a 1-meter guard column is used, then this process can only be done twice before the guard column needs to be replaced. The Rtx®-5MS GUARD comes with a built-in 5-meter guard column. This allows approximately 10 loops to be removed before the guard column is consumed.

DOES A BUILT-IN GUARD COLUMN INCREASE ANALYSIS TIME?

Since the additional 5 meters is not coated with stationary phase, the sample components move through it at the same speed as the carrier gas. Therefore, the increase in analysis time is typically less than 30 seconds.

Rtx®-5MS GUARD COLUMNS GUARANTEE LOW BLEED AND EXCELLENT INERTNESS

Many guard column users complain that the addition of a guard column results in poor peak shape of active compound such as acids and bases or increases the bleed level. Since the built-in guard column is an integral part of the analytical column, it is also tested for bleed and inertness. The test chromatogram, included with each column, insures that every Rtx®-5MS GUARD column exhibits excellent inertness and low bleed even for sensitive detectors such as the ion trap. Our test mix incorporates active components such as pentachlorophenol, dinitrophenol, and dimethylphenylamine at ap-

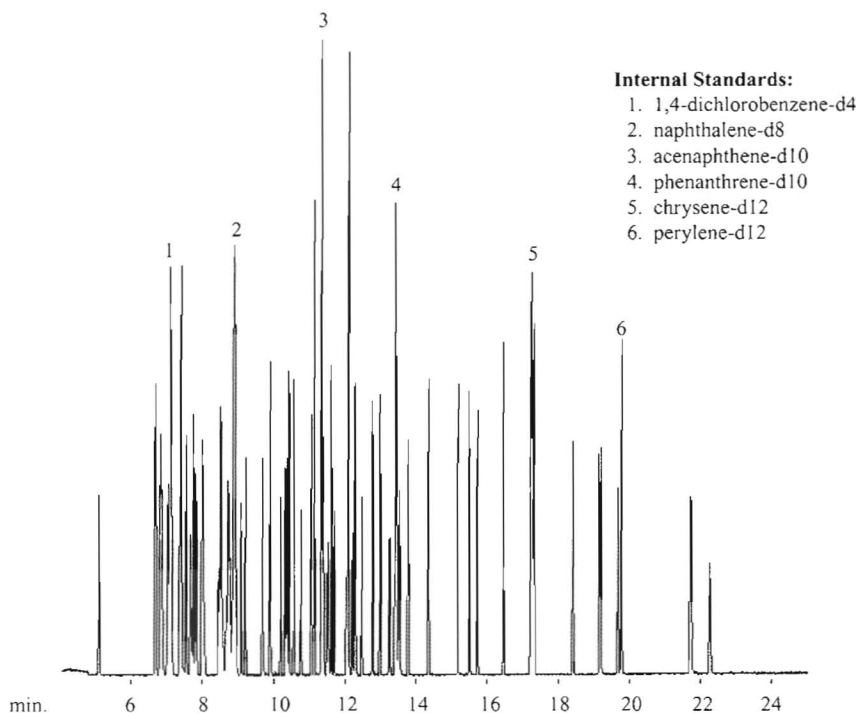
proximately 10ng on-column to insure that these columns meet the strictest requirements for analyzing semi-volatile pollutants. Figure 1 shows the analysis of semi-volatile pollutants at 20ng on the Rtx®-5MS GUARD column. Excellent inertness and low bleed are easily achieved without loss of resolution or increase in analysis time.

If you've never used a guard column when analyzing dirty samples, you can greatly increase the life expectancy of your column by using one. If you have used a guard column before, but have had problems making a good connection or had leaks with the connection, Restek has removed all the risks involved in attaching a guard column with the Rtx®-5MS GUARD column.

Run Conditions for Figure 1

COLUMN: 30m, 0.25mm ID, 0.25µm Rtx®-5MS GUARD (cat.# 12623-124)
SAMPLE: 2.0µl injection of semi-volatile calibration mix, **CONC.:** 20ng/µl
OVEN TEMP.: 45°C (hold for 3.5 min.) to 95°C @ 40°C/min. to 295° @ 17°C/min. (hold 2 min.) to 320°C @ 40°C/min. (hold 5.9 min.)
INJ. TEMP.: 250°
DET. TYPE: MSD, **DET. TEMP.:** 310°C
LINEAR VELOCITY: 32cm/sec. @ 40°C
SCAN RATE: 0.8 sec./scan,
SCAN RANGE: 35–500amu
FLOW RATE: 1.03ml/min. after EPC pressure pulse
IONIZATION: EI
ELECTRON RANGE: 70eV
SPLITLESS HOLD TIME: 0.95 min.

FIGURE 1: The Rtx®-5MS GUARD column shows excellent inertness and low bleed for the analysis of semi-volatile pollutants without increasing analysis time.



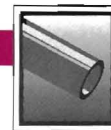
Analysis courtesy of Inchcape Testing Services - Aquatec Laboratories, Burlington, Vermont. Image file courtesy of Thru-Put Systems, Inc.

PRODUCT LIST

Rtx®-5MS GUARD (30 meter column with built-in 5 meter guard column)

ID	0.25µm	0.50µm	1.0µm	1.5µm
0.25mm	cat.# 12623-124	cat.# 12638-124	cat.# 12653-124	-----
0.32mm	cat.# 12624-125	cat.# 12639-125	cat.# 12654-125	-----
0.53mm	-----	cat.# 12640-126	cat.# 12655-126	cat.# 12670-126

Longer guard column lengths available upon request.



Analyzing Volatile Organics by Capillary Gas Chromatography

Analysts have several options when selecting a capillary column for volatile organic pollutants. Since these compounds are frequently found at trace levels in environmental samples, purge & trap concentrating techniques are used to improve detection limits. Therefore, the capillary column selected must be compatible with purge & trap equipment. The detection system also plays an important role in column selection. In recent years, the cost of mass spectrometers has been significantly reduced, which has led to their use as a common detection system for volatile organic pollutants. Two techniques for analyzing volatile organics using purge & trap concentration and mass spectrometric detection have emerged. One technique involves directly coupling the purge & trap system to a wide bore capillary column and using a jet separator or open split interface to reduce the flow entering the mass spectrometer. The other technique involves connecting the purge & trap unit to the capillary column through a split injection system to reduce the desorption flow and directly coupling the column to the mass spectrometer. Each technique can be effective for analyzing volatile organic pollutants with the proper system and column configuration.

WIDE BORE TECHNIQUE

Wide bore (0.53mm ID) columns, which operate at higher flow rates, are compatible with the desorb flow from the purge & trap system. Rapid trap desorption is critical for producing narrow peak band widths which results in improved resolution. By directly interfacing the purge & trap system to the column, all of the desorb flow is directed onto the column and sample loss is minimized. Although less efficient than narrow bore columns, wide bore columns provide greater sample capacity. This larger capacity allows calibration over a wider range of concentration before column overloading occurs. A longer column can be used to overcome the reduced efficiency of wide bore columns.

Specialized phases such as Restek's Rtx®-502.2 column have been designed to provide excellent separation of volatile organic pollutants. Figure 1 shows the analysis of 60 volatile pollutants on a 105 meter, 0.53mm ID, 3.0µm Rtx®-502.2 column. The column length and film thickness have been optimized to eliminate the need for cryofocusing or sub-ambient cooling. This Crossbond® column is coated with a dimethyl/diphenyl polysiloxane liquid phase that has a maximum operating temperature of 270°C, is resistant to damage from water va-

por, and can be solvent rinsed if contaminated. Also available is the MXT®-502.2, a fused silica lined stainless steel version of this column. This rugged metal column is ideal for portable applications since it cannot be broken even under harsh field conditions. The MXT®-502.2 provides the same resolution and advantages of the Rtx®-502.2 column.

The one significant disadvantage to using a wide bore column is the flow incompatibility of the column and the mass spectrometer vacuum system. Although the column flow rate needs to be high for fast trap desorption, the pumping capacity of most mass spectrometer vacuum systems is limited to approximately

Run Conditions for Figure 1

COLUMN: 105m, 0.53mm ID, 3.0µm

Rtx®-502.2 (cat.# 10910)

SAMPLE: 20ppb on-column concentration.

Tekmar purge & trap concentrator.

Tenax/Silica Gel/Charcoal Trap - 11 min.

purge, 2 min. desorb

OVEN TEMP.: 35°C (hold 10 min.) to 220°C @ 4°C/min. (hold 3 min.)

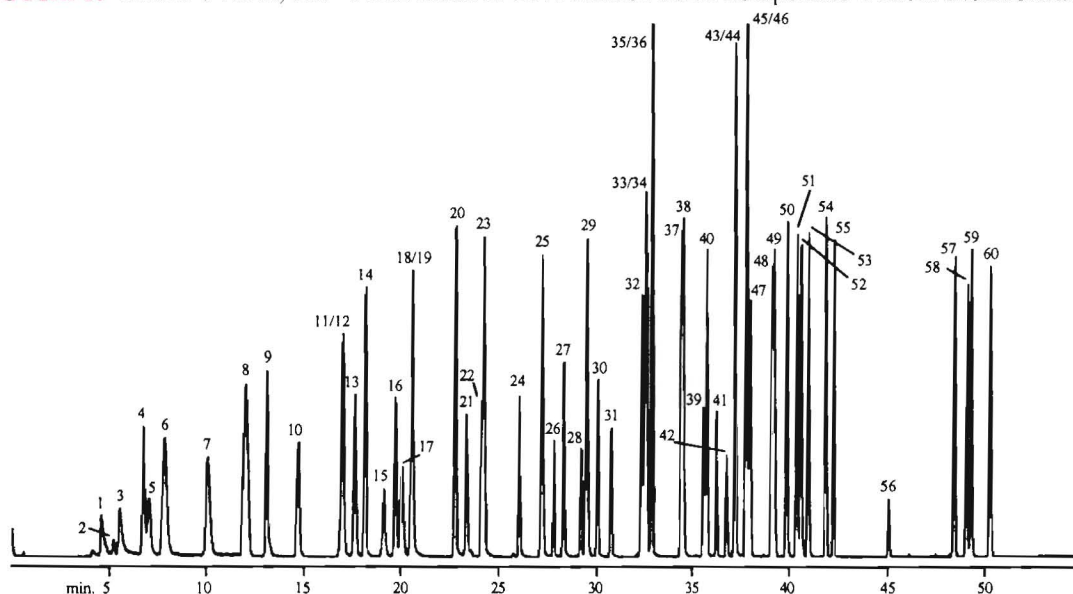
CARRIER: helium, **FLOW:** 10ml/min.

DET. TYPE: MSD, **DET. TEMP.:** 250°C

SCAN RANGE: 45-300AMU.

DESORB PREHEAT TEMP.: 220°C

FIGURE 1: Restek's 105m, Rtx®-502.2 resolves EPA Method 502.2 compounds without subambient cooling.

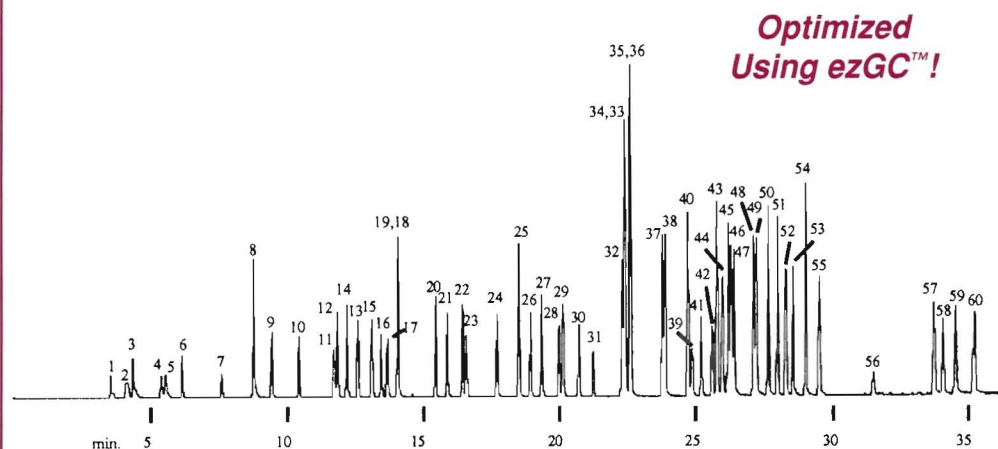




Compounds for Figures 1 & 2

1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. 1,1-dichloroethene
8. methylene chloride
9. trans-1,2-dichloroethene
10. 1,1-dichloroethane
11. 2,2-dichloropropane
12. cis-1,2-dichloroethene
13. chloroform
14. bromochloromethane
15. 1,1,1-trichloroethane
16. 1,1-dichloropropene
17. carbon tetrachloride
18. 1,2-dichloroethane
19. benzene
20. trichloroethene
21. 1,2-dichloropropane
22. bromodichloromethane
23. dibromomethane
24. cis-1,3-dichloropropene
25. toluene
26. trans-1,3-dichloropropene
27. 1,1,2-trichloroethane
28. 1,3-dichloropropane
29. tetrachloroethene
30. dibromochloromethane
31. 1,2-dibromoethane
32. chlorobenzene
33. 1,1,1,2-tetrachloroethane
34. ethylbenzene
35. *m*-xylene
36. *p*-xylene
37. *o*-xylene
38. styrene
39. bromoform
40. isopropylbenzene
41. 1,1,2,2-tetrachloroethane
42. 1,2,3-trichloropropane
43. propylbenzene
44. bromobenzene
45. 1,3,5-trimethylbenzene
46. 2-chlorotoluene
47. 4-chlorotoluene
48. tert-butylbenzene
49. 1,2,4-trimethylbenzene
50. sec-butylbenzene
51. *p*-isopropyltoluene
52. 1,3-dichlorobenzene
53. 1,4-dichlorobenzene
54. *n*-butylbenzene
55. 1,2-dichlorobenzene
56. 1,2-dibromo-3-chloropropane
57. 1,2,4-trichlorobenzene
58. hexachlorobutadiene
59. naphthalene
60. 1,2,3-trichlorobenzene

FIGURE 2: Volatile organic analysis using a 40m, 0.18mm ID, 1.0µm Rtx®-502.2 with a split/splitless injection port and directly interfaced into a mass spec source.



Run Conditions for Figure 2

COLUMN: 40m, 0.18mm ID, 1.0µm Rtx®-502.2 (cat.# 40915).
SAMPLE: 200ppb of VOA standards
OVEN TEMP.: 35°C (hold 4 min.) to 150°C @ 6°C/min., then to 220°C @ 8°C/min. (hold 8 min.)
INJ. TEMP.: 200°C, **DET. TEMP.:** 250°C,
DET. TYPE.: MSD
LINEAR VELOCITY: 21cm/sec. set @ 35°C
SPLIT RATIO: 30:1,
PURGE & TRAP: Tekmar LSC 2000
TRAP: Vocab™-4000,
PURGE TIME: 11 min.,
DESORB PREHEAT TEMP.: 250°C,
DESORB TIME: 2 min.,
DESORB FLOW RATE: 30ml/min.

1ml/min. Therefore, a jet separator or open split interface is required to prevent overloading the vacuum system. A jet separator is preferable to an open split interface since it does not result in a significant loss in sensitivity. However, a jet separator option for most mass spectrometer systems can add significantly to the cost.

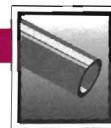
NARROW BORE TECHNIQUE

Narrow Bore (0.18-0.25mm ID) columns can also be used to analyze volatile organics. The GC/MS configuration is simpler and analysis times are normally faster, since shorter length

and thinner film columns can be used. The purge & trap transfer line is connected to a standard capillary inlet operated in the split mode. By operating in the split mode, the desorb flow rate can be reduced to a level that is compatible with both the narrow bore column and the mass spectrometer vacuum system. Typically a desorb flow of 20 to 30ml/min is used, and a split ratio between 20:1 and 30:1 is used to obtain a column flow of ~1ml/min. The major advantage to this technique is that it eliminates the need for a jet separator or open split interface since the column flow is compatible with the vacuum pumping system. The major disadvantage to this technique is that splitting the desorb flow results in a loss in sensitivity. This can be overcome by purging a larger sample volume. Figure 2 shows the analysis of volatile organic pollutants using a 40 meter, 0.18mm ID, 1.0µm Rtx®-502.2 column operated in the split mode. A 15 minute saving in analysis time with a slight improvement in resolution is achieved compared to the wide bore column technique.

INTERFACING TECHNIQUES

For either the wide bore or narrow bore technique, it is important to minimize dead volume between the purge & trap unit and the column. Any excess dead volume in a capillary system

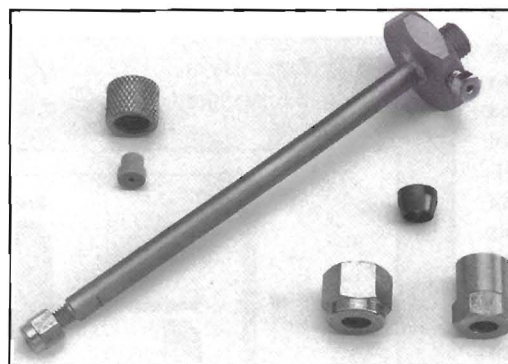


PRODUCT LIST

causes loss in resolution and should be avoided. Several options are available to reduce the volume between the purge & trap transfer line and the column. The simplest solution is to bypass the injection port and make a direct connection from the purge & trap transfer line to the column using a low dead volume fitting. However, this technique does not allow direct injections onto the column, which can be useful for troubleshooting purge & trap problems. A better technique is to connect the purge & trap transfer line to the injection port and reduce the volume by installing a 1mm ID injection port liner. The narrow diameter liner reduces dead volume in the injector but allows direct injections onto the column. Another technique for connecting a capillary column to the purge & trap unit is to use Restek's Low Volume Injector (LVI). The LVI also provides the convenience of direct injection for troubleshooting without loss in resolution. LVIs are available for several different injection systems.

The analysis of volatile organic pollutants by GC/MS can be accomplished with either narrow bore or wide bore Rtx[®]-502.2 capillary columns. Column choice is often dependent on analyst preference or the equipment available. The wide bore technique requires the use of a jet separator or open split interface, but generally offers lower detection limits. The narrow bore technique eliminates the need for a jet separator, but requires a split injection system which results in some loss in sensitivity. However, narrow bore columns offer improved resolution and reduced analysis time. The optimized Rtx[®]-502.2 stationary phase offers excellent separation for the common volatile pollutants listed in many environmental methods.

Rtx®-502.2				MXT®-502.2			
20m,	0.18mm ID,	1.0µm,	cat.# 40914	30m,	0.28mm ID,	1.6µm,	cat.# 70919
40m,	0.18mm ID,	1.0µm,	cat.# 40915	60m,	0.28mm ID,	1.6µm,	cat.# 70920
30m,	0.25mm ID,	1.4µm,	cat.# 10915	105m,	0.28mm ID,	1.6µm,	cat.# 70921
60m,	0.25mm ID,	1.4µm,	cat.# 10916	30m,	0.53mm ID,	3.0µm,	cat.# 70908
30m,	0.53mm ID,	3.0µm,	cat.# 10908	60m,	0.53mm ID,	3.0µm,	cat.# 70909
60m,	0.53mm ID,	3.0µm,	cat.# 10909	105m,	0.53mm ID,	3.0µm,	cat.# 70910
105m,	0.53mm ID,	3.0µm,	cat.# 10910				
Silcosteel® Fused Silica Lined Stainless Steel Tubing (Coiled)							
Highly inert Silcosteel® tubing is excellent as a transfer line for inert or active compounds.							
Length 6ft.	ID	OD		ID	OD		
	0.021" (0.53mm)	0.031" (0.79mm)		0.020" (0.51mm)	1/16" (1.6mm)		
	cat.#			cat.#			
	20563			20524			



Restek's low volume injector for purge & trap

Low Volume Injector (LVI):

LVI for HP Split/Splitless GC

Inlets: cat.# 21692

LVI for Varian Split/Splitless GC

Inlets: cat.# 21693

LVI for HP 5890 Septum Packed

Purge Port: cat.# 21698

Injection Port Sleeves:

1mm ID Split Sleeve for HP 5890 GCs:

cat.# 20972, each

cat.# 20973, 5-pk.

1mm ID Split Sleeve for Varian 1075/1077GCs:

cat.# 20970, each

cat.# 20971, 5-pk.

*Are you interested in learning more about Volatile Organics and other environmental analyses?
Call your local Restek distributor for more information on Restek's Environmental Seminar.*

MXT[®] Low Dead Volume Connector:

- Connect guard columns/transfer lines to MXT[®] columns.
- Low thermal mass tracks rapid oven temperature programming.



for 0.53mm ID by 0.031" OD
Silcosteel[®] transfer lines:
cat.# 20394

1/32", 0.5mm Vespel[®]/graphite ferrule for MXT[®] Connector to use fused silica tubing in an MXT[®] connector:
cat.# 20259, 5-pk.

1/16" Inert Silcosteel[®] Fittings

Connect tubing and maintain a truly inert pathway for your purge & trap. Each fitting is fused silica lined and deactivated.

1/16" Silcosteel[®] Union:
cat.# 20510, each



Don't drag your *Tail* on basic compound analysis

The reviews are in. The Rtx®-5 Amine is a success for chromatographers desperately in need of a column to perform efficient analyses of basic compounds. Plus, the Rtx®-5 Amine is cited in Environmental Protection Agency Method 1666. This column was developed with a unique deactivation technology that reduces adsorption and improves the response of basic compounds. In addition, Restek also introduced base deactivated guard columns. Base deactivated guard columns are made with the same technology as the Rtx®-5 Amine column to guarantee excellent response of basic compounds. Now Restek brings you the final step in preparing your GC system for basic compound analysis: base deactivated inlet sleeves and fused silica wool.

When a sample is injected into the hot injection port of a gas chromatographic system, it first contacts the liner (and/or its packing) as a liquid, vaporizes, and transfers as a gas into the column. The surface of fused silica (quartz) liners and wool contains silanol (SiOH) groups. The presence of acidic silanol groups results in reduced response of basic compounds when active components contact the sleeve as a liquid and are adsorbed by the fused silica surface during vaporization. If an amine sample is injected into a sleeve which does not have properly deactivated silanol groups, the sample undergoes acid/base interactions with the fused silica surface. Silanols can also hydrogen bond with basic compounds such as ethanolamines, primary amines and secondary amines. These phenomena result in peak tailing, or in extreme cases complete compound adsorption.

Through extensive research, Restek determined that standard sleeve and wool deactivations, although typically effective for many active compounds, do not have the optimum silanol deactivation chemistry for analyzing bases. By applying the unique deactivation chemistry utilized on the Rtx®-5 Amine column to inlet liners and fused silica wool, we have been able to achieve the ultimate in ba-

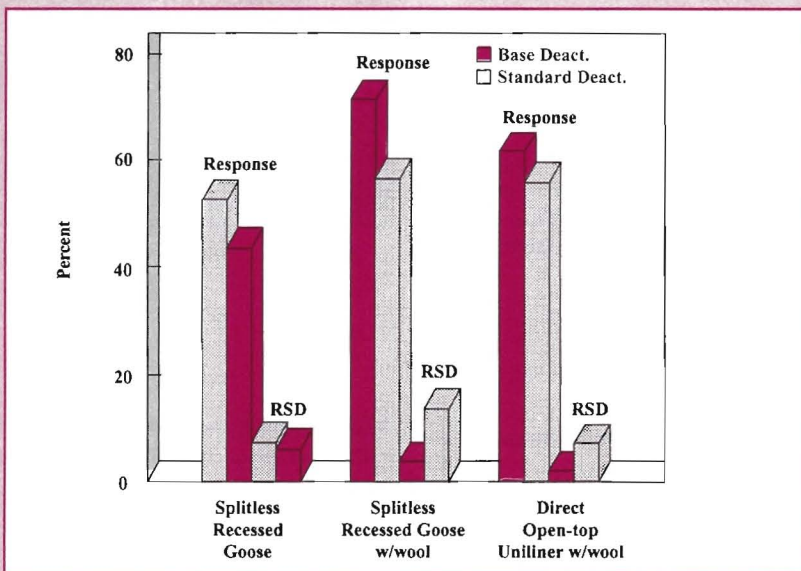
sic compound inertness for your GC: a completely inert, basic pathway for basic compound analysis! Table I shows a comparison of ethanolamine response between our standard deactivated sleeves/wool and the new base deactivated sleeves/wool. Improvements in all modes of injection (splitless and direct) are apparent. Using base deactivated fused silica wool in addition to base deactivated liners has many advantages in improving response and the standard deviation data.

Figure 1 shows two split injections of the Rtx®-5 Amine QA test mix on an Rtx®-5 Amine column. When using a base deactivated straight sleeve (chromatogram A), the response of two basic test probes, diethylenetriamine (peak 6) and diethanolamine (peak 7) is non-existent. The addition

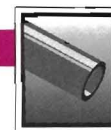
of base deactivated fused silica wool (chromatogram B) greatly improves the response of these test probes. There are several reasons for this dramatic difference.

When a sample is injected into a straight sleeve (split or splitless) without wool, the liquid travels down and contacts the bottom of the injection port. In a Hewlett-Packard split/splitless injection port, the inlet sleeve sits on a metal sealing disk. This disk is adsorptive to basic compounds. Additionally, the bottom of the sleeve becomes the main site where the sample is vaporized. If the column head is too far above this inlet disk, the sample vapor may not split at the calculated ratio $[(\text{column flow} + \text{split vent flow}) / \text{column flow}]$. These factors can contribute to sample adsorption and discrimination.

TABLE I: Response increases and RSDs decrease when base deactivated sleeves are compared to standard sleeves.



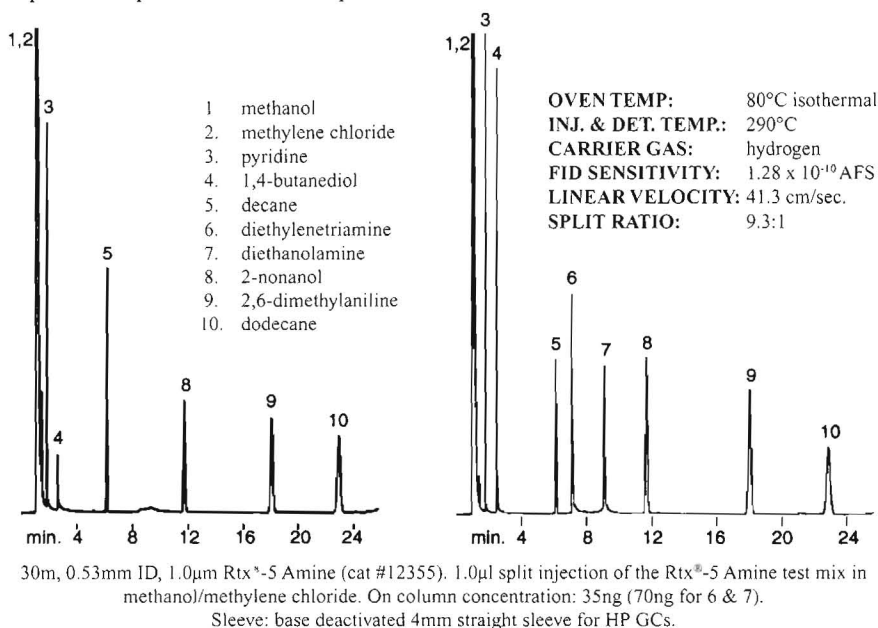
GC SYSTEM: HP 5890 II Plus with HP 7673 Autosampler
 COLUMN: 30m, 0.53mm ID, 3.0µm Rtx-5 Amine
 INJ./DET. TEMP.: 250°C/285°C
 DIETHANOLAMINE ON-COLUMN CONCENTRATION: 15ng
 INJECTIONS/SLEEVE: 5
 Each sleeve conditioned at 285°C for 1 hour prior to injections.



The addition of base deactivated fused silica wool solves these problems. In this case, the sample is injected and is trapped as a liquid inside the wool. Since wool has a high surface area, it efficiently transfers heat giving a rapid sample vaporization in the middle of the sleeve (not at the injector base). Contact between sample (as a liquid) and the injector base is therefore eliminated, and component vapor is split at the calculated ratio. Because the wool is base deactivated, basic components are not adsorbed and the inertness of the chromatographic system is complete.

With the advent of base deactivated fused silica sleeves and wool, in addition to our existing line of the Rtx®-5 Amine and base deactivated guard columns, you can be assured that your gas chromatographic system will be the most inert available on the market today.

FIGURE 1: The addition of base deactivated wool to a straight sleeve greatly improves response of basic test probes.



PRODUCT LIST

Rtx®-5 Amine (Crossbond® 5% diphenyl - 95% dimethylpolysiloxane)				
	df (µm)	temp. range	15-Meter (cat.#)	30-Meter (cat.#)
0.25mm ID	0.50	-60/315°C	12335	12338
	1.00	-60/305°C	12350	12353
0.32mm ID	1.00	-60/305°C	12351	12354
	1.50	-60/300°C	12366	12369
0.53mm ID	1.00	-60/290°C	12352	12355
	3.00	-60/285°C	12382	12385

Base Deactivated Inlet Sleeves			
	Each (cat.#)	5-pack (cat.#)	25-pack (cat.#)
4mm split straight w/ wool (for HP)	20781-211.1	20782-211.5	20783-211.25
Cyclo splitter (for HP)	20706-210.1	20707-210.5	—
4mm splitless straight (for HP)	20772-210.1	20773-210.5	—
2mm gooseneck (for HP)	20795-210.1	20796-210.5	20797-210.25
4mm gooseneck (for HP)	20798-210.1	20799-210.5	20800-210.25
Splitter w/ wool (fits Varian)	20792-211.1	20793-211.5	—
Frit Splitter (fits Varian)	20715-210.1	20716-210.5	—

If you do not see the sleeve you need, orders can be placed on a custom basis with the appropriate suffix number added. For base deactivated: each (-210.1), 5-packs (-210.5), 25-packs (-210.25). For base deactivated packed with base deactivated wool: each (-211.1), 5-packs (-211.5), 25-packs (-211.25).

Base Deactivated FS Guard Columns		
Nominal ID	Nominal OD	5-meter length
0.25mm	0.37 ± 0.04mm	cat.# 10000
0.32mm	0.45 ± 0.04mm	cat.# 10001
0.53mm	0.69 ± 0.05mm	cat.# 10002

Add -600 to the cat.# and receive 6 guard columns for the price of 5!

Base Deactivated FS Wool
10 grams: cat.# 20999

Amine Column Test Mix	
pyridine	0.6mg/ml
1,2-butanediol	0.6
n-decane	0.6
diethylenetriamine	1.2
diethanolamine	1.2
2-nonanol	0.6
2,6-dimethylaniline	0.6
n-dodecane	0.6

Packaged 1ml/ampul,
methanol/methylene chloride (1:1)
Cat.# 35002



The new Rtx[®]-Wax Column...

Try it, you'll like it!

- 20°C minimum operating temperature
- Thermally stable with guaranteed low bleed to 250°C
- Fast and efficient for BTEX analysis
- Excellent efficiency and inertness for analysis of aldehydes

Restek's Rtx[®]-Wax columns are made with a new Crossbond[®] polyethylene glycol (PEG) stationary phase. This new innovation in polymer technology has produced the most inert and efficient PEG columns currently available. The extended operating temperature range allows the analysis of compounds with a wide volatility range, while the Crossbond[®] technology ensures low bleed at temperatures as high as 250°C. The selectivity of the Rtx[®]-Wax is comparable to other bonded Carbowax[®] columns*, yielding excellent resolution of compounds ranging from intermediate to high polarity. This exceptional inertness and efficiency allows versatility in difficult analyses such as isomeric separations, aldehydes, and alcohols.

REDUCED MINIMUM OPERATING TEMPERATURE

Many PEG columns undergo a solid-liquid phase transition at temperatures below 40°C that results in a loss of efficiency, reduced sample capacity, and poor retention time reproducibility¹. The new Rtx[®]-Wax column can effectively operate at 20°C without displaying any adverse effects. This is especially advantageous for applications involving purge and trap and headspace analyses where volatile components must be cold trapped onto the column. Figures 1 and 2 demonstrate the difference in PEG column performance at 20°C. Figure 1 is a chromatogram of 8 volatile components on the Rtx[®]-Wax column. Figure 2 shows the same analysis and conditions on a bonded Carbowax[®] column. The Rtx[®]-Wax column provides better column efficiency and resolution of the analytes demonstrating its superior suitability for low temperature analyses.

THERMALLY STABLE TO 250°C

Crosslinking or bonding of the PEG stationary phase minimizes bleed to ensure accurate identification and quantitation of higher-boiling components. As a result, minimal detector contamination and extended column lifetime are observed. Only a small baseline rise is evident at the maximum operating temperature for the stationary phase. In comparison to similar PEGs from other manufacturers, the Rtx[®]-Wax exhibits the lowest bleed profile at 250°C. (Figure 3)

RESOLUTION OF BTEX ISOMERS

The new Rtx[®]-Wax offers the same selectivity as other Carbowax[®] columns for isomers of substituted aromatics. This is useful for BTEX analyses that require the specific quantitation of the individual xylene isomers. Figure 4 illustrates all components in the BTEX analysis (meta-, para-, and ortho- xylene) are completely resolved in just 18 minutes.

Rtx[®]-Wax maintains column efficiency at 20°C

FIGURE 1: Rtx[®]-Wax (cat.# 12455), 30m, 0.53mm ID, 1.0µm

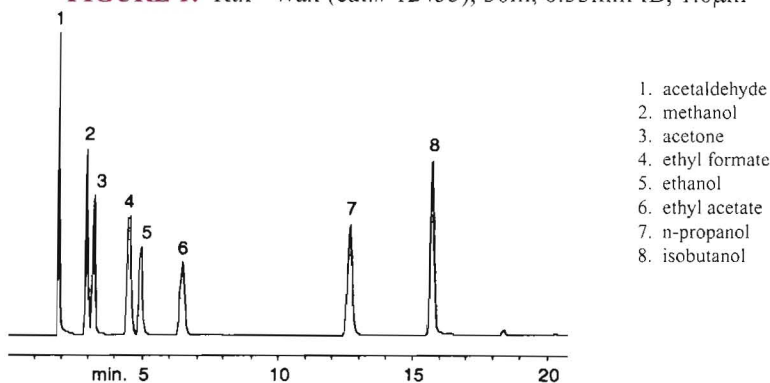
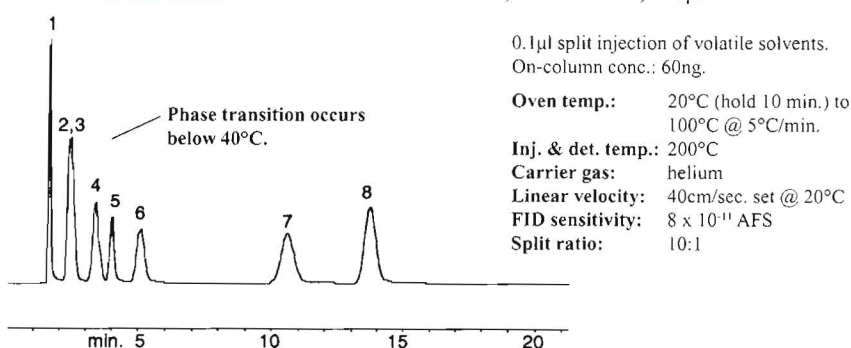


FIGURE 2: Bonded Carbowax[®] 30m, 0.53mm ID, 1.0µm



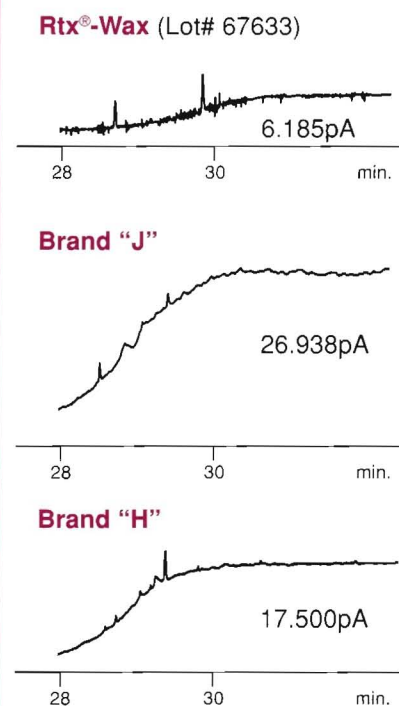
* Selectivity data available upon request.



In comparison with other available Carbowax® columns, the Crossbond® Rtx®-Wax column provides an extended operational temperature range, excellent efficiency, selectivity, and superior inertness. These advantages make it the logical choice for all Carbowax® applications.

¹ Sandra, P.; David, F.; Turner, K.A.; McNair, H.M.; Brownstein, A.D. "Observations with High-Molecular-Weight Polyethylene Glycol Stationary Phases in Capillary Gas Chromatography," *Journal of Chromatography*, 1989.

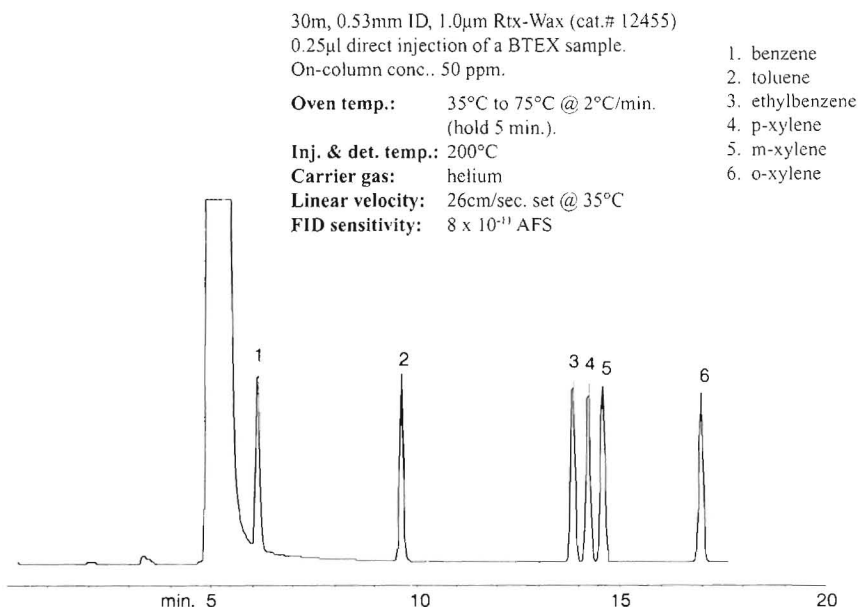
FIGURE 3: Rtx®-Wax exhibits lowest bleed profile at 250°C



Bleed profiles on 3 types of
PEG columns
30m, 0.25mm ID, 0.50µm

OVEN TEMP.: 40°C to 175°C @
6°C/min. to 250°C @ 15°C/min.
(hold 5 min.).
INJ. & DET. TEMP.: 250°C
CARRIER GAS: hydrogen
LINEAR VELOCITY: 45cm/sec.
set @ 40°C
FID SENSITIVITY: 8×10^{-11} AFS

FIGURE 4: Rtx®-Wax efficiently resolves all isomers in BTEX analysis



PRODUCT LIST

Rtx®-WAX Fused Silica Capillary Columns

mm ID	df (µ)	temp. range (°C)	15-meter	30-meter	60-meter
0.25	0.10	20-250	cat.# 12405	cat.# 12408	—
	0.25	20-250	cat.# 12420	cat.# 12423	cat.# 12426
	0.50	20-250	cat.# 12435	cat.# 12438	cat.# 12441
0.32	0.10	20-250	cat.# 12406	cat.# 12409	—
	0.25	20-250	cat.# 12421	cat.# 12424	cat.# 12427
	0.50	20-250	cat.# 12436	cat.# 12439	cat.# 12442
	1.00	20-240	cat.# 12451	cat.# 12454	cat.# 12457
0.53	0.25	20-250	cat.# 12422	cat.# 12425	—
	0.50	20-250	cat.# 12437	cat.# 12440	cat.# 12443
	1.00	20-240	cat.# 12452	cat.# 12455	cat.# 12458



Rt-608Pkd Silcosteel® Packed Column for the Analysis of Chlorinated Pesticides

- Restek's proprietary deactivation processes for Silcoport™ packing and Silcosteel® tubing provides unsurpassed inertness.
- Lowest DDT and endrin breakdown of any packed column on the market.
- Fused silica lined Silcosteel® tubing offers column inertness, durability and flexibility.
- Optimized Rt-2250/2401 stationary phase for resolution of chlorinated pesticides.
- Preconditioned phase requires only 30 to 45 minutes conditioning for low bleed and stable retention times.

LIMITATIONS OF PACKED COLUMNS FOR PESTICIDE ANALYSIS

Packed columns have been used for pesticide analysis for more than 25 years. These columns have been typically made of glass to reduce the amount of decomposition and breakdown of compounds such as endrin and 4,4'-DDT that can occur on metal columns. Due to their rigid nature, glass columns are manufactured to fit specific gas chromatographs. Often this prevents an analyst from moving a column from one instrument to another. Anyone who has installed a glass packed column realizes how fragile they are and how often the column breaks during installation.

The inertness of a packed column is limited by high surface area of the solid support. If not properly deactivated, the support can contribute to decomposition and breakdown of pesticides such as endrin and 4,4'-DDT. Since packed columns have limited separation power, the phase composition must be optimized to separate the analytes of interest. Although all these factors have limited the effectiveness of packed columns for pesticides analysis, new technology developed at Restek has overcome these problems.

IMPROVING COLUMN DURABILITY, FLEXIBILITY, AND INERTNESS

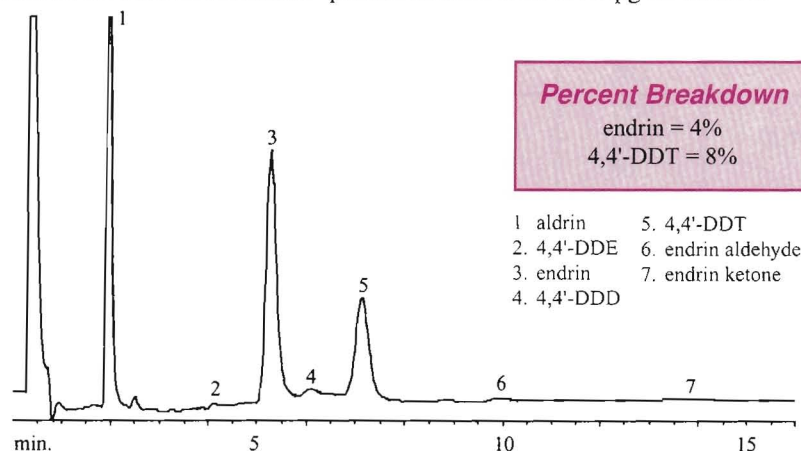
Restek has improved the durability, flexibility, and inertness of packed columns with the use of Silcosteel® tubing. Silcosteel® is fused silica lined stainless steel tubing which combines the durability of stainless steel with the inertness of fused silica. This enables the end user to handle the column without the concern of column breakage. Silcosteel® tubing also provides the analyst with flexibility, allowing the freedom to install the same column in different gas chromatographs. The inertness of the column is further enhanced by the deactivation of the tubing using technology developed for capillary columns.

Improving the inertness of the solid support was also an important consideration. Chromosorb®W, a diatomite-based solid support, is the most common choice for pesticide packed columns.¹ The chemists at Restek have developed a superior deactivation technology that results in significantly less decomposition and adsorption of active pesticides. Figure 1 demonstrates the inertness of the Rt-608Pkd Silcosteel® column and Silcoport™ support material using a 50pg endrin and 4,4'-DDT standard. The results show that there is only 4% decomposition for endrin and 8% for 4,4'-DDT.

FINDING THE OPTIMUM STATIONARY PHASE FOR PESTICIDE ANALYSIS

The stationary phases of conventional packed columns do not always resolve the compounds of interest required by many pesticide methods. Figure 2 is an example of a common packed column used for the analysis of chlorinated pesticides. Eighteen compounds found in EPA Method 608 were used for the analysis.² Three critical pairs of compounds, including 4,4'-DDE and dieldrin, endosulfan II and 4,4'-DDD, and endrin ketone and methoxy-chlor, are not fully resolved. Because of the lack of resolution using a common phase composition, the technique of window diagramming was used to determine the optimized phase composition for chlorinated pesticides. Window diagramming can predict the optimum stationary phase loading by plotting the retention of components versus the percent stationary phase.⁴ A stationary phase of specific composition is chosen from the window diagram that theoretically offers the best resolution of the analytes of interest. Figure 3 shows the resolution achieved by the optimized stationary phase predicted from the window diagram. Although not all components are completely resolved, there are significant improvements in the resolution of several critical pairs and a 66% reduction in analysis time.

FIGURE 1: Extremely low breakdown of endrin and DDT can be achieved with an Rt-608Pkd Silcosteel® packed column even at 50 pg on-column.



Run Conditions for Figure 1

2 meter, 2.0mm ID Rt-608Pkd (cat.# 80221-810)
OVEN TEMP.: 200°C isothermal
INJ. TEMP.: 250°C, **DET. TEMP.:** 250°C
COLUMN FLOW: helium, 30cc/min.
DET. TYPE: ECD

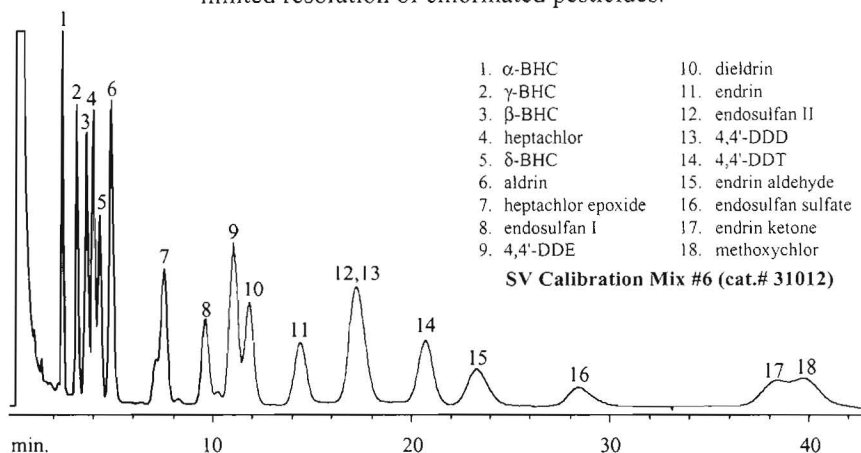


Another drawback of packed columns is long conditioning times. Restek has developed a technique for preconditioning the solid supports before they are packed. This allows the analyst to install, condition, and begin analyzing in a relatively short period of time. Conditioning periods for the pesticide packing range from 30 to 45 minutes even when using a sensitive electron capture detector (ECD).

Rt-608Pkd COLUMNS OFFER MANY BENEFITS FOR PESTICIDE ANALYSIS

The features of Restek's new Rt-608Pkd Silcosteel[®] column solve many of the shortcomings of packed columns for chlorinated pesticide analysis. The Silcosteel[®] tubing eliminates column breakage without sacrificing inertness. The tubing offers the flexibility required to move the same column to different instrument configurations. New techniques for deactivating the solid support minimize breakdown and adsorption of pesticides. Finally, the development of an optimized stationary phase results in improved separation of the common pesticides found in the Environmental Protection Agency's Method 608.

FIGURE 2: Conventional packed column offers limited resolution of chlorinated pesticides.



Run Conditions for Figure 2

2 meter, 4.0mm ID GP 1.5% SP-2250/1.95% SP-2401 on 100/120 Supelcoport[™]
(cat.# 2-3077, Lot# T-38853)
OVEN TEMP.: 200°C isothermal
INJ. TEMP. 250°C, DET. TEMP.: 250°C
COLUMN FLOW: helium, 60cc/min.
DET. TYPE: FID

Run Conditions for Figure 3

2 meter, 2.0mm ID Rt-608Pkd (cat.# 80221-810)
OVEN TEMP.: 200°C isothermal
INJ. TEMP. 250°C, DET. TEMP.: 250°C
COLUMN FLOW: helium, 40cc/min.
DET. TYPE: FID

PRODUCT LIST

Rt-608Pkd Column*

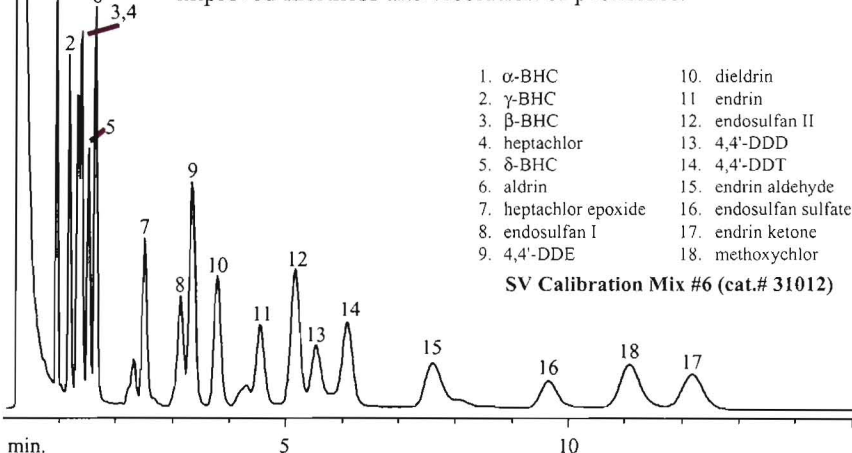
2m, 2.0mm ID cat.# 80221

2m, 4.0mm ID cat.# 80220

*Please specify instrument configuration when ordering. Add three digit suffix to column part number.

Instrument Configuration	Suffix
General configuration, fits most GCs	-800
HP 5880, 5890, 6890	-810
Varian 3700, Vista Series, FID	-820
Perkin Elmer 900, 3920, Sigma 1,2,3	-830

FIGURE 3: Rt-608Pkd Silcosteel[®] packed column exhibits improved inertness and resolution of pesticides.



¹Ottenstein, D.M., *Journal of Gas Chromatography*, "Column Support Material for use in Gas Chromatography". April, 1963.

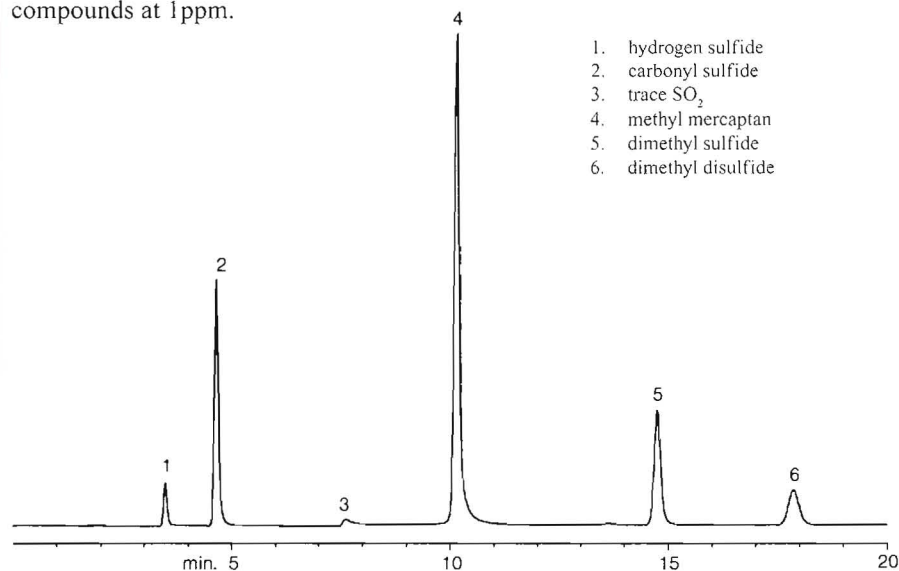
²EPA Method 608. Organochlorine Pesticides and PCB's. EPA 600 Series, Vol. 49, No. 209, October 1984.

³Thompson, John, F., Walker, Annita, C., and Moseman, Robert, F. *Journal of AOAC*, "Evaluation of Eight Gas Chromatographic Columns for Chlorinated Pesticides" Vol. 52, 1969.

⁴Laub, R.J. and Purnell, J.H. *Journal of Chromatography*, "Criteria for the Use of Mixed Solvents in Gas-Liquid Chromatography" Vol. 112, 1975.



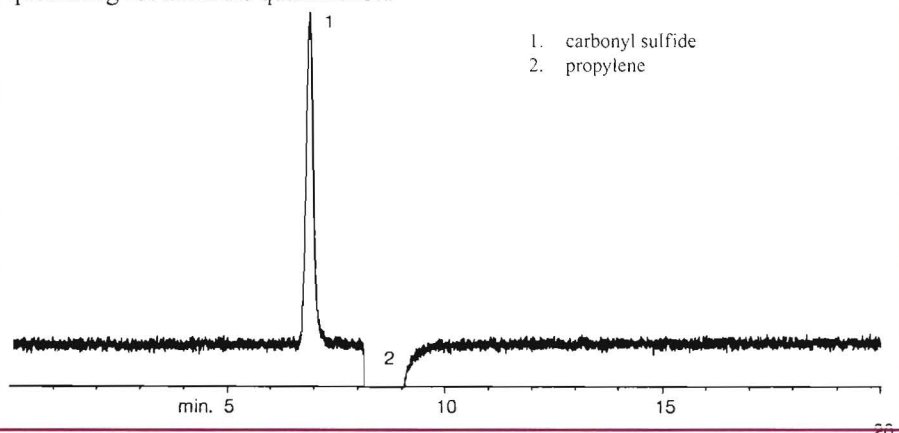
FIGURE 2: RT-Sulfur™ packed column shows good response of sulfur compounds at 1ppm.



Run Conditions for Figure 2

COLUMN: 2m x 1/8" x 2mm RT-Sulfur™ (cat.# 80252) **DETECTOR:** FPD (sensitivity: range 5)
SAMPLE: 1cc injection of sulfur compounds **CARRIER GAS:** helium
OVEN TEMP.: 60°C to 250°C @ 15°C/min. **FLOW:** 20ml/min.
 (hold 15 min.)

FIGURE 3: RT-Sulfur™ resolves COS from propylene to eliminate FPD quenching for accurate quantitation.



Run Conditions for Figure 3

COLUMN: 2m x 1/8" x 2mm RT-Sulfur™ (cat.# 80252) **DETECTOR:** FPD (sensitivity: range 3)
SAMPLE: 400µl injection **CARRIER GAS:** helium
OVEN TEMP.: 60°C to 100°C @ 5°C/min. **FLOW:** 20ml/min.
 (hold 15 min.)

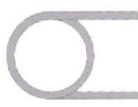




PRODUCT LIST

RT-Sulfur™ Packed Columns

Length x OD x ID	Cat.#
2m x 3/16" x 4mm*	80250
1m x 3/16" x 4mm*	80251
2m x 1/8" x 2mm*	80252
1m x 1/8" x 2mm*	80253
2m x 1/16" x 1mm	19800
1m x 1/16" x 1mm	19801
2m x 0.95mm x 0.75mm	19802
1m x 0.95mm x 0.75mm	19803

* Please include instrument configuration suffix to catalog number.

Instrument Configurations

	
General Configuration Suffix # -800	HP 5880, 5890, 5987 Suffix # -810
	
Varian 3700, Vista Series, FID Suffix # -820	PE 900-3920, Sigma 1,2,3 Suffix # -830
	Other configurations are available. Please contact your local Restek distributor for details.
PE Auto System 8300,8400, 8700 (Not On-Column) Suffix # -840	

Call about Restek's complete line of Silcosteel® packed columns. A wide variety of packings and phases are available on inert & unbreakable Silcosteel® tubing. Restek's bonding chemistry and stringent manufacturing guidelines insure the highest quality packed columns on the market today!



Analyze Sulfur Compounds at Trace Levels Using RT-Sulfur™ Packed Column

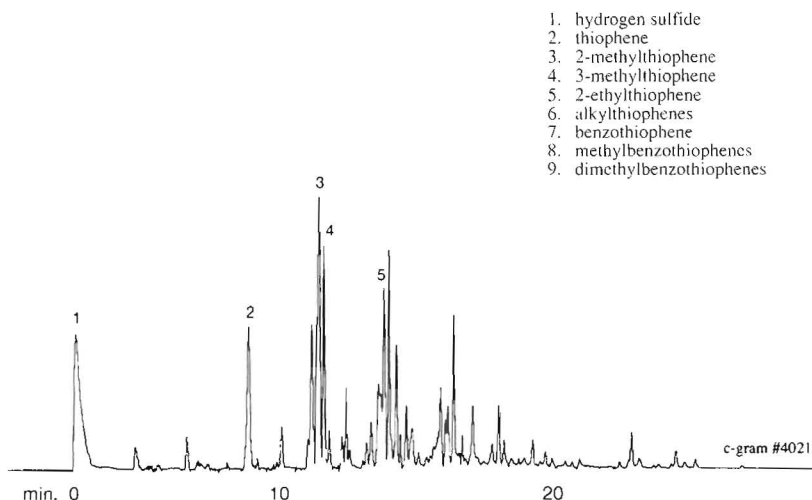
The analysis of sulfur compounds by gas chromatography remains an important but difficult application. The presence of sulfur compounds in petroleum products can have detrimental effects on the longevity and performance of catalysts used in hydrocarbon processing. Also, the toxicity and odor problems associated with sulfur compounds and their effect on air quality emphasizes the importance of quantitating trace levels of these compounds in petroleum streams.

A common method for the analysis of sulfur compounds by GC utilizes a thick film 100% polydimethylsiloxane (Rtx®-1) capillary column. Low levels of sulfur compounds can be quantified in a hydrocarbon stream by using a sulfur specific detector, such as a sulfur chemiluminescence detector. Figure 1 shows the analysis of sulfur compounds using a 30 meter, 0.32mm ID, 4.0µm Rtx®-1 capillary column. A thick film is required to obtain sufficient retention of volatile sulfur compounds such as hydrogen sulfide. However, this thick film results in prolonged analysis time for higher molecular weight sulfur compounds.

Another option for the analyst is to use packed columns filled with divinylbenzene porous polymers. Volatile sulfur compounds such as hydrogen sulfide (H₂S) and carbonyl sulfide (COS) are easily retained on this packing and are better separated from hydrocarbons which can interfere with quantitation on some sulfur specific detectors. The major disadvantage to using packed columns for sulfur analysis is non-reproducibility at trace levels caused by adsorption on the porous polymer and the column walls.

Research at Restek has led to the solution to these adsorption problems and the development of the RT-Sulfur™ packed column for trace level sulfur analysis. A deactivation process was developed for the divinylbenzene porous polymer which eliminates adsorption of sulfur compounds even at low ppb concentration. This innovative process has been thoroughly investigated and is thermally stable to 250°C.

FIGURE 1: Analysis of sulfur compounds using an Rtx®-1 capillary column.



Run Conditions for Figure 1

COLUMN: 30m, 0.32mm ID, 4.0µm Rtx®-1 (cat.# 10198)
SAMPLE: 1.0µl split injection of naphtha containing 500ppm total sulfur
OVEN TEMP.: 35°C to 275°C @ 10°C/min. (hold 5 min.)
INJ. & DET. TEMP.: 275°
DETECTOR: Atomic Emission Detector (181nm)
CARRIER GAS: helium
LINEAR VELOCITY: 24cm/sec. (0.8ml/min.)
SPLIT RATIO: 10:1

The other major cause of sulfur compound adsorption on packed columns was the column material. Glass columns offer the inertness necessary for the analysis of trace level sulfur compounds but are fragile and variations in inner diameter produce column to column variability. Although more rugged than glass, traditional stainless steel columns completely adsorb sulfur compounds at trace levels. By utilizing Restek's proprietary Silcosteel® process, an inert submicron layer of flexible fused silica can be deposited on the stainless steel tubing surface. The Silcosteel® deactivation process renders the stainless steel inert to sulfur compounds, even at trace levels. Because the Silcosteel® layer is flexible, these columns are ideal for process, field, or laboratory instruments without fear of column breakage.

Figure 2 shows the analysis of several sulfur compounds at the 1ppm level. Even at these low levels all peaks are symmetrical and well resolved. The polarity of the sulfur packed column offers good separation of most sulfur compounds from interfering hydrocarbons to insure accurate quantitation. Figure 3 shows the separation of carbonyl sulfide from industrial grade propylene.

Restek's new RT-Sulfur™ packed column gives the analyst a reliable, reproducible tool for the analysis of trace level sulfur compounds. The unique deactivation process for porous polymers has produced a packing with no adsorption of sulfur compounds. The incorporation of Restek's Silcosteel® process to packed stainless steel columns has opened the door for applications previously unattainable on traditional metal packed columns by providing an inert column surface.



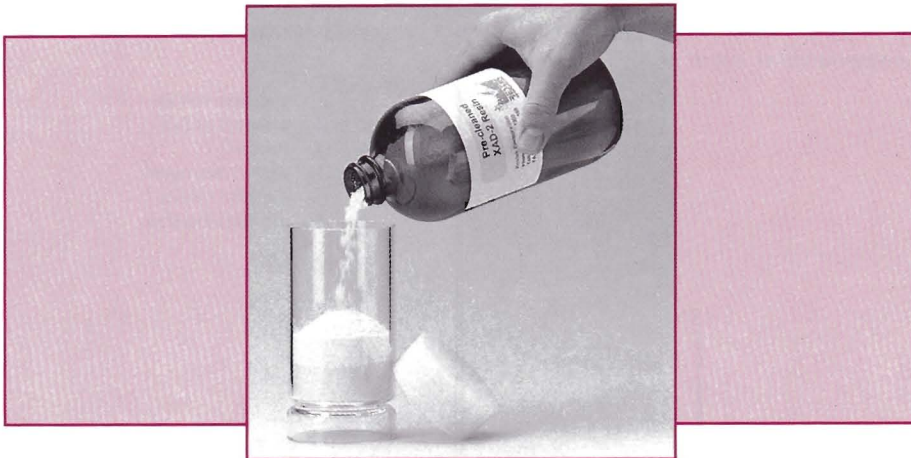
Ultra-Clean XAD-2 Resin for Air Sampling

- GC tested and certified clean by TO-13 protocol.
- Available in 100 & 500 gram sizes.
- Larger quantities available on request.

Amberlite XAD-2 resin is a common adsorbent used for collection of semi-volatile airborne components. Several of the Toxic Organic Methods specify the use of XAD-2 as the collection medium for polynuclear aromatic hydrocarbons (PAHs) and pesticides. TO-13 requires XAD-2 resin to be placed between polyurethane foam (PUF) plugs in a glass cartridge. The air is passed through the cartridge and the more volatile PAHs are trapped on the XAD-2 resin due to its high collection efficiency for PAHs. The glass cartridge, containing the PUF and XAD-2, is then extracted with solvent using a soxhlet. The extract is concentrated and analyzed by capillary GC.

Even though XAD-2 resin is an excellent adsorbent for trapping PAHs, it requires extensive cleanup because many of its impurities are PAH compounds. The TO-13 method specifies a clean up procedure to prepare the XAD-2 adsorbent for use. However, to meet the minimum level of contamination, the XAD-2 resin may require several repetitions of this cleanup procedure. Many laboratories have developed their own cleanup procedure that can take more than a week to effectively clean up the XAD-2 to meet the requirements of the TO-13 method. Restek has developed a cleaning procedure for XAD-2 resin that meets the minimum contamination levels specified in method TO-13. To ensure cleanliness, each batch is tested by capillary GC using a flame ionization detector (FID).

To demonstrate the cleanliness of Ultra-Clean XAD-2 resin, we followed the TO-13 test procedure. A 55 gram sample of Ultra-Clean XAD-2 was soxhlet extracted with 500 mls of methylene chloride. The solvent was then concentrated, using a Kuderna-Danish (KD) concentrator to a final volume of 1 ml. A 10ng/ μ l internal standard mix was added to the 1 ml concentrate since the TO-13 protocol specifies that the background contamination should be below 10 ng per component. The spiked extract was then analyzed by GC/FID. Figure 1 shows the GC/FID chromatogram of Ultra-Clean XAD-2 resin. The background easily meets the 10ng per component specified in the TO-13 method.



TO-13 requires XAD-2 resin to be placed between polyurethane foam (PUF) plugs in a glass cartridge.

Eliminate the time consuming chore of cleaning and testing XAD resin with Ultra-Clean XAD-2. Its purity exceeds all requirements of the Environmental Protection Agency's (EPA) Method TO-13. Low ECD background allows Ultra-Clean XAD-2 to be used for the collection of chlorinated pesticides and PCBs. It is available in both 100 and 500 grams sizes or larger bulk quantities upon request.

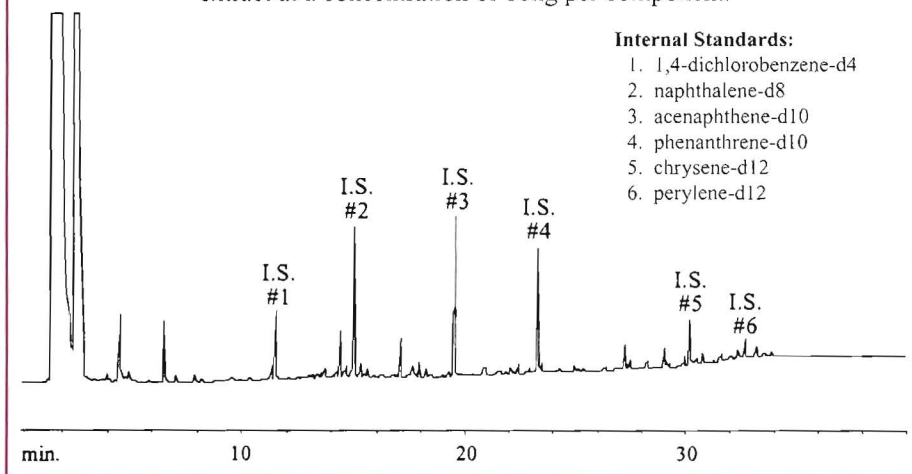
PRODUCT LIST

Ultra-Clean XAD-2 Resin

100 grams: cat.# 24230

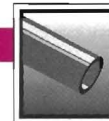
500 grams: cat.# 24231

FIGURE 1: Ultra-Clean XAD-2 resin easily meets the requirement of EPA method TO-13. A six component internal standard mix was spiked into the extract at a concentration of 10ng per component.



Run Conditions for Figure 1

30m, 0.53mm ID, 0.50 μ m Rtx[®]-5 (cat.# 10240)
INJ. SIZE: 1 μ L, **MODE:** splitless, **SPLITLESS HOLD TIME:** 0.50 min.
OVEN TEMP: 40°C (hold 6 min.) to 300°C @ 10°C/min. (hold 15 min.)
INJ. TEMP: 250°C, **DET. TYPE:** FID, **DET. TEMP:** 300°C
COLUMN FLOW: helium, **LINEAR VELOCITY:** 50cm/sec. set @ 40°C
DET. SENSITIVITY: 8 X 10⁻¹¹ AFS



Hydroguard™ Water Resistant Guard Tubing/Transfer Lines in MXT® and Fused Silica Columns

When transfer lines from purge & traps, air monitoring equipment, or other instruments carry condensed water vapor, the deactivated tubing of a column quickly becomes active due to the creation of free silanol groups. These silanol groups subsequently cause adsorption of active oxygenated compounds such as alcohols and diols.

Restek's chemists have addressed this concern and found a solution, our Hydroguard™ deactivation process. By using a unique deactivation chemistry, a high density surface is created that is not readily attacked after an aggressive hydrolysis treatment. The high density surface coverage effectively prevents water vapor from reaching the fused silica surface beneath the Hydroguard™ deactivation layer.

Analysts using guard tubing or transfer line tubing that will be exposed to condensed water vapors should use Hydroguard™ tubing and transfer lines. For those analysts injecting organic solvents, Restek's standard deactivated guard tubing is preferred.

5m length Hydroguard™ FS Guard Columns & Transfer Line 6-pks.

Nominal ID	cat.#
0.25mm	10079-600
0.32mm	10080-600
0.53mm	10081-600

Use Hydroguard™ Tubing for Connecting GCs to:

- Purge and trap systems
- Headspace analyzers
- Air concentrator units
- Air analysis equipment
- Other instruments that trap/release water vapors
- Any analytical instrument that needs an inert, water resistant pathway

Benefits of Hydroguard™ Tubing:

- Resists degradation from water injections.
- Withstands harsh "steam cleaning" chromatography processes.
- Increases column lifetime.
- Reduces effects of dirty samples on column performance.
- Reduces downtime and maintenance.
- Protects expensive analytical columns.
- Prevents damage from harmful materials.

Hydroguard™ MXT® Guard Columns & Transfer Lines

Nominal ID	Nominal OD	5m length	10m length	30m length	60m length**
0.28mm	0.53 ± 0.025mm	70080	70083	70086	70089
0.53mm	0.74 ± 0.025mm	70081	70084	70087	70090

Hydroguard™ Fused Silica Guard Columns & Transfer Lines

Nominal ID	Nominal OD	5m length	10m length	30m length*	60m length**
0.05mm*	0.363 ± 0.012mm	10075	----	----	----
0.10mm*	0.363 ± 0.012mm	10076	----	----	----
0.15mm	0.363 ± 0.012mm	10077	----	----	----
0.18mm	0.34 ± 0.01mm	10078	----	----	----
0.25mm	0.37 ± 0.04mm	10079	10082	10085	10088
0.32mm	0.45 ± 0.04mm	10080	10083	10086	10089
0.53mm	0.69 ± 0.05mm	10081	10084	10087	10090

* Not tested with the Grob mix due to a high pressure drop.

** Recommendation: Cut 60m guard columns into shorter lengths.

Using full length may cause peak distortion.

+ 30 and 60-meter lengths are banded in 5-meter sections and mounted in a Restek cage.

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International Edition

THE RESTEK ADVANTAGE

15



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