

# Analyze Chlorinated Pesticides, PCBs, and Chlorinated Herbicides

**Using Rtx®-CLPesticides and Rtx®-CLPesticides2 Fused Silica Columns**

- Reduce down time by running multiple methods on a single column set.
- Simultaneously detect and confirm 20 chlorinated pesticides.
- Speed up analysis times—less than 7 minutes on 0.32mm columns.



**Chromatography Products**

[www.restek.com](http://www.restek.com)

# Dual Column Analysis for Chlorinated Pesticides, PCBs, and Herbicides Using an Rtx®-CLPesticides Column Pair

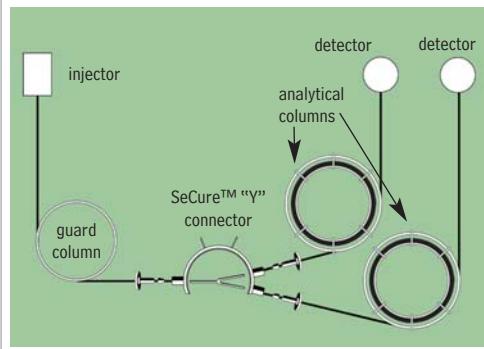
Rtx®-CLPesticides and Rtx®-CLPesticides2 columns are engineered for faster analyses and reduced downtime. New phase film thicknesses and optimized run conditions allow rapid analysis without sacrificing column capacity—meaning much faster sample throughput for your laboratory. These columns are designed for dual column analysis, which reduces injection port maintenance and allows primary and confirmation analyses from a single injection.

Dual column analysis cuts injection port maintenance time in half by coupling two analytical columns to a single guard column and injection port using a “Y” connector (Figure 1). This instrument set-up offers many advantages including:

- Method compliant results in half the time. Provides simultaneous quantification and confirmation using columns with different selectivity.
- Reduced contamination; guard column traps high molecular weight compounds, protecting the analytical columns.
- Resolution and retention times are unaffected by maintenance. Guard column can be trimmed instead of the analytical columns.
- Consistent vaporization and split for enhanced reproducibility.

In addition to compatibility with a dual column set-up, the Rtx®-CLPesticides and Rtx®-CLPesticides2 columns are ideal for multiple environmental ECD methods. Here we demonstrate the effectiveness of this column pair for chlorinated pesticides, PCBs as Aroclors, and chlorinated herbicides. Speed up your analyses and reduce downtime using the versatile Rtx®-CLPesticides/Rtx®-CLPesticides2 column pair in a dual column configuration.

**Figure 1** Perform dual column analysis using a single injector and guard column with split flow onto two analytical columns.



## Save Time and Money—Use One Column Pair for Multiple Methods

### Chlorinated Pesticide Analysis

Four different internal diameters (0.18, 0.25, 0.32, and 0.53mm ID) are suitable for chlorinated pesticides analysis, and all offer excellent chromatographic results (Figures 2 and 3). Baseline resolution is achieved for all primary EPA Method 8081B target compounds. Analysis times reflect simultaneous quantification and confirmation and range from 7 to 9 minutes, depending on column dimensions and instrument conditions.

### PCBs as Aroclors Analysis

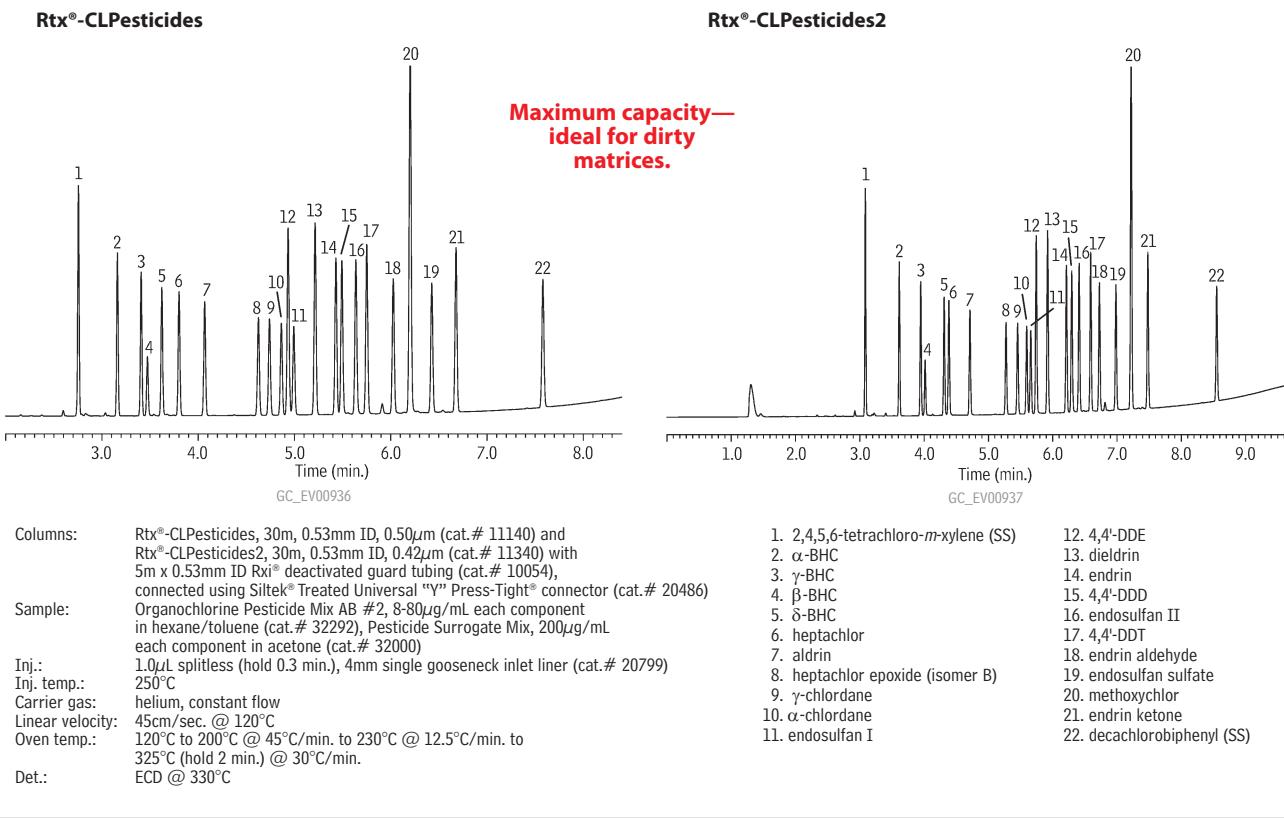
Aroclors can be identified and quantified on Rtx®-CLPesticides and Rtx®-CLPesticides2 columns using the same instrument set-up and run conditions used for chlorinated pesticides analysis. Aroclor patterns are easily distinguishable and the resolution obtained allows the analyst to select 5 peaks from each standard for quantification according to EPA Method 8082A. Figures 4 and 5 show the individual Aroclor mixes and the differences among them.

### Chlorinated Herbicides Analysis

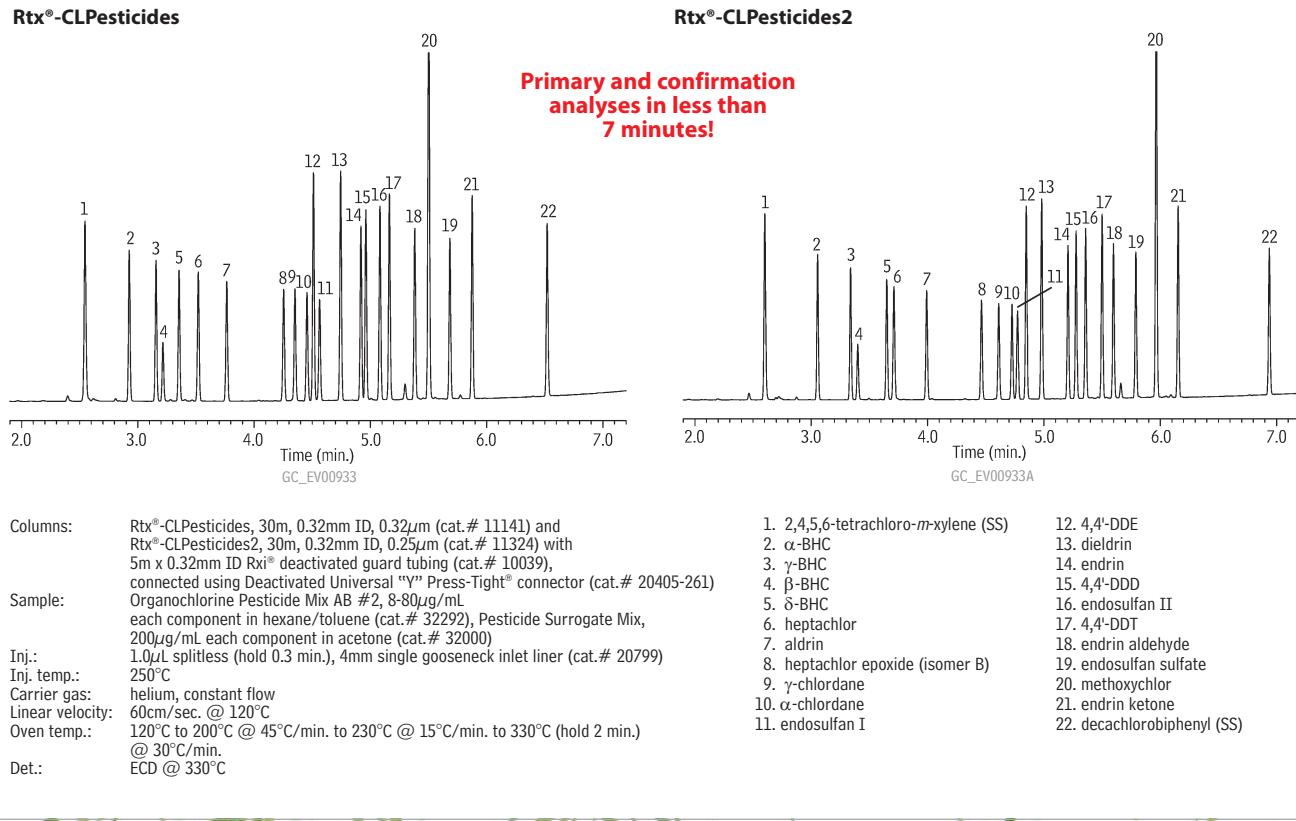
Chlorinated herbicide analysis can be accomplished in 13 minutes on the Rtx®-CLPesticides and Rtx®-CLPesticides2 column pair for all 17 target compounds in EPA Method 8151A. One pair of compounds (bentazon and picloram methyl esters) coelutes on the Rtx®-CLPesticides column, but these compounds are fully resolved on the Rtx®-CLPesticides2 column (Figure 6). Many laboratories acquire chlorinated herbicides on the same instrument set-up used for chlorinated pesticide and PCB analyses. However, regardless of the system you use, contamination from the sample preparation procedure for chlorinated herbicides can occur. Therefore, maintenance should be performed to clean the injection port and guard column after analyzing chlorinated herbicide samples.



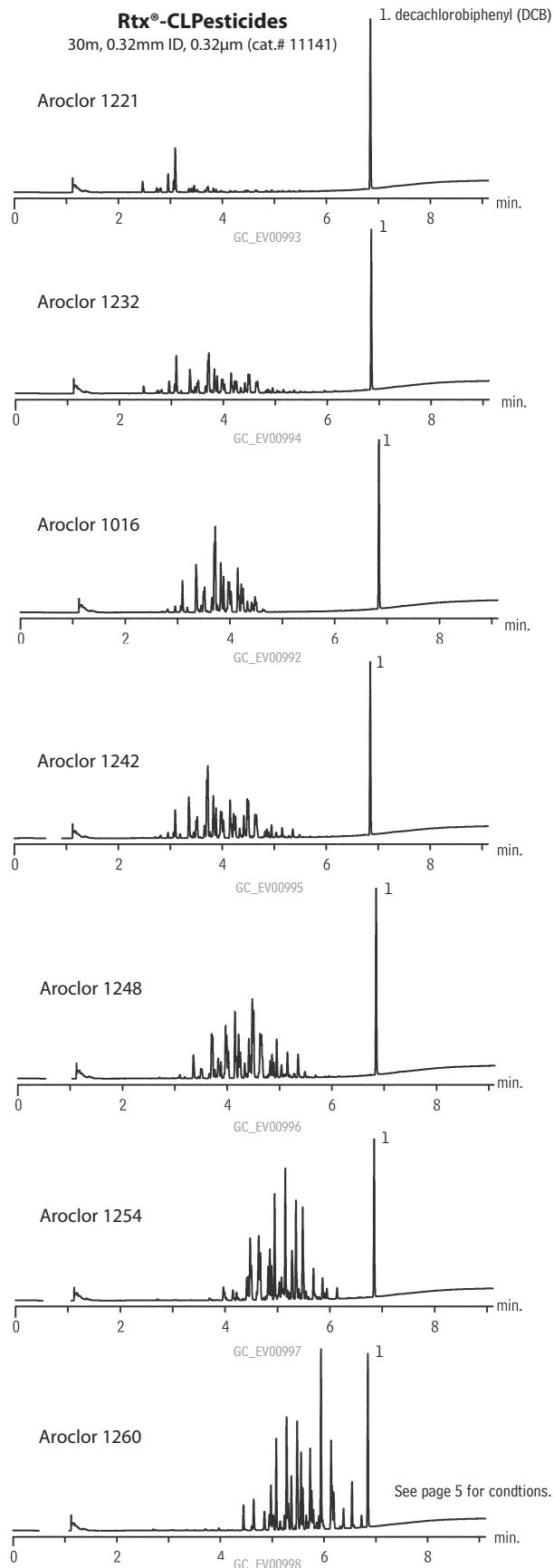
**Figure 2** Chlorinated pesticides resolved on the **0.53mm ID Rtx®-CLPesticides/ Rtx®-CLPesticides2 column pair.**



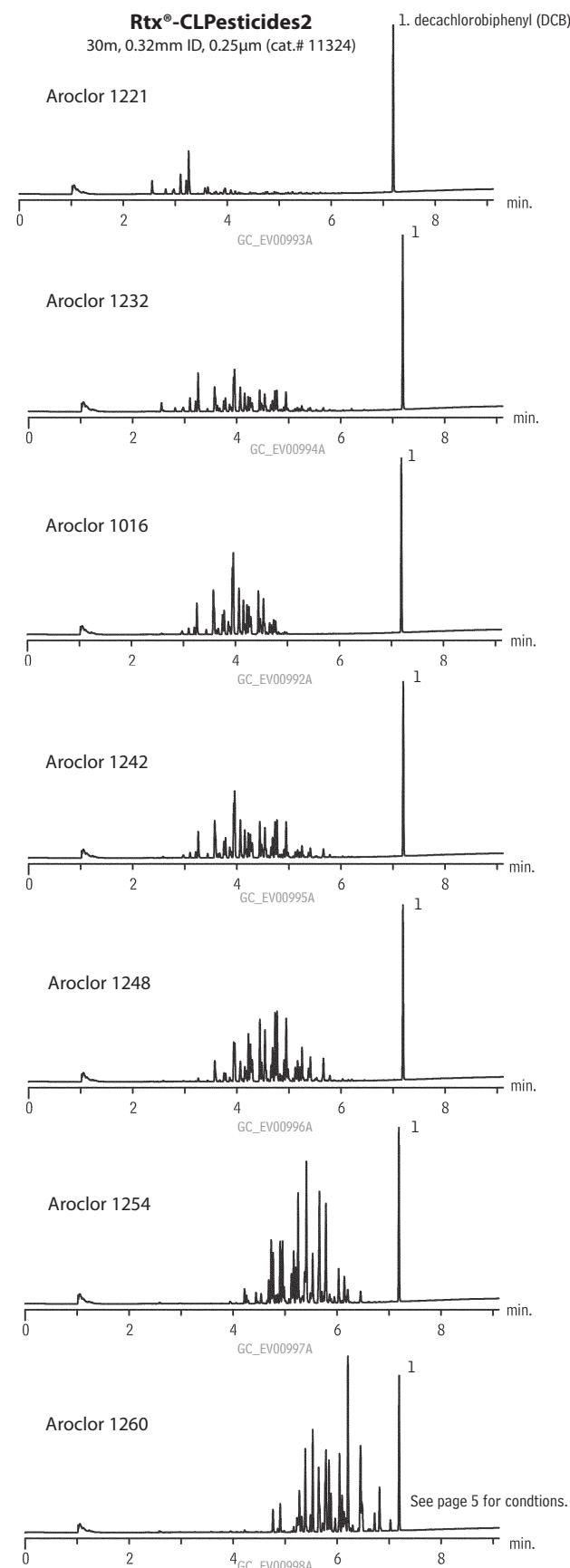
**Figure 3** Chlorinated pesticides resolved on the **0.32mm ID Rtx®-CLPesticides/ Rtx®-CLPesticides2 column pair.**



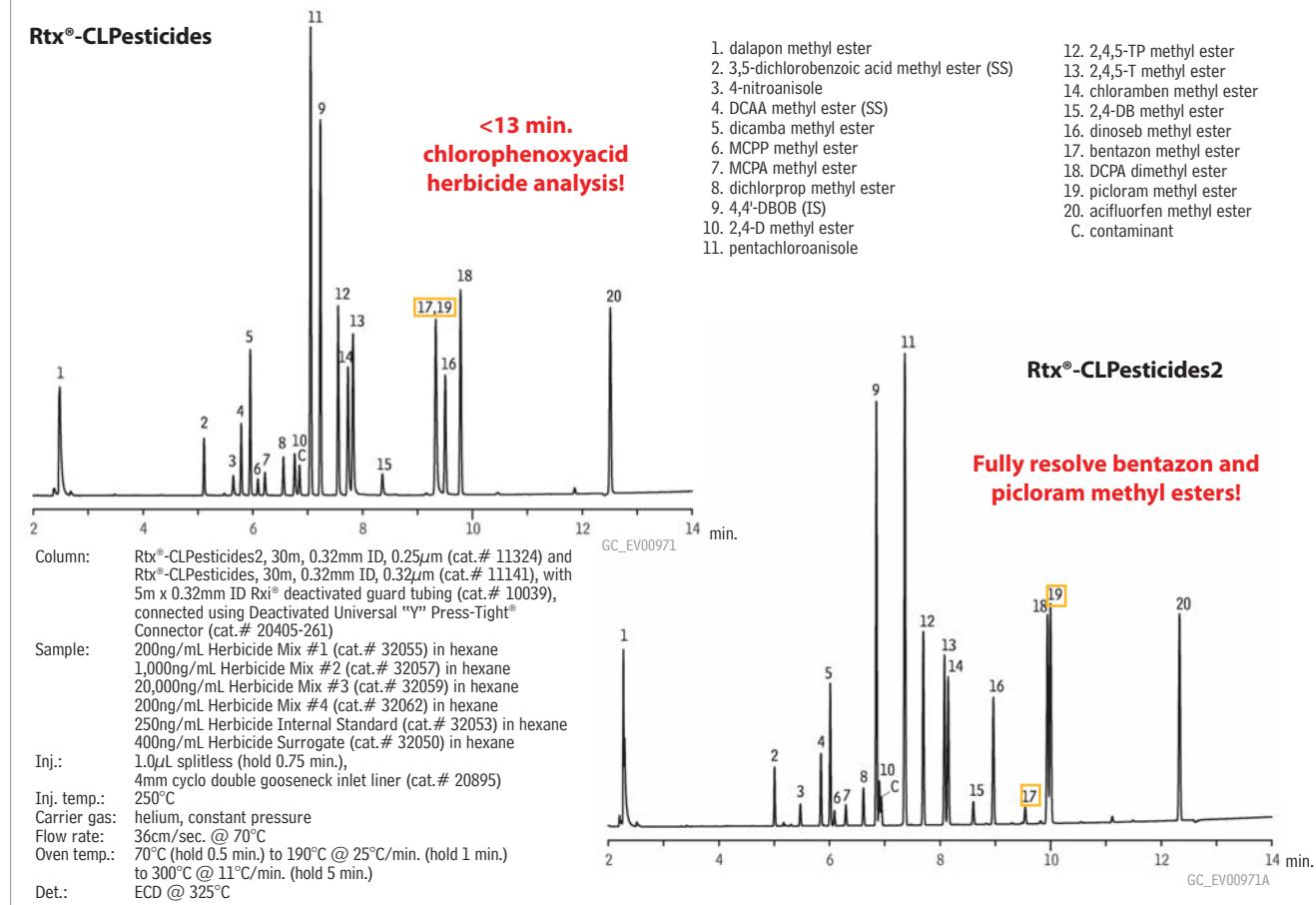
**Figure 4** Aroclor analysis on the Rtx®-CLPesticides column.



**Figure 5** Aroclor analysis on the Rtx®-CLPesticides2 column.



**Figure 6** Excellent resolution of chlorinated herbicides on the Rtx®-CLPesticides/ Rtx®-CLPesticides2 column pair.



**Conditions for Figures 4 & 5:** Sample: each Aroclor compound 1,000 $\mu$ g/mL in hexane (cat.# 32006) diluted to 1,000ppb, decachlorobiphenyl (BZ #209) 200 $\mu$ g/mL in acetone (cat.# 32029) diluted to 100ppb; Inj.: 1.0 $\mu$ L pulsed splitless @ 30psi (hold 0.3 min.), 4mm cyclo double gooseneck inlet liner (cat.# 20895); Inj. temp.: 250°C; Carrier gas: helium, constant flow; Linear velocity: 60cm/sec. @ 120°C; Oven temp.: 120°C to 200°C @ 45°C/min. to 230°C @ 15°C/min. to 330°C (hold 2 min.) @ 30°C/min.; Det.: ECD @ 330°C.

## Get Set for Dual Column Analysis!

### Analytical Columns

Improved resolution and faster analysis times, compared to 1701 or phenyl phases, make the Rtx®-CLPesticides/Rtx®-CLPesticides2 column pair ideal for analyzing chlorinated pesticides, PCBs as Aroclors, and chlorinated herbicides. These columns offer alternate selectivity, meeting method requirements for elution order changes and relative retention time shifts. Column bleed is extremely low at 330°C, allowing high boiling point contaminants to be baked off of the column, extending column lifetime.

**save now!**

See column kits on next page.

### Rtx®-CLPesticides Columns (fused silica)

ID	df ( $\mu$ m)	temp. limits	10-Meter	15-Meter	20-Meter	30-Meter	60-Meter
0.10mm	0.10	-60 to 310/330°C	43101				
0.18mm	0.18	-60 to 310/330°C	42101		42102		
0.25mm	0.25	-60 to 320/340°C		11120		11123	11126
0.32mm	0.32	-60 to 320/340°C				11141	
	0.50	-60 to 320/340°C		11136		11139	
0.53mm	0.50	-60 to 300/320°C		11137		11140	

### Rtx®-CLPesticides2 Columns (fused silica)

ID	df ( $\mu$ m)	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	
	0.50	-60 to 320/340°C				11325	
0.53mm	0.42	-60 to 300/320°C		11337		11340	

**restek**  
**innovation!**

- Very low bleed
- Faster analysis



### Rtx®-CLPesticides Column Kits

#### also available

Kits for 0.18 and 0.25mm ID columns are available. Visit [www.restek.com](http://www.restek.com) to inquire.

##### 0.32mm ID Rtx®-CLPesticides Kit      cat.# 11196 (kit),

###### Includes:

	cat.#
30m, 0.32mm ID, 0.32µm Rtx-CLPesticides Column	11141
30m, 0.32mm ID, 0.25µm Rtx-CLPesticides2 Column	11324
Universal Angled "Y" Press-Tight Connector	20403
5m, 0.32mm ID Siltek Guard Column	10027

##### 0.53mm ID Rtx®-CLPesticides Kit      cat.# 11197 (kit),

###### Includes:

	cat.#
30m, 0.53mm ID, 0.50µm Rtx-CLPesticides Column	11140
30m, 0.53mm ID, 0.42µm Rtx-CLPesticides2 Column	11340
Universal Angled "Y" Press-Tight Connector	20403
5m, 0.53mm ID IP Deactivated Guard Column	10045

## Injection Port Liners

The Rtx®-CLPesticides and Rtx®-CLPesticides2 column pair will work with both split or splitless injection techniques and any liner geometry. Various inlet liners are used for pesticide analysis; the four most common are the single gooseneck, double gooseneck, cyclo double gooseneck, and the Drilled Uniliner®.

The Drilled Uniliner® inlet liner provides the most inert sample pathway and eliminates injection port discrimination because the sample is funneled directly onto the column without contacting the metal injection port. Using a Drilled Uniliner® inlet liner eliminates the need to replace the inlet seal at the bottom of the injection port, substantially reducing maintenance time and expense.

The gooseneck liners are also commonly used for pesticide analysis and work best with pressure pulsing conditions. The best gooseneck liner to use with pressure pulsing is the cyclo double gooseneck liner. This liner has a screw-type sample pathway which collects nonvolatile material at the beginning of the screws and offers more surface area to vaporize the sample prior to reaching the entrance of the column.

## Guard Columns

The quality of guard column used for pesticide analysis is important because some compounds will degrade on columns that are not properly deactivated. The initial quality of the guard column is measured by inertness using endrin and 4,4'-DDT as test probes. There are two types of guard columns recommended for pesticide analysis: Rxi® guards and Siltek® treated guards. Both offer excellent inertness for pesticides; however, the Siltek® treated surface is slightly more inert than standard deactivation processes. Table I shows the excellent inertness offered by both the Rxi® guard and Siltek® treated tubing.

### Rxi® Guard/Retention Gap Columns

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.	10-Meter	10-Meter/6-pk.
0.25mm	0.37 ± 0.04mm	10029	10029-600	10059	10059-600
0.32mm	0.45 ± 0.04mm	10039	10039-600	10064	10064-600
0.53mm	0.69 ± 0.05mm	10054	10054-600	10073	10073-600

### Siltek®-Deactivated Guard /Retention Gap Columns/Transfer Lines

Nominal ID	Nominal OD	5-Meter	10-Meter
0.25mm	0.37 ± 0.04mm	10026	10036
0.32mm	0.45 ± 0.04mm	10027	10037

### Liners for Agilent GCs

ID* x OD & Length (mm)	qty.	cat.#
Gooseneck Splitless (4mm)		
4.0mm x 6.5mm x 78.5mm	5-pk.	20799
Double Gooseneck Splitless (4mm)		
4.0mm x 6.5mm x 78.5mm	5-pk.	20785
Cyclo Double Gooseneck (4mm)		
4.0mm x 6.5mm x 78.5mm	5-pk.	20896
Drilled Uniliner (hole near top)		
4.0mm x 6.3mm x 78.5mm	5-pk.	21055

### Dual Vespel® Ring Inlet Seals for Agilent GCs



#### 0.8mm ID Dual Vespel® Ring Inlet Seal

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

\*Cross disks are also available. Visit [www.restek.com](http://www.restek.com).

**Table I** Average percent breakdown of endrin and 4,4'-DDT.

Guard Type	% Breakdown	
	Endrin	4,4'-DDT
Rxi guard column	1.6	1.6
Siltek treated guard column	2.1	0.6
Rtx-CLPesticides column w/o guard	1.0	0.1

Analytical column: Rtx®-CLPesticides2 column (cat.# 11324) attached with a Press-Tight® connector. On-column concentration: endrin @ 50pg, 4,4'-DDT @ 100pg; Inlet liner: 4mm cyclo double gooseneck. n=10.

# Essentials for Dual Column Analysis

## Connectors

The best chromatography for dual column analysis is obtained using the Universal "Y" Press-Tight® connector. The internal design of the taper allows the column to seal to the glass surface and minimizes dead volume. To strengthen this connection, Restek developed the SeCure™ "Y" connector, which uses a C-clamp to hold the columns in place, assuring a reliable connection.

The MXT™-Union connectors are an alternative connector system that uses special ferrules designed to eliminate dead volume. The MXT™-Union is made of stainless steel and is deactivated with Siltek® treatment making an inert sample pathway. The special ferrules used to make the connection are designed to eliminate the dead volume when installing the columns.

### MXT™ "Y"-Union Connector Kits for Fused Silica Columns

Each kit contains the MXT™ union, three 1/32-inch nuts and three one-piece fused silica adaptors.

Description	qty.	cat.#
For 0.25mm ID Fused Silica Columns	kit	21389
For 0.32mm ID Fused Silica Columns	kit	21388
For 0.53mm ID Fused Silica Columns	kit	21387



### SeCure™ "Y" Connector Kits

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

Description	Ferrules Fit Column ID	qty.	cat.#
SeCure "Y" Connector Kit	0.18/0.25/0.28mm	kit	20276
SeCure "Y" Connector Kit	0.32mm	kit	20277
SeCure "Y" Connector Kit	0.45/0.53mm	kit	20278
Knurled nut		3-pk.	20279



The SeCure™ "Y" connector's open design allows visual confirmation of the seal.

### Graphite Ferrules for SeCure™ "Y" Connectors

Buy extra to keep spares on hand.

Ferrule ID	Fits Column ID	Graphite 10-pk.	Graphite 50-pk.
0.4mm	0.18/0.25/0.28mm	20200	20227
0.5mm	0.32mm	20201	20228
0.8mm	0.45/0.53mm	20202	20224



### Universal "Y" Press-Tight® Connectors

Great for dual column confirmation!

Description	ea.	3-pk.
Universal "Y" Press-Tight Connector	20405	20406
Deactivated Universal "Y" Press-Tight Connector	20405-261	20406-261
Siltek Treated Universal "Y" Press-Tight Connector	20485	20486



## Sample Preparation

### Resprep™ SPE Cartridges: Normal Phase

Hydrophilic (polar) adsorbents used to extract hydrophilic analytes from nonpolar matrices, such as organic solvents (e.g., polar contaminants from sample extracts).

	3mL/500mg (50-pk.)	6mL/500mg (30-pk.)	6mL/1000mg (30-pk.)	6mL/1000mg (100-pk.)
Florisil (EPA SW 846 methods and CLP protocols)	24031 24032*	— 26086**	24034 26085**	26205 —
Silica (EPA SW 846 methods)	24035 24036*	— —	24038 —	— —

\*Teflon® frits    \*\*Glass tubes with Teflon® frits



All cartridges are polypropylene and have polyethylene frits unless otherwise noted.



### CarboPrep™ SPE Cartridges

SPE Cartridge	Tube Volume, Bed Weight	qty.	cat.#
CarboPrep 90	3mL, 250mg	50-pk.	26091
CarboPrep 90	6mL, 500mg	30-pk.	26092
CarboPrep 200	3mL, 250mg	50-pk.	26088
CarboPrep 200	6mL, 500mg	30-pk.	26087

**Excellent for Pesticide Residue Cleanup!**



Small, compact unit—  
easy to hold and operate.

### Leak Detector Facts

#### Detectable gases:

helium, nitrogen, argon,  
carbon dioxide

#### Battery:

Rechargeable Ni-MH, 7.2-volt

#### Operating Temperature Range:

32°-120°F (0°-48°C)

#### Humidity Range:

0-97%

#### CE Approved

## Assure a Leak Free System!

### Restek Electronic Leak Detector

Improve your chromatography using a Restek Leak Detector! Gas leaks can cause detector noise, baseline instability, and shorter column lifetimes. Avoid these costly problems by using a Restek Leak Detector to reliably detect minute gas leaks.

Description	qty.	cat.#
Leak Detector with 110Volt Battery Charger	ea.	22451
Leak Detector with 220Volt European Battery Charger	ea.	22451-EUR
Leak Detector with 220Volt UK Battery Charger	ea.	22451-UK

## Analytical Reference Materials

### Organochlorine Pesticide Mix AB #1

(20 components)

aldrin	dieldrin
α-BHC	endosulfan I
β-BHC	endosulfan II
δ-BHC	endosulfan sulfate
γ-BHC (lindane)	endrin
α-chlordane	endrin aldehyde
γ-chlordane	endrin ketone
4,4'-DDD	heptachlor
4,4'-DDE	heptachlor epoxide (isomer B)
4,4'-DDT	methoxychlor
200µg/mL each in hexane:toluene (1:1), 1mL/ampul	
cat. # 32291	

### Organochlorine Pesticide Mix AB #2

(20 components)

aldrin	8µg/mL	endosulfan I	8
α-BHC	8	endosulfan II	16
β-BHC	8	endosulfan sulfate	16
δ-BHC	8	endrin	16
γ-BHC (lindane)	8	endrin aldehyde	16
α-chlordane	8	endrin ketone	16
γ-chlordane	8	heptachlor	8
4,4'-DDD	16	heptachlor epoxide	
4,4'-DDE	16	(isomer B)	8
4,4'-DDT	16	methoxychlor	
dieldrin	16		
In hexane:toluene (1:1), 1mL/ampul			
cat. # 32292			

### Organochlorine Pesticide System

#### Evaluation Mix

4,4'-DDT	200µg/mL	endrin 100µg/mL
In methyl <i>tert</i> -butyl ether, 1mL/ampul		
cat. # 32417		

### Pesticide Surrogate Mix

decachlorobiphenyl	2,4,5,6-tetrachloro- <i>m</i> -xylene
200µg/mL each in acetone, 1mL/ampul	
cat. # 32000	

### Organochlorine Pesticide Resolution Check Mix (with surrogates) (22 components)

aldrin	10µg/mL	endosulfan I	10
α-BHC	10	endosulfan II	20
β-BHC	10	endosulfan sulfate	20
δ-BHC	10	endrin	20
γ-BHC (lindane)	10	endrin aldehyde	20
α-chlordane	10	endrin ketone	20
γ-chlordane	10	heptachlor	10
decachlorobiphenyl (SUR)	20	heptachlor epoxide	
dieldrin	20	(isomer B)	10
4,4'-DDD	20	methoxychlor	100
4,4'-DDE	20	2,4,5,6-tetrachloro-	
4,4'-DDT	20	<i>m</i> -xylene (SUR)	10

In hexane:toluene, 1mL/ampul  
cat. # 32454

### Aroclor Solutions

Volume is 1mL/ampul. Concentration is µg/mL.

Compound	Solvent	Conc.	cat.#
Aroclor 1016	H	1,000	32006
Aroclor 1221	H	1,000	32007
Aroclor 1232	H	1,000	32008
Aroclor 1242	H	1,000	32009
Aroclor 1248	H	1,000	32010
Aroclor 1254	H	1,000	32011
Aroclor 1260	H	1,000	32012
Aroclor 1262	H	1,000	32409
Aroclor 1268	H	1,000	32410

H=hexane

### Decachlorobiphenyl (BZ #209)

decachlorobiphenyl	2,4,5,6-tetrachloro- <i>m</i> -xylene
200µg/mL in acetone, 1mL/ampul	
cat. # 32029	

Restek Trademarks:  
CarboPrep, MXT, Press-Tight,  
Resprep, Rtx, RxI, SeCure,  
Siltek, Uniliner.

Other Trademarks:  
Teflon, Vespel (E.I. du Pont de  
Nemours & Co., Inc.), Florisil  
(US Silica Co.).



Restek U.S. • 110 Benner Circle • Bellefonte, PA 16823 • 814-353-1300 • 800-356-1688 • fax: 814-353-1309 • [www.restek.com](http://www.restek.com)

Restek France • phone: 33 (0) 1 60 78 32 10 • fax: 33 (0) 1 60 78 70 90 • e-mail: [restek@restekfrance.fr](mailto:restek@restekfrance.fr)

Restek Ireland • phone: 44 2890 814576 • fax: 44 2890 814576 • e-mail: [restekeurope@aol.com](mailto:restekeurope@aol.com)

Thames Restek U.K. LTD • phone: 44 1494 563377 • fax: 44 1494 564990 • e-mail: [sales@thamesrestek.co.uk](mailto:sales@thamesrestek.co.uk)

Restek GmbH • phone: +49 (0) 6172 2797 0 • fax: +49 (0) 6172 2797 77 • e-mail: [info@restekgmbh.de](mailto:info@restekgmbh.de)

Lit. Cat.# EVFL1013-INT

© 2008 Restek Corporation.

