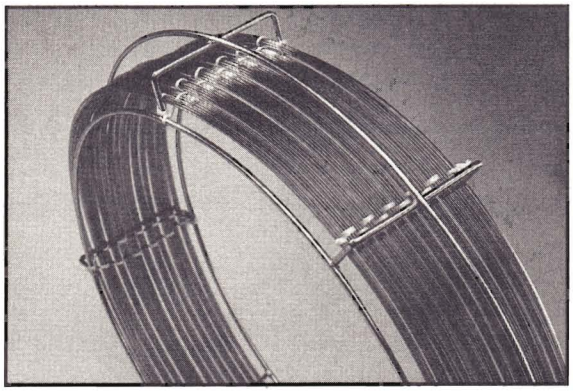
THE RESTEK ADVANTAGE

# NEW! Rtx®-5 Amine Capillary Columns

## *Analyze Amines and Other Strongly Basic Compounds*

- Eliminate the need to derivatize basic compounds.
- Minimal priming effects.
- For concentrations as low as 5ng on-column.



The reproducible analysis of basic compounds by capillary gas chromatography has always been difficult due to the presence of acidic silanol (SiOH) functional groups on the surface of fused silica tubing. Because common deactivation schemes do not completely remove or react with these silanols, the fused silica surface can remain acidic. When basic compounds, such as amines, are analyzed on an acidic surface, they are retained due to acid/base interactions. Chromatographically, this results in reduced response and severe peak tailing of the basic compounds.

The wizards at Restek have developed a new Rtx®-5 Amine column with unique deactivation technology to improve response and reduce tailing of basic compounds. Analyses that previously required derivitization or another analytical technique such as HPLC can now be performed on the Rtx®-5 Amine column. This column can also be used to analyze neutral compounds with the same efficiency as our standard Rtx®-5 columns.

We have developed a stringent quality assurance test mix containing several basic compounds such as pyridine, diethylenetriamine, diethanolamine, and 2,6-dimethylaniline. The on-column concentrations of these analytes range from 10-20ng. Specifications for the response and separation of these components ensure that each Rtx®-5 Amine column delivers consistent results.

### New Deactivation Technology for Analyzing Basic Compounds

The analysis of basic compounds is most commonly accomplished with Carbowax® (PEG) columns doped with a basic salt. Certain compounds, such as alkylamines and diamines can be successfully analyzed on base deactivated Carbowax® columns. Unfortunately, other high pKa compounds such as

## *in this issue...*

ethanolamines cannot. Also, the polar characteristics of the Carbowax® phase can often result in prolonged analysis time for many of these compounds.

Typical deactivation techniques used for less polar siloxane based stationary phases do not prevent the adsorption of amine-functional compounds. Figure 1a shows typical peak shapes for the analysis of ethanolamines on an Rtx®-5 capillary column. Note the reduced response and poor peak shape of the ethanolamines relative to an internal standard of equal concentration. Figure 1b shows the same compounds analyzed with the new Rtx®-5 Amine column. The peak height and shape are much improved compared to a standard Rtx®-5 column, indicating this new deactivation technology is effective for analyzing basic compounds.

### The Rtx®-5 Amine column analyzes alkylamines, diamines, triamines, and nitrogen-containing heterocycles

Alkylamines, diamines, triamines, and nitrogen-containing heterocycles are common analytes found in lubricants, cosmetic products, and used as fuel additives. The Rtx®-5 Amine column delivers consistent analytical results for a wide variety of amine compounds found in many commercial products. Figure 2 shows an industrial sample which contains several different types of ethyleneamine and piperazine derivatives.

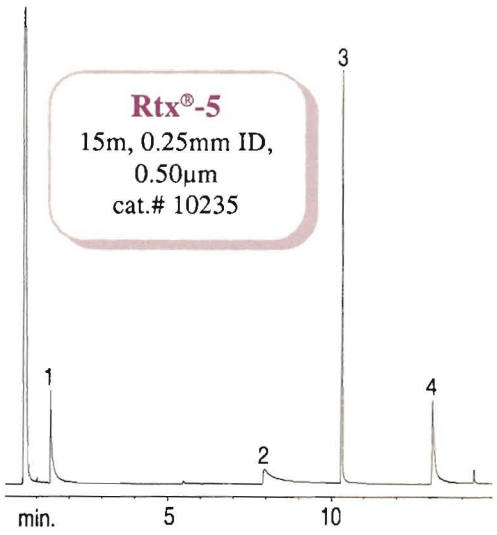
### Tired of derivatizing your basic drug analytes?

The Rtx®-5 Amine column can save time and money by eliminating the need to derivatize basic pharmaceutical compounds. Figure 3 shows the analysis of seven basic drugs in their salt or free base form. Our studies have found that the dissociation of salt forms is most efficient in a hot injection port (315°C) with a slow injection rate (3-5sec./µl). Accurate qualitative and quantitative analyses of underivatized drugs can be achieved with the Rtx®-5 Amine column.

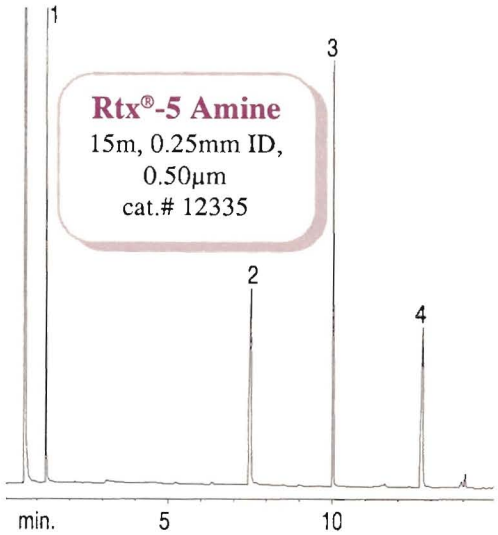
If your lab is analyzing amines or other strongly basic compounds, the new Rtx®-5 Amine columns give accurate and consistent results. These columns are available in 15 and 30 meter lengths; 0.25, 0.32, and 0.53mm IDs; and film thicknesses from 0.50 to 3.0µm. Each column is rigorously tested with a special mixture of amines to ensure column-to-column reproducibility.

**Figure 1** - The new Rtx®-5 Amine column shows improved response and peak shape for ethanolamines over standard Rtx®-5 columns.

**1a)**




**1b)**



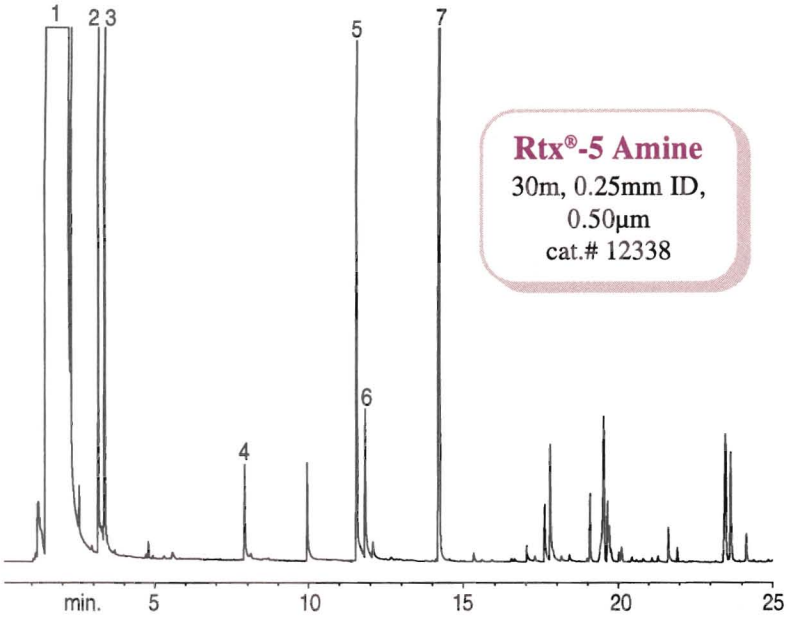

## Peak List and Run Conditions for Figures 1a and 1b

1. monoethanolamine
2. diethanolamine
3. triethylene glycolmonomethylether (IS)
4. triethanolamine

1.0µl split injection of ethanolamine mix in methanol, on-column concentration=34ng

| | |
|---|---|
| **Oven temp.:** | 50°C (hold 2 min.) to 180°C @ 10°C/min. (hold 2 min.) |
| **Inj./det. temp.:** | 280°C/300°C |
| **Carrier gas:** | hydrogen |
| **Linear velocity:** | 43cm/sec. set @ 50°C |
| **FID sensitivity:** | 6.4 x $10^{-11}$AFS |
| **Split ratio:** | 58:1 |

**Figure 2** - Obtain excellent peak symmetry and response of a wide range of industrial amines on the Rtx®-5 Amine column.




**Rtx®-5 Amine**
30m, 0.25mm ID,
0.50µm
cat.# 12338

ethyleneamines (and impurities) industrial sample:

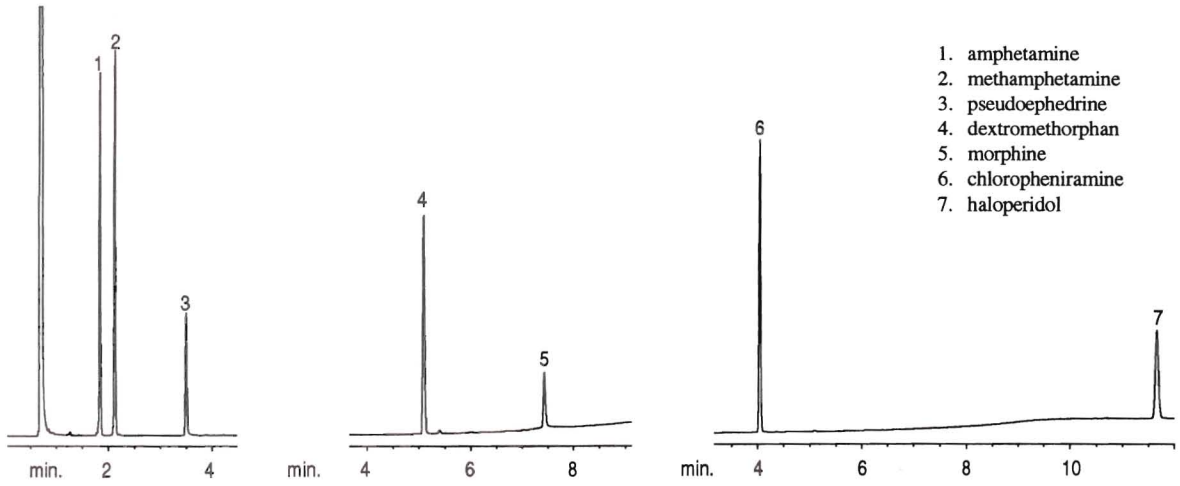
1. isopropanol
2. monoethanolamine
3. ethylenediamine
4. piperazine
5. diethylenetriamine
6. aminoethylethanolamine
7. aminoethylpiperazine

remaining impurities consist of ethyleneamine and piperazine derivatives

3.0µl split injection of Ethyleneamine Industrial Sample
concentration ~5-80ng on-column

| | |
|---|---|
| **Oven temp.:** | 40°C (hold 4 min.) to 315°C @ 10°C/min. (hold 5 min.) |
| **Inj./det. temp.:** | 315°C |
| **Carrier gas:** | hydrogen |
| **Linear velocity:** | 43cm/sec. set @ 40°C |
| **FID sensitivity:** | 6.4 x $10^{-11}$AFS |
| **Split ratio:** | 20:1 |

**Figure 3** - Eliminate the need for derivatization of basic drugs using the Rtx®-5 Amine column.




1. amphetamine
2. methamphetamine
3. pseudoephedrine
4. dextromethorphan
5. morphine
6. chloropheniramine
7. haloperidol

15m, 0.25mm ID, 1.0µm Rtx®-5 Amine column (cat.# 12350)
1.0µl split injection of basic drugs in methanol, on-column concentration=39ng

| | |
|---|---|
| **Oven temp.:** | 225°C to 315°C @ 10°C/min. (hold 10 min.) |
| **Inj./det. temp.:** | 315°C |
| **Carrier gas:** | hydrogen |
| **Linear velocity:** | 43cm/sec. set @ 225°C |
| **FID sensitivity:** | 6.4 x $10^{-11}$AFS |
| **Split ratio:** | 58:1 |


**New! Rtx®-5 Amine Columns**

| | df(µm) | 15-meter | 30-meter |
|---|---|---|---|
| **0.25mm ID** | 0.50 | 12335 | 12338 |
| | 1.00 | 12350 | 12353 |
| **0.32mm ID** | 1.00 | 12351 | 12354 |
| | 1.50 | 12366 | 12369 |
| **0.53mm ID** | 1.00 | 12352 | 12355 |
| | 3.00 | 12382 | 12385 |

# Analyzing Drinking Water Volatiles Listed in EPA Method 524.2, Rev. 4.0

In August 1992, EPA Method 524.2, Rev. 4.0 added 24 new compounds to the existing list of 60 volatile drinking water contaminants. The analytical procedures remained the same and include a purge and trap system to concentrate the volatile organics, a capillary gas chromatographic column to separate the components, and a mass spectrometer for the measurement and detection of the analytes.

Monitoring 84 compounds in a single analysis requires care in selecting the proper column and analysis conditions.[1] A 105 meter, 0.53mm ID, 3.0µm, Rtx®-502.2 column (cat.# 10910) was used for the separation. This long, thick film capillary column eliminates the need for sub-ambient cooling. The GC parameters were optimized using *ezGC™* method development software. The run conditions recommended by the software are 35°C for 8 minutes, then program to 220°C at 9°C/min. Using this temperature profile, the overall analysis time was reduced to 34 minutes. This fast analysis time is possible when using a mass spectrometer because it is not necessary to resolve all components. Even if coelutions occur, extracted ion profiles can be used to accurately identify and quantitate the individual compounds.

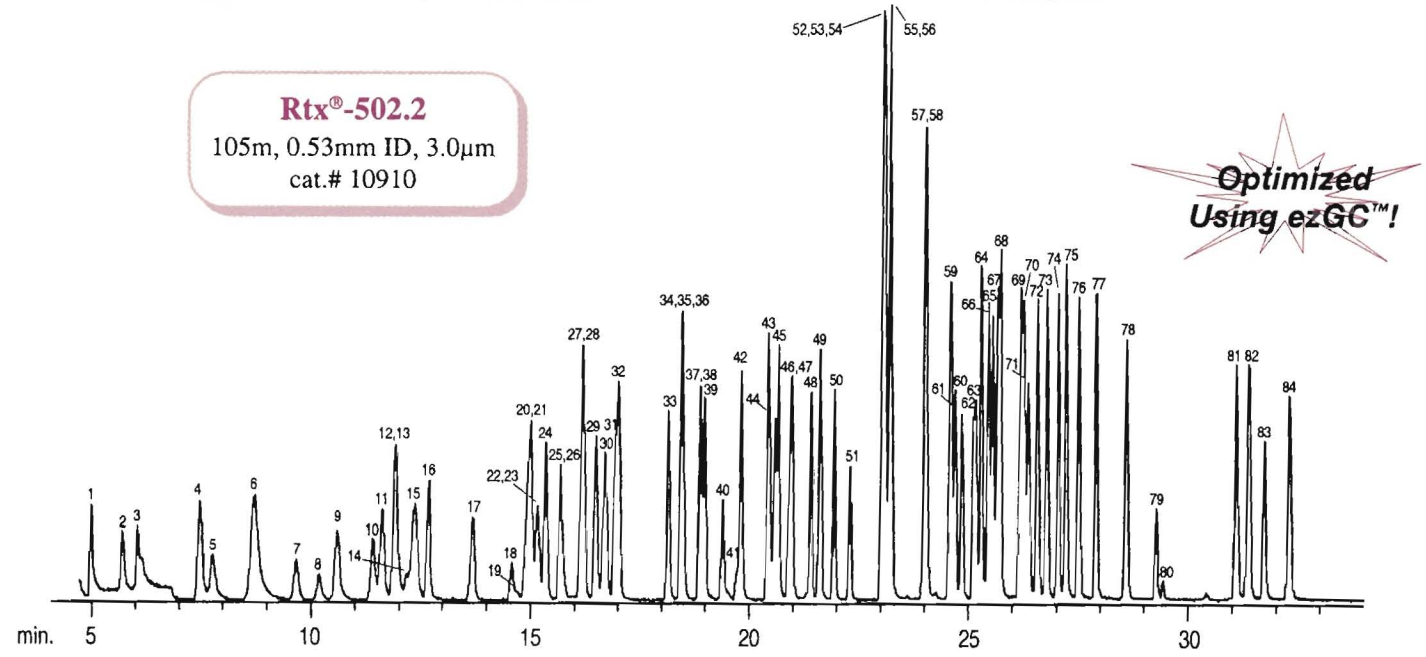
Figure 1 shows the analysis of the 84 component standard at a concentration of 100ppb in water. The standard was spiked into a 5ml purge vessel and purged for 11 minutes at a flow rate of 40ml/min. Once trapped, the sample was desorbed for 2 minutes at 250°C, with a desorb preheat temperature of 245°C. The desorb flow was set at 10ml/min. to transfer a narrow sample band to the capillary column. A low volume injector interface was used to connect the purge and trap transfer line to the GC. This interface prevents band broadening within the injection port. Before entering the vacuum system of the mass spectrometer, the column flow was split 10:1 using an open split interface. Alternatively, a jet separator can be used to reduce the carrier gas flow prior to entering the mass spectrometer. With the addition of 24 compounds, a scan range of 35 - 260 amu was required to obtain the primary quantitation ions of all 84 analytes. A solvent delay of 4.7 minutes was used to eliminate the $CO_2$ peak.

To assist in instrument calibration for EPA Method 524.2, Rev. 4.0, Restek now offers standards for the expanded target list. These standards are available from stock and include a convenient kit that covers the revised method. As with all Restek environmental mixtures, full data packs are available for each mixture to meet audit requirements.

EPA method 524.2, Rev. 4.0 has increased the total analyte list to 84 compounds. For the optimum analysis of these components, a 105 meter, Rtx®-502.2 capillary column is recommended. By using *ezGC™* method development software, run conditions were optimized to reduce analysis time to 34 minutes.

1. Munch, Jean W., Eichelberger, James W., *Journal of Chromatographic Science*, Vol. 30, Dec. 1992, pp 471-477.

**Figure 1** -All 84 components listed in EPA Method 524.2, Rev. 4.0 can be analyzed in 34 minutes.



# Standards for EPA Method 524.2, Rev. 4.0

For the 60 original compounds listed in EPA Method 524.2, Rev. 1.0, use Restek's 502.2 Calibration Mixes 1 through 6. For EPA Method 524.2, Rev. 4.0 compounds, add Restek's Calibration Mixes 7 & 8.

### *524 Calibration Mix #7*

2000µg/ml each in 1ml P & T methanol, 1ml per ampule.

| | |
|---|---|
| acetone | allyl chloride |
| acrylonitrile | 2-butanone |
| methyl acrylate | tetrahydrofuran |
| methyl methacrylate | 4-methyl-2-pentanone |
| ethyl methacrylate | 2-hexanone |
| pentachloroethane | nitrobenzene |

| | | |
|---|---|---|
| Cat.# | 30202 | each |
| | 30202-500 | each w/data pack |
| | 30202-510 | 5-pack |
| | 30202-520 | 5-pack w/data pack |
| | 30302 | 10-pack w/data pack |

### *524 Calibration Mix #8*

2000µg/ml each in 1ml P & T methanol, 1ml per ampule.

| | |
|---|---|
| diethyl ether | iodomethane |
| carbon disulfide | methyl tert-butyl ether |
| propionitrile | methacrylonitrile |
| 1-chlorobutane | chloroacetonitrile |
| 2-nitropropane | 1,1-dichloropropanone |
| trans-1,4-dichloro-2-butene | hexachloroethane |

| | | |
|---|---|---|
| Cat.# | 30203 | each |
| | 30203-500 | each w/data pack |
| | 30203-510 | 5-pack |
| | 30203-520 | 5-pack w/data pack |
| | 30303 | 10-pack w/data pack |

### *524 Internal Standard/Surrogate Mix*

2000µg/ml each in 1ml P & T methanol, 1ml per ampule.

| | |
|---|---|
| fluorobenzene | 1,2-dichlorobenzene-d4 |
| 4-bromofluorobenzene | |

| | | |
|---|---|---|
| Cat.# | 30201 | each |
| | 30201-500 | each w/data pack |
| | 30201-510 | 5-pack |
| | 30201-520 | 5-pack w/data pack |
| | 30301 | 10-pack w/data pack |

### *524 Revision 4.0 VOA Kit*

Contains 1ml each of catalog #'s:

- 524 Internal Standard/Surrogate Mix (cat.# 30201)
- 524 Calibration Mix #7 (cat.# 30202)
- 524 Calibration Mix #8 (cat.# 30203)
- 502.2 Calibration Mix #1 (cat.# 30042)
- 502.2 Calibration Mix #2 (cat.# 30043)
- 502.2 Calibration Mix #3 (cat.# 30044)
- 502.2 Calibration Mix #4 (cat.# 30045)
- 502.2 Calibration Mix #5 (cat.# 30046)
- 502.2 Calibration Mix #6 (cat.# 30047)

| | | |
|---|---|---|
| Cat.# | 30204 | each kit |
| | 30204-500 | each kit w/data packs |

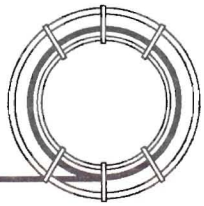
1. dichlorodifluoromethane
2. chloromethane
3. vinyl chloride
4. bromomethane
5. chloroethane
6. trichlorofluoromethane
7. diethyl ether
8. acetone
9. 1,1-dichloroethene
10. methyl iodide
11. allyl chloride
12. methylene chloride
13. carbon disulfide
14. acrylonitrile
15. MTBE
16. trans-1,2-dichloroethene
17. 1,1-dichloroethane
18. MEK
19. propionitrile
20. 2,2-dichloropropane
21. cis-1,2-dichloroethene
22. methacrylonitrile
23. methacrylate
24. chloroform
25. bromochloromethane
26. THF
27. 1,1,1-trichloroethane
28. 1-chlorobutane
29. 1,1-dichloropropene
30. carbon tetrachloride
31. 1,2-dichloroethane
32. benzene
33. trichloroethene
34. 1,2-dichloropropane
35. methyl methacrylate
36. chloroacetonitrile
37. bromodichloromethane
38. nitropropane
39. dibromomethane
40. MIBK
41. 1,1-dichloropropanone
42. cis-1,3-dichloropropene
43. toluene
44. ethyl methacrylate
45. trans-1,3-dichloropropene
46. 2-hexanone
47. 1,1,2-trichloroethane
48. 1,3-dichloropropane
49. tetrachloroethene
50. dibromochloromethane
51. 1,2-dibromoethane
52. chlorobenzene
53. 1,1,1,2-tetrachloroethane
54. ethylbenzene
55. *m*-xylene
56. *p*-xylene
57. *o*-xylene
58. styrene
59. isopropylbenzene
60. bromoform
61. 1,1,2,2-tetrachloroethane
62. 1,2,3-trichloropropane
63. trans-1,4-dichloro-2-butene
64. n-propylbenzene
65. bromobenzene
66. 1,3,5-trimethylbenzene
67. 2-chlorotoluene
68. 4-chlorotoluene
69. t-butylbenzene
70. 1,2,4-trimethylbenzene
71. pentachloroethane
72. sec-butylbenzene
73. p-isopropyltoluene
74. 1,3-dichlorobenzene
75. 1,4-dichlorobenzene
76. n-butylbenzene
77. 1,2-dichlorobenzene
78. hexachloroethane
79. 1,2-dibromo-3-chloropropane
80. nitrobenzene
81. 1,2,4-trichlorobenzene
82. hexachlorobutadiene
83. naphthalene
84. 1,2,3-trichlorobenzene

## Peak List and Run Conditions for Figure 1

105m, 0.53mm ID, 3.0µm Rtx®-502.2 (cat.# 10910)
HP 5971MSD, Tekmar LS-3000 concentrator with Tekmar Low Volume Interface
100ppb of 524 Revision 4.0 VOA Kit (cat.# 30204)

| | |
|---|---|
| **Oven temp.:** | 35°C (hold 8 min.) to 220°C @ 9°C/min. (hold 5 min.) |
| **Det. temp.:** | 285°C |
| **Carrier gas:** | helium |
| **Scan range:** | 35-260amu |
| **Trap type:** | VOCARB™ 3000 |
| **Purge time:** | 11 min. @ 40 ml/min. |
| **Desorb preheat temp.:** | 245°C |
| **Desorb temp.:** | 250°C |
| **Desorb time:** | 2 min. |
| **Desorb flow rate:** | 10 ml/min. |

# Hints for the Capillary Chromatographer



## Using Electron Capture Detectors

Electron capture detectors (ECDs) are common GC detectors used to analyze compounds with electronegative functional groups such as nitroaromatics, halogens, and oxygenates. Many of these compounds are frequently encountered in analyses including pesticides, polychlorinated biphenyls, lead containing compounds, and clinical/forensic samples.

ECDs are very sensitive detectors providing accurate responses in the picogram and femtogram range. They are considered selective detectors because their response is not uniform and is very dependent on the individual component's affinity for electrons. Therefore, a polyhalogenated compound will have a much greater response than a monohalogenated compound.

### Detector Operation

The ECD uses a radioactive source placed within the cell to emit beta particles. The carrier gas flows past the beta source and is ionized producing positive ions and a cloud of free electrons within the cell. These free electrons are captured by a positive electrode producing a stable background current which is amplified and used as a reference. When a sample component with an electron affinity enters the detector, it captures some of these electrons and decreases the current. This indicates the presence of the sample component within the detector. The ECD is the only common GC detector that uses a decrease in signal as a method of detection.

The $^{63}Ni$ pulsed detector is the most commonly used ECD. The $^{63}Ni$ foil contains a very small amount of radiation (usually less than 15 millicurie) and is sealed by the manufacturer inside the detector cell. Normally the GC manufacturer holds a general radiological license which covers their ECDs and a specific site license is not required by the NRC. To comply with NRC regulations, it is necessary to perform a radioactivity leak test every six months to verify the cell is not leaking radiation above the allowable limit. Arrangements can be made with the manufacturer or through an outside agency to obtain a leak test kit.*

### Detector Gases

For proper ECD operation, the detector make-up gas must be ionizable. Helium and hydrogen, the two most common carrier gases used for capillary chromatography, do not readily ionize and, therefore, are not recommended as an ECD make-up gas. Since capillary column carrier gas flow rates are low (typically < 10cc/min), an ionizable make-up gas can be used to produce the desired electron cloud. The make-up gas is added at high flow rates to produce a stable signal (Table I). The two most common make-up gases used with ECDs are nitrogen and 5% methane in argon ($Ar/CH_4$). Nitrogen gives better sensitivity than $Ar/CH_4$. However, $Ar/CH_4$ yields a greater dynamic range than nitrogen. Both nitrogen and $Ar/CH_4$ are not recommended as carrier gases with capillary columns and should only be used as make-up gases.

### Operating Hints

Because ECDs are extremely sensitive detectors, it is imperative the entire GC system be absolutely leak-free. Otherwise oxidation of the $^{63}Ni$ foil will occur and increased noise, baseline drift, and decreased lifetime of the detector will result. The best way to check the system for leaks is with a thermal conductivity leak detector (TCD) (cat.# 21605 or 20130). TCDs are recommended over liquid leak detectors for capillary chromatographic systems because they are very sensitive, easy to use, and there is no risk of contamination. Using a liquid leak detector like Snoop® can result in contamination of both the column and the detector if a leak is present. Even though the system is under positive pressure, liquid leak detectors can be drawn into the column at the leak point via the Venturi effect.

Moisture and oxygen traps are necessary for both the carrier gas and make-up gas or excessive detector noise will result. An indicating oxygen trap (cat.# 20624 or 20602) should be installed at the bulkhead inlet fitting to remove oxygen from

**Table I**
Operating Hints from Various Manufacturers

| | Radiation Source | Detector Insertion Distance | Make-up Flow Rate |
|---|---|---|---|
| HP 5890 | $^{63}Ni$ | 7.2cm (back of nut) | 50-60ml/min. |
| Varian 3300/3400, 3600, 3700 | $^{63}Ni$ | 13.2cm (back of nut)<br>11.5cm (back of nut)** | 20-30ml/min. |
| Shimadzu 9A, 14A, 17A | $^{63}Ni$ | 9.0cm (tip of ferrule) | 30-40ml/min. |
| PE Autosystem | $^{63}Ni$ | 6.5cm (back of nut) | 30ml/min. |

* Restek has been satisfied with the services of Detector Service Center (919)469-0259 and C.J. Bruyn & Co. (800)252-7896. Each wipe test costs approximately $20 to $25.
**metal column insertion distance

both the carrier gas and makeup gas lines. A molecular sieve trap (cat.# 20686) must be installed prior to the oxygen purifier to remove trace levels of water. Excessive noise and baseline instability will result if a molecular sieve trap is not used on an ECD, particularly if the GC does not come equipped with a small internal carrier/make-up gas line trap.* A hydrocarbon trap is not usually necessary since ECDs do not respond to hydrocarbon contamination. Also, be sure to use carrier gas and make-up gas regulators that are equipped with stainless steel diaphragms to avoid oxygen permeation.

Because ECDs are so sensitive, always precondition columns out of the detector. Install the column into the injector but not into the detector. Verify flow through the column and condition the column at the maximum test temperature for several hours, preferably overnight. Remember, the detector port must be capped to prevent air from oxidizing the $^{63}$Ni foil. Before removing or installing a column into the ECD, always cool the detector below 100°C to prevent oxidation of the $^{63}$Ni foil. *Never heat the ECD without a column installed or without capping the detector port!*

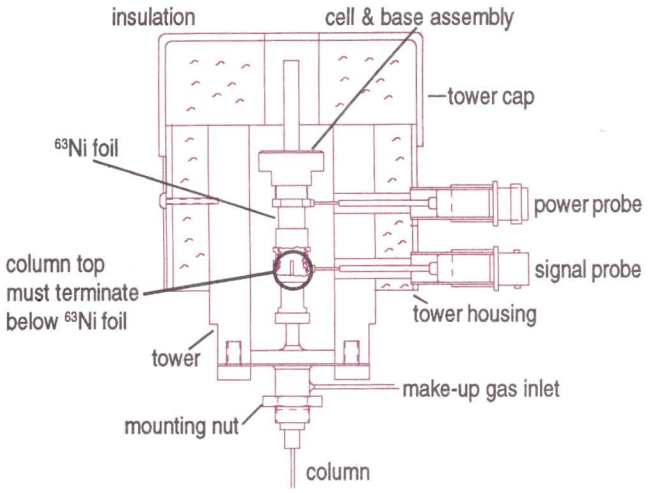
## Detector Maintenance

Baseline instability or a high background signal is often an indication of a contaminated ECD cell.** With HP ECDs, a signal greater than 50 (500 Hz) indicates a contaminated system. A signal greater than 10 indicates contamination in Varian ECDs. Often, contaminants deposited on the radioactive foil can be removed by heating. For routine maintenance, thermal cleaning is recommended. To thermally clean an ECD, first cool the detector below 100°C, remove the column and cap off the detector port. Next, establish a make-up gas flow of 50 to 60 ml/min. and set the oven temperature to 250°C. For HP GCs, heat the ECD to 350°C for 3 to 12 hours. If the background signal continues to be high (>60), the detector should be returned to HP for cleaning. Varian recommends heating their ECD to 400°C for 6-12 hours. Monitor the output signal. It should initially increase in magnitude, then decrease. When the signal has reached a stable plateau, the foil has been cleaned as much as possible. Varian also suggests thermally cleaning their ECDs using hydrogen as a purging agent.†

## Troubleshooting

Changes in sensitivity are sometimes related to the detector. A reduced response is often the result of an incorrect installation distance. It is critical that the column end terminates within the electron cloud located inside the cell. If the column end is installed too far into the detector and terminates above the cloud, or if the column is not installed far enough and terminates below the electron cloud, reduced response of sample components will be observed (Figure 1). Always install columns to the manufacturer's recommended insertion distance. Common detector insertion distances are shown in Table I.

**Figure 1** - Adhere to manufacturer's insertion distances or optimize distance based on response.




Make-up gas flow also has a great impact on sensitivity. If the make-up gas flow is set improperly reduced sensitivity can result. Regularly check make-up gas flow and adjust if necessary. Set make-up gas flows according to the manufacturer's instructions.

Another common problem associated with electron capture detectors is caused by frequent heating and cooling of the detector when making changes to the system. The base screws and/or Vespel®/graphite ferrules can loosen with temperature changes. Always make sure all connections to the ECD are leak-free to prevent oxygen influx.

Once electron capture detectors are set up properly configured, they require little optimization. Remember, the ECD is a concentration dependent detector. Therefore, carrier gas flow rates must be kept constant and leaks eliminated. Since ECDs are very sensitive, they are easily affected by contamination. Molecular sieve and oxygen traps must be placed on all gas lines and changed on a regular basis. Every effort should be taken to prevent foil contamination which can lead to reduced sensitivity.

**References:**


1. *Varian 3300/3400 Gas Chromatograph Operator's Manual*, Vol. 2, Varian Associates, Inc. 1990.
2. *HP 5890 Series II Reference Manual*, Edition 2, Hewlett-Packard, October 1989.
3. *Gas Chromatograph GC-14A Instruction Manual*, Shimadzu Corporation.
4. Buffington, Rosemary and Wilson, Michael K., *Detectors for Gas Chromatography - A Practical Primer*, Hewlett-Packard Co., Avondale, PA, 1991.
5. Hill, Herbert and McMinn, Dennis, ed., Detectors for Capillary Chromatography, John Wiley & Sons, New York, 1992.
6. *Perkin Elmer Auto System GC Operator's Manual*, Perkin Elmer Corp. June 1991.


*Often excessive ECD noise or baseline instability can be traced to a contaminated internal trap. Routine replacement is highly recommended.
**Negative peaks in the baseline indicate an $O_2$ leak present in the system. A positive baseline rise is often indicative of column or detector contamination.
†For more information on hydrogen cleaning, refer to the electron capture detector section of the Varian manual.

## NEW! Polar Deactivated Guard Columns

*Provide Optimum Wettability for Polar Compounds*

Chemists who are using polar analytical columns can now increase column lifetime and protect their expensive analytical column from harmful chemical damage. The life expectancy of a capillary column is greatly increased by using a 5-meter, deactivated, uncoated fused silica guard column. This prevents non-volatile contamination of the analytical column. Since the guard column is uncoated, sample components are allowed to enter the analytical column freely, while non-volatile contaminants are deposited in the guard column. Once contamination degrades performance, short lengths of the guard column can be removed. When the guard column is totally contaminated, replace it with a new one.

| Polar Deactivated Guard Columns | | |
|---|---|---|
| | 5 meter length | 10 meter length |
| ID | cat.# | cat.# |
| 0.25mm | 10065 | 10068 |
| 0.32mm | 10066 | 10069 |
| 0.53mm | 10067 | 10070 |

## SFC/CE Narrowbore Tubing

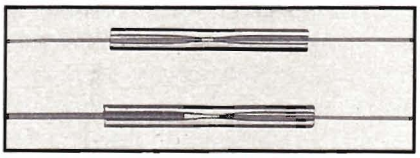
Restek now offers both untreated and deactivated narrow bore tubing for use in CE or as GC transfer lines, or as SFC restrictors. The IP deactivation process provides a phenyl methyl deactivated surface that gives optimum wettability and inertness for both non-polar and polar compounds. Deactivated tubing is available in continuous lengths up to 20 meters.

| SFC/CZE Narrowbore Tubing | | | |
|---|---|---|---|
| ID | nominal OD | untreated | IP deactivated |
| | | cat.# | cat.# |
| 0.025mm | .36 mm | 10091 | 10097 |
| 0.05mm | .36 mm | 10092 | 10098 |
| 0.075mm | .36 mm | 10093 | 10099 |
| 0.10mm | .36 mm | 10094 | 10100 |
| 0.15mm | .36 mm | 10095 | 10101 |
| 0.18mm | .36 mm | 10096 | 10102 |

## NEW! Deactivated, Universal Press-Tight® Connectors

- High temperature silanization for excellent inertness.
- Ideal for trace analysis of active compounds.
- Works with tubing ODs from 0.30 to 0.75mm (0.18 to 0.53mm ID)
- Available in economical 25 and 100 packs.

Restek's Universal Press-Tight® Connectors are made from rugged, highly inert fused silica and work with most common tubing diameters. However, if your application requires an extremely inert surface, we now offer connectors that have been treated with the same high temperature deactivation process used for our inlet sleeves. Ideal for the analysis of pesticides, semi-volatile pollutants, and clinical/forensic samples.



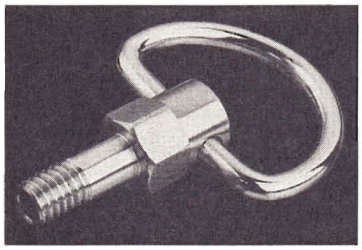
**Deactivated Press-Tight® Connectors**
cat.# 20429, 5-pk.
cat.# 20430, 25-pk.
cat.# 20431, 100-pk.

# We're Nuts About HP!

*Restek offers several uniquely designed fittings to fit HP GCs*

## Finger Tight Nut for HP 5890 GCs

- Rapidly tighten columns without wrenches.
- Avoid stripped threads from over-tightening.
- Two versions available, one for use with HP short ferrules and another for standard graphite ferrules.
- Both versions can be used with 0.25, 0.32, or 0.53mm ID columns.
- Wrench pad for use with Vespel®/graphite ferrules.
- 316 stainless steel body.
- Similar to HP part# 5020-8293 & 5020-8292 except Restek's can be used with Vespel® ferrules.

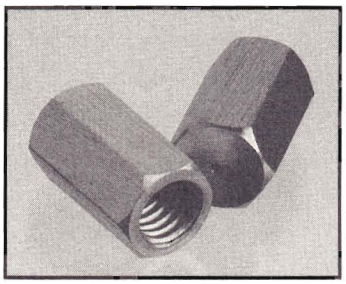


**Finger Tight Nut*:**
**Standard Swagelok®-type ferrules:**
cat.# 21312, each

**HP-type "short" ferrules:**
cat.# 21311, each

**purchase ferrules separately*

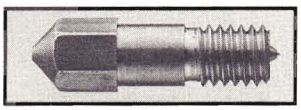
## MSD Source Nut



The MSD source nut bore has been changed from 0.8mm to 1.2mm to permit easy removal of stuck ferrules with a standard tapered needle file (cat.# 20106). The nuts still match the specifications of the manufacturer's original part and are made of brass to prevent thread stripping on the transfer line. Similar to HP part # 05988-20066.

**MSD Source Nut:** cat.# 20643, 2-pk.

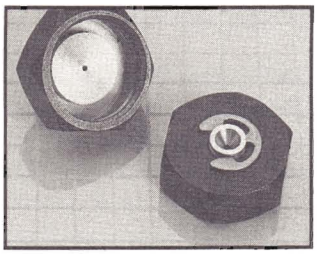
## Detector Plug Nut for HP GCs



Need to cap off or thermally clean a dirty detector? Need to check detector or make-up gas flow rates? Want to prevent $H_2$ from accidentally diffusing into the oven from an unused detector base? Use Restek's brass Plug Nut for HP GCs. Similar to HP part # 5020-8294.

**Detector Plug Nut:** cat.# 21883, 2-pk.

## Septum Nut for HP 5890 GCs (Autosampler Injections)



Ensure a leak-free injection port by using Restek's septum nut. This new high quality stainless steel nut is similar to HP part # 18740-60835. The thread design and needle guide allow easy penetration and prevent premature septum coring.

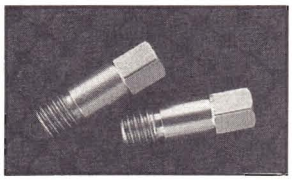
**Septum Nut:** cat.# 20631, each

## Needle Guide Septum Nut for HP 5890 GCs (Manual Injections)

Increase septa lifetime and decrease maintenance requirements with Restek's septum nut for 26 gauge needle. This new septum nut directs the needle through the same hole, minimizing coring and leakage. For additional details on how our Needle Guide Septum Nut can extend septa lifetime, request Restek's technical tip, "Extending Septa Life", from *The Restek Advantage*, Vol. 4 No. 6. This nut is similar to HP part# 18740-60835 except with a 26 gauge hole.

**Needle Guide Septum Nut:** cat.# 21309, each

## Stainless Steel Capillary Nuts for HP GCs



Restek now offers two stainless steel nuts for HP 5890 GCs. One version incorporates a deeper recess that allows the use of longer-based, Swagelok®-type graphite or Vespel®/graphite capillary ferrules. The other version has a shallow recess for use with shorter HP-type ferrules. This version is similar to HP part# 05921-21170.

**Recessed Capillary Nut:** cat.# 20883, 2-pk.
**Standard Capillary Nut:** cat.# 21884, 2-pk.

**For more information on Restek's Inlet Supplies for HP GCs, please call your local distributor.**

# 0.32mm ID Rt-Alumina™ PLOT Columns

## *For fast and efficient analysis of light hydrocarbons*

Rt-Alumina™ PLOT columns have proven to be fast and reliable for the analysis of light hydrocarbons. Now, the Restek wizards have developed a 0.32mm ID Rt-Alumina™ PLOT column which improves efficiency and reduces analysis time for many applications.

The 0.32mm ID and 0.53mm ID Rt-Alumina™ PLOT columns exhibit the same unique selectivity, but the 0.32mm ID offers faster analysis times. Figure 1 shows the comparison of a 50 meter, 0.53mm ID and a 60 meter, 0.32mm ID Rt-Alumina™ PLOT column. Both columns achieve baseline separation of all 17 saturated and unsaturated hydrocarbons. The thinner alumina layer on the 0.32mm ID column results in a significant savings in analysis time. Even though the 0.32mm ID column is 10 meters longer, the analysis time is only 10 minutes, compared to 16 minutes with the 0.53mm ID column.

In addition to shorter analysis time, the 0.32mm ID offers improved resolution due to the higher plate count. The 0.32mm ID columns exhibit over 1600 plates/meter, compared to ~1000 plates/meter for the 0.53mm ID columns. The big advantage of the 0.53mm ID column is capacity. Since the 0.53mm ID column has a thicker alumina layer, its capacity is higher than the 0.32mm ID column. For trace level analysis, capacity may not be an important consideration. However, for purity determinations, the increased capacity of the 0.53mm ID columns can prevent column overload.

### Effects of Water on the Rt-Alumina™ PLOT Column

The selectivity of the Rt-Alumina™ PLOT column can be affected by water contamination. This will cause changes in elution and retention of some components. By conditioning the column for 8 hours at 200°C with carrier gas, water can be removed and the proper selectivity restored.

### Column Reproducibility is Guaranteed

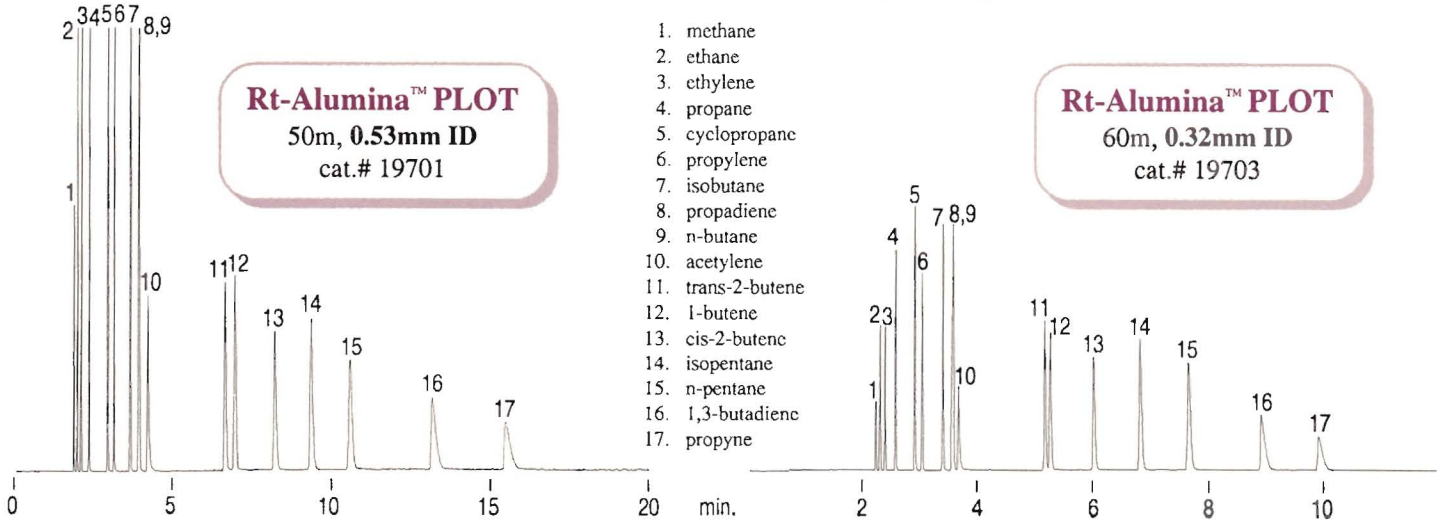
The chromatograms shown in Figure 1 are examples of the test mix used to evaluate every Rt-Alumina™ PLOT column. Pentane is used to calculate k (capacity factor) which verifies proper film thickness. A ratio of isobutane to acetylene retention is used to monitor the deactivation of the Alumina Oxide layer. The number of plates per meter is also calculated to determine column efficiency. Finally, the retention indices for acetylene and 1-butene are used to verify the selectivity of the $Al_2O_3$ phase. Every Rt-Alumina™ PLOT column is guaranteed to meet stringent QA specifications.

Restek's Rt-Alumina™ PLOT columns offer a choice for fast and reproducible analysis of hydrocarbon streams or purity analysis. With the introduction of 0.32mm ID Rt-Alumina™ PLOT columns, analysts have the option of enhanced efficiency and faster analysis times for light hydrocarbon separations.

### Rt-Alumina™ PLOT Columns

| | |
|---|---|
| 60m, 0.32mm ID | Cat.# 19703 |
| 30m, 0.32mm ID | Cat.# 19702 |
| 50m, 0.53mm ID | Cat.# 19701 |
| 30m, 0.53mm ID | Cat.# 19700 |

**Figure 1** - The 0.32mm ID Rt-Alumina™ PLOT column exhibits a faster analysis time than the 0.53mm ID column for a mixture of C1 to C5 hydrocarbons.




50µl split injection
**Oven temp.:** 80°C isothermal
**Inj./det. temp.:** 200°C
**Carrier gas:** helium
**Linear velocity:** 42cm/sec. set @ 80°C (5.6ml/min.)
**Split flow:** 40ml/min.
**FID sensitivity:** 1.28 x $10^{-10}$AFS

30µl split injection
**Oven temp.:** 80°C isothermal
**Inj./det. temp.:** 200°C
**Carrier gas:** helium
**Linear velocity:** 46cm/sec. set @ 80°C (2.4ml/min.)
**Split flow:** 60ml/min.
**FID sensitivity:** 1.28 x $10^{-10}$AFS

# Optimizing the Analysis of Chlorophenoxy Herbicides

Chlorine substituted phenoxyacetic acids, such as 2,4-D, MCPA, and 2,4,5-T, were introduced as selective weed killers in the 1940's. Due to their growth-regulating and herbicidal activities against broadleaf weeds, they have been commonly used for weed control on cereal crops, grasslands, and lawns. 2,4-D and 2,4,5-T were also the primary defoliant agents used in Agent Orange in Vietnam. Today, chlorophenoxy herbicides are still used as commercially available lawn weed killers.[1]

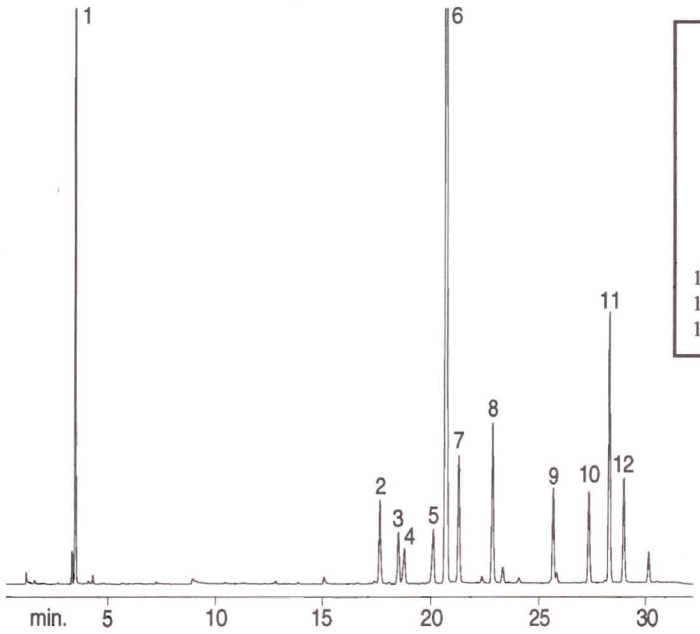
Chlorophenoxy herbicides are applied as either esters or salts which are easily metabolized by plants. The esters are oil soluble, but can also be applied as emulsions in water. The salts are typically highly soluble in water and are used as aqueous concentrates. Because the chlorophenoxy herbicides are spread on top of the soil or grass and then leach into the ground, there is great potential for ground water contamination. Chlorophenoxy herbicides readily degrade in the environment and, for many years, were not considered an environmental or public concern. However, potential hazards to public health and environmental quality led to the development of methods for the analysis of these herbicides. US EPA Methods 615 (municipal/industrial waste water), and 8150 (solid waste) were developed to monitor chlorophenoxy herbicides in environmental samples.[2,3]

Difficulties exist in the analysis of chlorophenoxy herbicides by gas chromatography. In their free acid form, these herbicides have limited volatility and are prone to irreversible adsorption. Therefore, they are most frequently analyzed as methyl esters. Since these herbicides can be applied as several different types of esters or as a salt, they must first be converted to the free acid form, then derivatized into methyl esters for analysis by gas chromatography. Methylation increases herbicide volatility and overcomes matrix interferences of herbicides extracted from soil. Despite this derivatization step, problems such as poor resolution, matrix interference, and peak misidentification are still experienced by analysts.

Using *Pro ezGC™*, computer modeling software, the analysis of chlorophenoxy herbicides was optimized with 30m, 0.53mm ID, 0.5µm Rtx®-35 (cat.# 10440) and Rtx®-5 (cat.# 10240) columns. Figures 1 and 2 show chromatograms of 10 derivatized chlorophenoxy herbicides listed in EPA Methods 615 and 8150B. All compounds are baseline resolved on the Rtx®-35 and the analysis is complete in 30 minutes. The Rtx®-35/Rtx®-5 dual column confirmational system produces a different elution pattern on each phase, offering a fast, positive peak identification.

Another important consideration when analyzing environmental samples for chlorophenoxy herbicide contamination is accurate instrument calibration. The key to accurate calibration is high quality chemical standards. Restek offers chlorophenoxy herbicide calibration, internal, and surrogate standards that can be used for EPA Methods 515.1, 615 and 8150. Each mixture is available in either the free acid form or the methyl ester form. The free acid mixtures can be used to verify analyte recovery or derivatization procedures. The derivatized mixtures can be used for instrument calibration. Compounds are separated into mixtures based on their

**Figure 1** - The Rtx®-35 gives baseline resolution for all 12 chlorophenoxy herbicides.




1. dalapon
2. DCAA (S)
3. dicamba
4. MCPP
5. MCPA
6. DBOB (IS)
7. dichloroprop
8. 2,4-D
9. 2,4,5-TP
10. 2,4,5-T
11. dinoseb
12. 2,4-DB

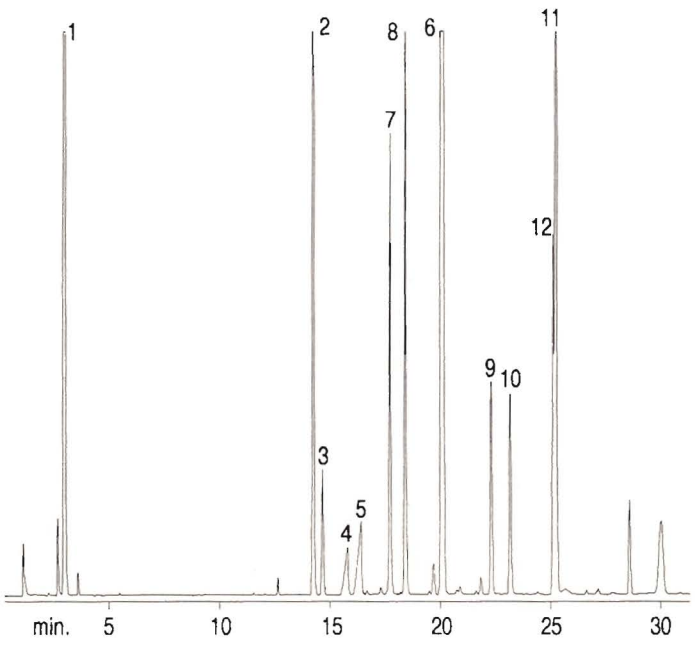
30m, 0.53mm ID, 0.50µm **Rtx®-35** (cat.# 10440)

0.5µl direct injection of Chlorophenoxy Herbicides, on-column concentration=10-10,000µg/ml

| | |
|---|---|
| **Oven temp.:** | 60°C to 150°C @ 8°C/min. (hold 5 min.), then to 210°C @4°C/min. |
| **Inj./det. temp.:** | 250°C/275°C |
| **Carrier gas:** | helium |
| **Linear velocity:** | 35cm/sec. set @ 60°C |
| **ECD sensitivity:** | 60kHz FS |

Optimized Using Pro ezGC™

**Figure 2** - Use the Rtx®-5 for confirmation of peak identification.




30m, 0.53mm ID, 0.50µm **Rtx®-5** (cat.# 10240)

0.5µl direct injection of Chlorophenoxy Herbicides,
on-column concentration=10-10,000µg/ml

**Oven temp.:** 60°C to 150°C @ 8°C/min. (hold 5 min.), then to 210°C @ 4°C/min.
**Inj./det. temp.:** 250°C/275°C
**Carrier gas:** helium
**Linear velocity:** 35cm/sec. set @ 60°C
**ECD sensitivity:** 160kHz FS
***Please see Figure 1 for peak list.***

*Optimized Using Pro ezGC™!*

response to allow labs to custom tailor their working calibration standards. As with all Restek environmental standards, these herbicides are thoroughly tested for purity and quantitative accuracy. All raw materials are tested by several analytical techniques including DSC (Differential Scanning Calorimetry), GC-FID, and GC/MS. The final product mixtures are tested in replicate using GC-FID.

The continued use of herbicides increases concerns of contamination. Laboratories must be able to determine the presence or absence of herbicide contamination in environmental samples. Capillary columns such as the Rtx®-35 and Rtx®-5 improve the quality of chlorophenoxy herbicides analysis by offering high resolution, inertness, and reproducible results. In addition to these columns, chemical standards are available for EPA methods 515.1, 615 and 8150B. Together, these products enhance the analysis of chlorophenoxy herbicides.

**References**

1. Kaufman, D.D., Kearney, P.C., Herbicides, Vol.1, *Chemistry, Degradation, and Mode of Action*, 2nd ed., Marcel Dekker, Inc., New York and Basel.
2. USEPA, *Methods for the Organic Chemical Analysis of Municipal and Industrial Wastewater: Method 615*, "Determination of Chlorinated Herbicides in Industrial and Municipal Wastewater".
3. USEPA, *SW-846 Test Methods for Evaluating Solid Waste*, 3rd Edition, Final Update I; Method 8150A, "Chlorinated Herbicides by Gas Chromatography".