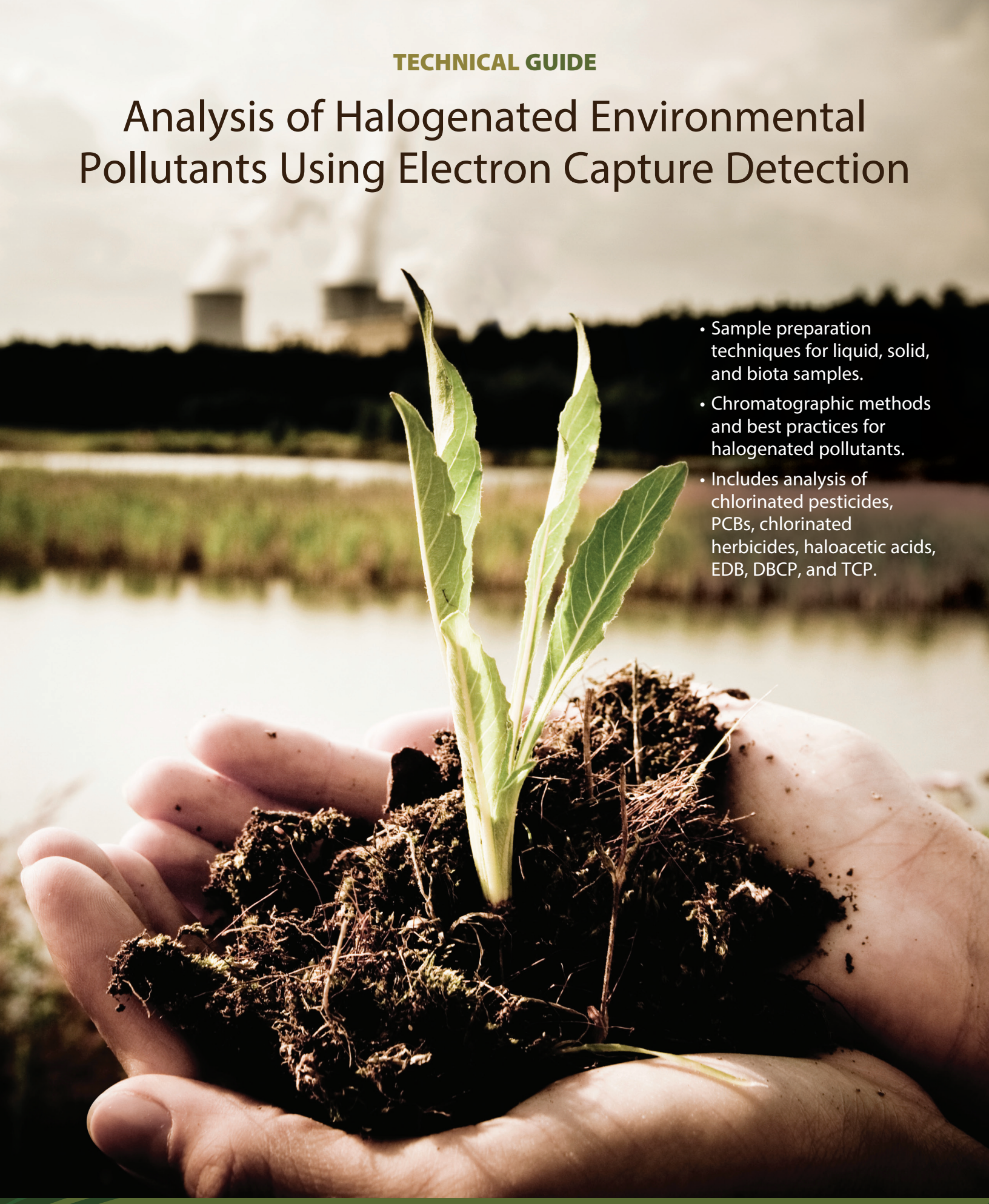
TECHNICAL GUIDE

Analysis of Halogenated Environmental Pollutants Using Electron Capture Detection



- Sample preparation techniques for liquid, solid, and biota samples.
- Chromatographic methods and best practices for halogenated pollutants.
- Includes analysis of chlorinated pesticides, PCBs, chlorinated herbicides, haloacetic acids, EDB, DBCP, and TCP.

Overview

This guide discusses analytical methods for monitoring halogenated pollutants in a wide variety of matrices including water, solids (soils and plant materials), and biota (fish and animal tissue). Classes of compounds such as chlorinated pesticides, polychlorinated biphenyls (PCBs), chlorinated herbicides, disinfection byproducts, and fumigants are among the pollutants addressed here. Gas chromatography (GC) combined with electron capture detection (ECD) is a common technique used for analyzing these compounds. ECD provides high sensitivity to these electronegative compounds, which results in extremely low detection limits. To ensure accurate identification, each sample can be analyzed simultaneously using a dual ECD instrument and two columns of differing selectivity for confirmational analysis.

Extremely low detection limits, combined with interferences from sample matrices, present an analytical challenge for laboratories involved in chlorinated pesticide and herbicide analyses. Samples are often highly contaminated with non-target compounds such as lipids and hydrocarbons, and the methods require rigorous quality control protocols. In addition, some of these compounds can break down or be adsorbed during the analytical procedure leading to inaccurate results.

Using Restek's Rtx®-CLPesticides column set, coupled with the methods and recommendations presented in this guide, will make your analyses easier and allow you to perform other environmental testing, such as PCB analysis, using the same columns and GC setup.

This guide offers information on the extraction, cleanup, and GC analysis of chlorinated pesticides, PCBs, herbicides, disinfection byproducts, and fumigants from a wide range of sample matrices. Also included are listings of products to simplify the analysis of these compounds.

Sample Preparation Methods for Liquid, Solid, and Biota Samples

Proper extraction procedures must be followed in order to ensure reproducible quantitative transfer of target analytes from the sample matrix. However, the wide variety of sample matrices combined with the extremely low levels of compounds typically found in environmental samples presents a challenge when extracting samples. Basic instructions for the most common techniques for liquid, solid, and biota samples are presented here. In some cases, the pH of the sample may need to be adjusted to ensure that the compounds of interest are extracted from the sample matrix. Specific methods, such as the U.S. Environmental Protection Agency (EPA) methods referred to in this guide, can be consulted for details on solvent type and pH adjustments.

Sample Preparation Products

See **pages 25–27** or visit **www.restek.com**

Liquid Samples

Separatory funnel extraction (EPA Method 3510) or automated liquid-liquid extraction (EPA Method 3520) are common extraction techniques for liquid samples. Extractions using separatory funnels are less time consuming and less expensive to set up, but have issues with samples that tend to form emulsions with the extraction solvent. Automated liquid-liquid extractors are less labor intensive and present fewer issues with emulsion-forming samples, but are more expensive to set up and can be more difficult to clean. Solid phase extraction (EPA Method 3535) can be used to extract pesticide compounds from aqueous samples that have low particulate levels and minimal matrix interferences, such as drinking water samples.

Separatory Funnel Extraction

The typical aqueous sample volume is 1-liter and a 2-liter separatory funnel is used for extraction (Figure 1). The sample pH is measured and adjusted to conform to the method requirements. A specified volume of method-recommended solvent is added to the sample and shaken for two minutes. The separatory funnel should be vented periodically during shaking to prevent pressure build-up. It is critical to shake all samples in the same manner to minimize variations in extraction efficiency. The best way to ensure consistency is to use a mechanical separatory funnel shaker. The extraction step is repeated two more times with fresh aliquots of solvent to achieve quantitative recovery of all analytes. Extracts from all three separations are combined into the same collection vessel.

Continuous Liquid-Liquid Extraction

Continuous liquid-liquid extraction systems offer the convenience of unattended extraction, as well as significant advantages for samples that form emulsions. However, proper glassware washing procedures are critical because, due to the extended contact time of the organic compounds with the glass surfaces, reactive compounds can break down if these surfaces become contaminated. Liquid-liquid extractors are available in two versions: conventional and accelerated. The accelerated type uses a hydrophobic membrane to separate the aqueous and organic phases, and the extraction time can be reduced by ⅓ to ¼ of the conventional extractor time. Note that the volume of solvent is based on the design of the extractor.

Solid Phase Extraction (SPE)

Solid phase extraction (SPE) can also be used for the extraction of chlorinated pesticides and PCBs from aqueous samples (EPA Methods 3535 and 508.1). SPE allows faster extraction times and a significant reduction in solvent use. The aqueous sample is passed through a cartridge or disk that contains a C18 packing material. The pesticides and PCBs are adsorbed by the C18 packing. The analytes are then eluted from the cartridge or disk using a small volume of ethyl acetate and methylene chloride. When using SPE, it is extremely important to follow the method recommendations. There are several manufacturers of C18 cartridges and disks (Figure 2), which are the typical media used for these compounds, and the extraction steps will vary somewhat depending

Figure 1: Separatory funnel.

Figure 2: SPE cartridges and disks commonly used for extracting chlorinated pesticides and PCBs from aqueous samples.

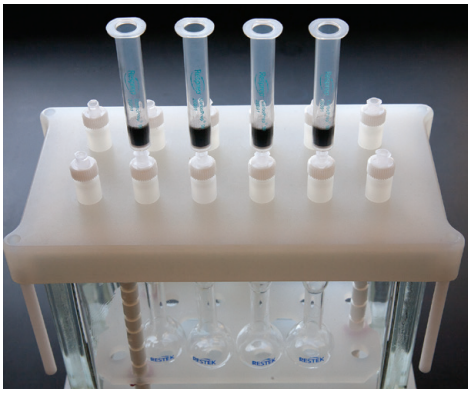


Figure 3: Soxhlet extractor.

Figure 4: Dionex ASE® system.

on the manufacturer and the format of the extraction media. Disks are more commonly used for the extraction of larger volumes of aqueous samples because they allow more rapid flow compared to tubes, which reduces extraction time. The two biggest issues with SPE are the clogging of the disk or tube with suspended solids and the breakthrough of targeted compounds. The use of pre-filters may reduce clogging of the extraction media. Breakthrough can occur if the sample contains a high level of organic material that will overload the capacity of the adsorbent. Samples with high concentrations of suspended solids and/or high organic content should not be extracted by SPE techniques.

Solid and Biota Samples

For solids and biota samples, Soxhlet or sonication extraction methods have historically been the most commonly used approach. In recent years, new extraction technologies, such as pressurized fluid and microwave extraction, have become more prevalent because they reduce solvent usage and are less labor intensive.

Soxhlet and Sonication Extraction

Soxhlet and sonication extraction work well for extracting halogenated pollutants from solid matrices and biological materials (Figure 3). Soxhlet extraction is time consuming and requires a large volume of solvent for extraction. Sonication (i.e., ultrasonic extraction) is a faster technique, but requires constant operator attention. In both techniques, problems such as contamination are attributed to either contaminated reagents, especially sodium sulfate, or poor laboratory practices being used when transferring sample extracts. Samples are commonly mixed with sodium sulfate to achieve a sandy consistency prior to solvent addition. Using granular sodium sulfate is recommended because some pesticides can adsorb to the powdered material.

Pressurized Fluid/Microwave/Supercritical Fluid Extraction

Several automated extraction methods have been developed for solid and biological samples. Pressurized fluid extraction (EPA Method 3545) runs unattended and utilizes much smaller solvent volumes than Soxhlet or sonication procedures. Two commercially available pressurized fluid extraction systems are the Dionex ASE® system (Figure 4) and the Applied Separations PSE system. Both systems are capable of extracting multiple samples unattended. Microwave extraction (EPA Method 3546) can be useful for automated extraction as well. Microwave extraction extracts multiple samples simultaneously. Commercially available microwave extraction systems are offered by Milestone Scientific and CEM Corporation. Supercritical fluid extraction (EPA Method 3560) has been promoted for a number of years as a "solventless" extraction technique for environmental samples. Its application is limited due to severe matrix-related variation, resulting in the need to modify the SFE conditions depending on soil type, water content, sample size, and the type of analytes. This ultimately requires additional sample preparation prior to the actual extraction. These requirements have limited the use of SFE as an extraction technique for many environmental applications.

Extraction Solvent Selection

For all sample extractions, use pesticide residue grade solvents and verify their purity prior to use by analyzing solvent blanks. This ensures that interferences are not caused by impurities in the solvent. To perform a solvent assay, reduce 300 to 400 mL of solvent to a final volume of 1 mL, and exchange to hexane for analysis by GC-ECD.

Since soil and biota samples are essentially wet particles, acetone and dichloromethane usually are used in a 1:1 combination as the extraction solvent. The acetone is needed to adequately penetrate into the soil particle so that compounds contained in the particle can be extracted. Several other solvent systems can be used for unique extractions, but generally this combination works for most applications.

When using dichloromethane as an extraction solvent it is important to note that it can form hydrochloric acid spontaneously without a stabilizer present. There are two classes of stabilizers: stabilizers that keep hydrochloric acid from forming, and stabilizers that eliminate hydrochloric acid upon formation. Methanol is used to stop hydrochloric acid from forming. It is not recommended to extract aqueous samples, or solid samples that contain water, using methanol-stabilized dichloromethane. The methanol will partition into the water, leaving an unstable extract. Hydrochloric acid forms quickly in unstabilized dichloromethane, and injection of an acidic solvent will result in reactive liners and columns. The second type of stabilizer, amylene (1-pentene), is commonly used to reduce hydrochloric acid formation. It is recommended to use dichloromethane stabilized with amylene since it has a low boiling point and will not interfere with early eluting target compounds.

Extract Drying

After the extraction has been completed, the extract is dried by passing it through anhydrous sodium sulfate. Removing water from the dichloromethane with sodium sulfate is critical before the extract is concentrated to final volume. Dichloromethane can hold approximately 1 mL of water per 100 mL of solvent. If water remains in the extract, it will partition out of the extract when the volume is reduced. If this occurs, either the dichloromethane will evaporate first, leaving only water in the collection vessel, or a two-layer extract will form. In either event, the recoveries of the target compounds will be lower than desired, and the presence of water will interfere with gas chromatographic analysis. The best way to remove the water is to filter the dichloromethane extract through granular sodium sulfate held in a funnel with a high-quality grade (Whatman® 541) filter paper or glass wool (Figure 5). Approximately 30 g of sodium sulfate is sufficient for most samples. *This step must not be skipped!* After drying the sample, thoroughly rinse the sodium sulfate to ensure that the entire sample is transferred to the collection vessel. Avoid using powdered sodium sulfate since some compounds can be adsorbed onto the smaller particles. Use only a 10-60 mesh granular sodium sulfate for best results. It also is important that this material be free from organic contaminants. Use pesticide residue grade sodium sulfate and store it in glass containers. If purchased in bulk packages where exposure to plastic is an issue, bake the material in a muffle furnace. To bake the sodium sulfate, spread it no more than 1 inch thick in a Pyrex®-type glass container, place into a muffle furnace, and bake at 400 °C for a minimum of two hours. After this time, place hot sodium sulfate into a glass container and securely cap the container to prevent re-adsorption of moisture from the atmosphere.

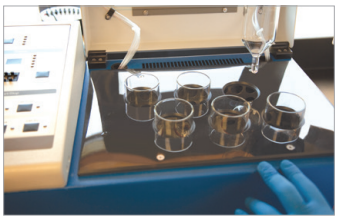
Figure 5: Drying the sample extract with sodium sulfate.

Bulk Sodium Sulfate

See **page 26** or visit

www.restek.com/**spe**

Figure 6: Extract concentrators.



TurboVap®

Reacti-Vap on Reacti-Therm

Kuderna-Danish (KD)

Glassware

See **page 25** or visit

www.restek.com/**glassware**

Extract Concentration

After drying the sample extract, the extraction solvent must be reduced in volume to concentrate the analytes prior to analysis. Kuderna-Danish (KD) type concentrators are most commonly used for this purpose. Alternatively, automated concentrators such as TurboVap® or RapidVap® concentrators can be used to reduce the solvent volume (Figure 6). If dichloromethane was the only solvent used as the extraction solvent, it is important to exchange it with hexane or isooctane during the concentration process. Even a small amount of dichloromethane will overwhelm an electron capture detector (ECD). Add 1 to 2 mL of hexane or isooctane to the sample extract prior to the evaporation process. Since dichloromethane is more volatile than hexane or isooctane it will evaporate first leaving the concentrated sample in a methylene chloride-free solution. The extract should never be allowed to dry completely during the concentration step. This could lead to loss of the more volatile components.

Extract Cleanup for Chlorinated Pesticides and PCBs

Sample extract cleanup is probably the most important step in maintaining long-term instrument and column performance. Generally, when instrument and column problems arise, they are caused by exposure of the injection port and the column to nonvolatile compounds from the sample matrix. Environmental samples often contain high molecular weight hydrocarbons, sulfur compounds, and phthalate esters. Plant and biota samples will contain pigments and lipids. While this nonvolatile material cannot be completely eliminated, some interferences can be reduced to levels where they become much less of a problem for the inlet and analytical column. Solid phase extraction (SPE) is the most common technique for removing these interfering compounds from the sample extracts. The choice of cleanup will depend on the type and concentration of the interfering compounds and the analytes being monitored. While cleanup procedures may add some additional processing time and cost, the resulting increase in instrumental stability, decrease in instrument and column maintenance, and potential improvements in detection limits can reduce the overall time and cost of analysis.

The two most common SPE cleanup materials used for chlorinated pesticide analysis are Florisil® and carbon adsorbents. These materials are available in pre-packed cartridges or in bulk for laboratories wishing to pack their own cartridges. Florisil® adsorbent is a magnesium silicate which is useful in removing polar contaminants from sample extracts. It is also useful for retaining co-extractants, such as phenols, that may interfere with GC analysis of pesticides. Florisil® material must be properly cleaned and activated prior to use. Activation requires heating to 130 °C for 16 hours in an oven. It is very important to check each lot of Florisil® adsorbent to ensure minimal background and to verify that the packing is at maximum activity level in order to maintain the expected retention capacity. Resprep® pre-packed Florisil® SPE cartridges are method-tested and suitable for most applications. For ultra-trace work, glass SPE cartridges with PTFE frits may be required to eliminate any trace interferences that might leach from the cartridge (Figure 7).

Figure 7: Glass SPE cartridge for extract cleanup.

Chromatographic-grade graphitized carbon is a versatile, nonporous adsorbent, which has been successfully used to remove high molecular weight nonpolar interferences from pesticide extracts. It is available in commercially prepacked SPE cartridges. In general, carbon elutes polar compounds first, then nonpolar compounds. For this reason, carbon makes a very good adsorbent to remove nonpolar matrix interferences from sample extracts. Since graphitized carbon retains or extracts a variety of compounds, the extraction system can be adjusted to retain and elute aliphatic, aromatic, polar, and nonpolar analytes. For optimal recoveries, compounds of interest should be applied in weak solvents or solutions with low solubility for the analytes, and eluted with strong solvents. Mixed solvent systems that include dichloromethane often are the most effective for elution. For ECD analysis it is critical to exchange the eluted sample into a non-halogenated solvent prior to analysis. Note that, due to the variability and high capacity of graphitized carbon, all fractionation and elution volumes should be verified for each lot of material. In addition, caution should be taken when using graphitized carbon to clean extracts for PCB congener analysis because the coplanar PCB congeners BZ#77, 81, 126, and 169 are very strongly retained and do not elute using the above solvent. These congeners can be eluted using a 1:1 mixture of ethyl acetate and toluene. Other cleanup techniques, such as sulfur removal using activated copper and gel permeation chromatography (GPC) may be used when necessary.

Analyzing Halogenated Pollutants

Choosing the most appropriate analytical column and injection technique are critical for achieving optimal results when testing for halogenated pollutants in environmental samples. A wide range of capillary columns have been recommended for the separation of different halogenated pollutants, which can result in frequent column changes and recalibration of the GC system. Using an Rtx®-CLPesticides column set for multiple methods is a much better approach, as these columns have been developed to separate a wide range of halogenated pollutants. Use of this column pair can result in shorter analysis time, improved separation, and increased sample throughput in the laboratory. Due to its high sensitivity for halogenated compounds, electron capture detection (ECD) is commonly used for halogenated pollutants. GC-MS is also used for analyzing halogenated pollutants, particularly now that newer, more sensitive instruments are available. Proper instrument setup and optimization are critical for accurate and consistent analyses. Many of these important considerations will be addressed here.

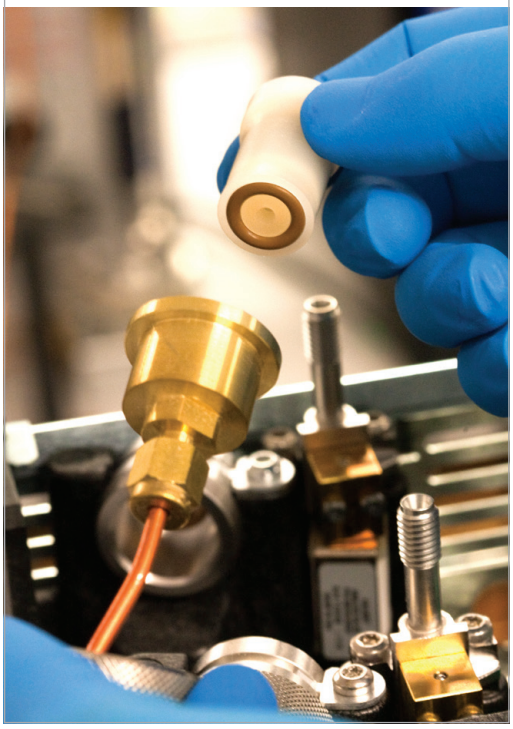
Column Selection

A wide variety of columns have been used for analyzing chlorinated pesticides and herbicides. The ideal column for this analysis would completely separate the compounds of interest, demonstrate excellent inertness to minimize breakdown and adsorption, and also exhibit low bleed at the high temperatures required to elute the target compounds and high molecular weight matrix interferences. Since confirmation is often required, dual column systems using two columns of different selectivity are usually used. It is critical that the primary and secondary (confirmational) columns be significantly different in selectivity in order to obtain the elution order and retention time changes that are needed to verify analytical results. In addition, both columns must be of the same length and internal diameter to ensure the same amount of sample is delivered to each column.

The Rtx®-CLPesticides and the Rtx®-CLPesticides2 columns were developed for the analysis of a wide range of halogenated pollutants. These two columns have significantly different selectivities and can be used for accurate reporting of many halogenated pollutants, which eliminates the need to change columns when analyzing samples for different methods. Both columns feature excellent inertness, low bleed, and high thermal stability. The high inertness of the Rtx®-CLPesticides column set ensures minimal breakdown and adsorption of active compounds such as endrin, DDT, and methoxychlor. The maximum temperature limit of the Rtx®-CLPesticides column and the Rtx®-CLPesticides2 column is up to 340 °C (depending on configuration), which allows analysts to use high temperature treatment in order to remove high boiling point contaminants. In addition, these columns can be operated under identical temperature programmed conditions for simultaneous confirmational analysis, while still providing different elution patterns. Each column is available in 0.18 mm, 0.25 mm, 0.32 mm, and 0.53 mm inner diameters (ID) and the phase ratio of each dimension has been optimized for maximum resolution and reduced analysis time.

Although Rtx®-CLPesticides columns are available in all common ID dimensions, the 0.32 mm ID size is typically recommended. This size provides the best combination of sample loading capacity and analysis time. If your sample extracts are particularly contaminated, you may find that the 0.53 mm ID columns allow for longer lasting calibration because of their larger capacity. Columns of 0.18 mm and 0.25 mm ID provide better resolution, but do not have sufficient sample loading capacity for contaminated or high concentration samples. In most cases, the 0.32 mm ID is the size of choice for pesticide and herbicide analyses. Example chromatograms are shown in the method section of this guide beginning on page 14.

Figure 8: Carbon trap installed on GC split vent.



Because ECDs are very sensitive detectors, you may need to condition your column overnight. Before doing so, check thoroughly for leaks and use high-quality oxygen-removal traps to purify your carrier gas. Visit **www.restek.com/gc-condition** for column conditioning instructions.

Injection Techniques

Several injection techniques can be used for analyzing halogenated pollutants: splitless, direct, and on-column injections. Splitless injection requires careful optimization for consistent and accurate results. Direct injection is a variation of splitless injection that offers some advantages when analyzing compounds that can react with active sites in the injection system. Cool on-column injection has also been used for analyzing these compounds. Each of these techniques, as well as their advantages and disadvantages, are discussed below.

Splitless Injection

Splitless injection is a heated vaporization technique that requires a split/splitless injection system. Using a syringe, the sample is introduced into a glass liner inside the heated inlet. This causes the sample to vaporize; however, the injection port must be hot enough to completely vaporize the solvent and the analytes. During the splitless injection mode, the split valve (purge valve) is closed, allowing all of the carrier gas flow and injected sample to be directed into the column. The split valve remains closed for a short time (30 sec to 2 min) after the injection in an attempt to transfer as much of the vaporized sample as possible from the injection port onto the column. After this splitless hold, the purge vent is opened, and the remaining solvent and non-transferred sample are vented out of the injection port. Since an ECD is a non-destructive detector, the purge vent should have a carbon trap (Figure 8) attached to prevent any organic compounds from being vented into the laboratory. The purge time must be optimized to ensure that the maximum amount of analyte is transferred to the column, while minimizing the amount of solvent. Generally, the purge time is determined by maximizing the area count of the last eluting analyte. This technique allows for the maximum amount of sample to be delivered to the column while excess solvent and contaminants are purged from the system, which can lead to attaining lower detection limits.

Splitless injection is prone to inertness problems because of the long residence time in the inlet and exposure of the analytes, such as endrin and 4,4'-DDT, to reactive surfaces in the injection system. The liner must be properly deactivated to prevent adsorption or breakdown and should be cleaned or replaced frequently to maintain proper inertness. Nonvolatile and very high boiling compounds from the sample matrix that are not vaporized can be left behind in the injection system. These materials will eventually contaminate the injection port liner and the injection system itself, leading to adsorption and lower response of active compounds. Another inertness problem is caused when the vaporized sample expands outside the liner, exposing reactive analytes to the metal surfaces inside the injector. The most common active area in the injection port is at the bottom. The vaporized sample cloud can expand past the column and come in contact with the metal disk (inlet seal) below the liner when using an Agilent® injection port. Installing gold-plated inlet seals will provide greater inertness. These inlet seals should be replaced along with the liner during routine maintenance because they will also become contaminated from nonvolatile sample residue.

Direct Injection

Direct injection is also a heated vaporization technique. The major difference between splitless and direct injection is how the column connects to the injection system. In conventional splitless injection, the liner is open at the bottom to allow the sample and solvent to vent out of the injector when the split valve is opened. With direct injection, the analytical column seals into a specially designed Uniliner® inlet liner using a tapered press-fit connection at the bottom of the liner. This allows the entire sample to enter the column and the GC system and eliminates analyte contact with the active metal surfaces below the bottom of the liner (Figure 9). An added benefit of using this type of injection is the reduction in injection port discrimination, which results in improved response for higher boiling compounds. When using a Uniliner® inlet liner, the split valve (purge valve) should be set in the off mode and a "higher than normal" flow rate should be used to contain the sample vapor cloud in the liner.

Cool On-Column Injection

In cool on-column injections, the needle is inserted directly into the column and the sample extract is deposited. On-column injections typically provide the narrowest peak width and the best reproducibility compared to other injection techniques. While on-column injection works extremely well for relatively clean samples, it is not a recommended technique for samples that contain nonvolatile residue. On-column injections are best suited for the analysis of drinking water sample extracts, where analyte concentration levels are usually low and the amount of nonvolatile material is relatively small. Conventional on-column injections are typically less than 1 µL and require the use of a 0.53 mm ID analytical column or a 0.53 mm ID guard column installed in the inlet and connected to a smaller diameter analytical column.

Dual Column Analysis

For confirmation purposes, two columns of differing selectivity are used when analyzing halogenated pollutants. This requires a GC system with dual electron capture detectors. The two most common techniques for installing two columns into the same injection system are a "Y" connector or a two-hole ferrule (Figure 10). There are several advantages of using a "Y" connector and guard column, which is why this configuration is recommended. First, this setup allows the user to perform splitless, direct, or on-column injections. Second, guard columns are commonly used to protect the analytical column from nonvolatile sample contaminants and increase column lifetime. Guard columns are deactivated, but uncoated, lengths of fused silica tubing that are connected to the front of the analytical column in order to protect it. Typically, a 5 m section of guard column is connected to the single leg side of

Figure 9: Direct injection using a Uniliner® inlet liner.

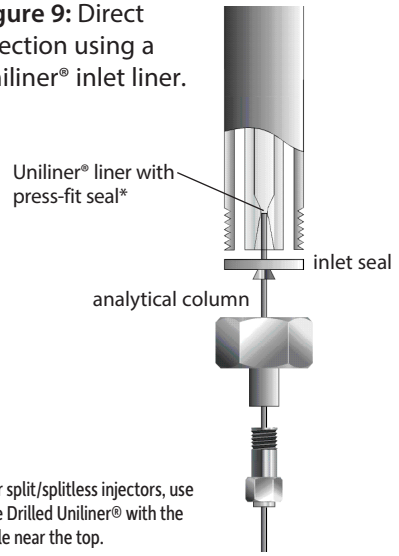
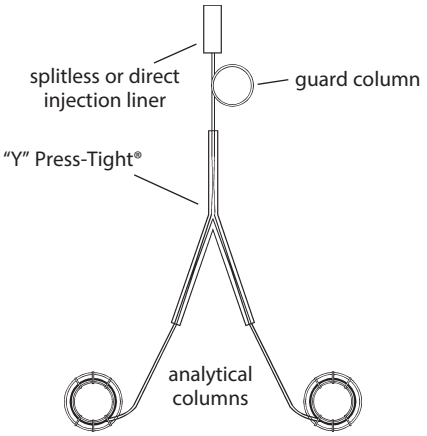
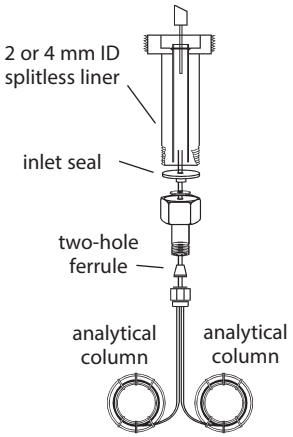


Figure 10: Dual column setups for different injection techniques.

A. Using a "Y" Press-Tight® connector for splitless or direct injection is recommended.



B. Alternatively, two-hole ferrules can be used for splitless injection.



How to Obtain a Leak-Tight Seal Using a Press-Tight® Connector

Press-Tight® connectors are easy to use, but leaks will occur if they are not properly sealed. The keys to successful sealing are:

1. Using a SeCure® "Y" connector kit.
2. Making a clean, square cut on the column.
3. Moistening the end of the column with methanol before seating it into the connector.
4. Using a Restek® electronic leak detector (cat.# 22655) to verify a leak-free connection at high inlet pressure (30–50 psi) following an oven cycle.

Guard Columns and Connectors

See **pages 28–29** or visit www.restek.com

the "Y" and the injector. The two analytical columns are then installed into the other two legs of the "Y" and into separate detectors. There are three styles of "Y" connectors. The more common Press-Tight® "Y" connector uses concentric compressive forces to form a leak-tight seal under normal operating pressures. This is also available in a SeCure® "Y" configuration for added stability. The MXT® "Y" connector is a metal connector that uses polyimide ferrules (see photos below). A less common approach is using a two-hole ferrule for installing two columns into one injector, but this requires two separate guard columns and separate connections between the guard columns and each of the analytical columns. The sample split between the two analytical columns will be dictated by their lengths and IDs. If the columns are the same length and ID, the split between the two columns will be approximately even. This setup cannot be used for on-column injection because the syringe needle can only enter one column. It also cannot be used for direct injections since only one column can be installed in the liner.

Electron Capture Detector Systems

One benefit of the ECD is its sensitivity to halogenated compounds, which allows extremely low detection limits for many of these compounds. Another benefit of using an ECD is its selectivity for electronegative compounds, which can eliminate some of the interferences in the sample matrix. To function properly, an ECD requires either nitrogen or 5% methane in argon (P5) as a make-up gas. The linearity of ECDs for a 16- to 100-fold concentration range is sufficient to pass most method calibration requirements. Linearity for ECDs is strongly affected by the flow rate of the make-up gas. To achieve linear response for chlorinated pesticides on an ECD, start by setting the flow rate of the make-up gas according to the manufacturer's recommendation and run a calibration curve including α-BHC and methoxychlor. Using these response factors, calculate the relative standard deviation (RSD) of each compound. Adjust the make-up gas flow rate so the percent RSD of these two compounds is approximately the same. Increasing the make-up gas flow will improve the linearity of α-BHC but make linearity worse for methoxychlor. The remaining pesticides will exhibit linear curves once the make-up gas has been set to give good linearity for α-BHC and methoxychlor. For more information on operating and maintaining your ECD, please consult your manufacturer's manual.

Analytical Methods

Chlorinated Pesticides and PCBs

Several methods have been developed for the analysis of chlorinated pesticides and PCBs (Table I). The Rtx®-CLPesticides and the Rtx®-CLPesticides2 column combination results in the separation of the majority of chlorinated pesticides and is suitable for PCB analysis as well. PCBs are most frequently analyzed as multi-component technical mixtures called Aroclors. Aroclor mixtures are differentiated by a numbering system that indicates the chlorination level of the mixture (e.g., Aroclor 1242 is 42% chlorine by weight). The increasing level of chlorination generates mixtures with components that have increasing numbers of chlorine atoms. This results in unique peak patterns for each of the Aroclor mixtures. The critical aspect of Aroclor analysis is pattern recognition, which is used to identify the specific Aroclor present in the sample. This can be difficult due to weathering or biodegradation of the sample. Also, some samples may contain more than one Aroclor which can greatly confuse pattern recognition.

Table I: Target analyte lists for chlorinated pesticides and PCBs in EPA methods.

Compound	CAS Registry Number	Method 508.1	Method 608	Method 8081B	Method 8081B Add'l Compounds	Method 8082A
Alachlor	15972-60-8	√			√	
Aldrin	309-00-2	√	√	√		
Aroclor 1016*	12674-11-2	√	√			√
Aroclor 1221*	11104-28-2	√	√			√
Aroclor 1232*	11141-16-5	√	√			√
Aroclor 1242*	53469-21-9	√	√			√
Aroclor 1248*	12672-29-6	√	√			√
Aroclor 1254*	11097-69-1	√	√			√
Aroclor 1260*	11096-82-5	√	√			√
Atarazine	1912-24-9	√				
Butachlor	23184-66-9	√				
α-BHC (α-HCH)	319-84-6	√	√	√		
β-BHC (β-HCH)	319-85-7	√	√	√		
δ-BHC (δ-HCH)	319-86-8	√	√	√		
γ-BHC (γ-HCH, Lindane)	58-89-9	√	√	√		
Captafol	2425-06-1				√	
Carbophenothion	786-19-6				√	
cis-Chlordane	5103-71-9	√		√		
trans-Chlordane	5103-74-2	√		√		
Chlorneb	2675-77-6	√			√	
Chlorobenzilate	510-15-6	√		√		
Chloropropylate	5836-10-2				√	
Chlorothalonil	1897-45-6	√			√	
Cyanazine	21725-46-2	√				
DBCP	96-12-8			√		
DCPA (Dacthal)	1861-32-1	√			√	
4,4'-DDD	72-54-8	√	√	√		
4,4'-DDE	72-55-9	√	√	√		
4,4'-DDT	50-29-3	√	√	√		
Diallate	2303-16-4			√		
Dichlone	117-80-6				√	
Dichloran	99-30-9				√	
Dicofol	115-32-2				√	
Dieldrin	60-57-1	√	√	√		
Endosulfan I	959-98-8	√	√	√		
Endosulfan II	33213-65-9	√	√	√		
Endosulfan sulfate	1031-07-8	√	√	√		
Endrin	72-20-8	√	√	√		
Endrin aldehyde	7421-93-4	√	√	√		
Endrin ketone	53494-70-5		√	√		
Etridiazole	2593-15-9	√			√	
Halowax-1000	58718-66-4				√	
Halowax-1001	58718-67-5				√	
Halowax-1013	12616-35-2				√	
Halowax-1014	12616-36-3				√	
Halowax-1051	2234-13-1				√	
Halowax-1099	39450-05-0				√	
Heptachlor	76-44-8	√	√	√		
Heptachlor epoxide	1024-57-3	√	√	√		
Hexachlorobenzene	118-74-1	√		√		
Hexachlorocyclopentadiene	77-47-4	√		√		
Isodrin	465-73-6			√		
Methoxychlor	72-43-5	√		√		
Metoachlor	51218-45-2	√				
Metribuzin	21087-64-9	√				
Mirex	2385-85-5				√	
Nitrofen	1836-75-5				√	
trans-Nonachlor	39765-80-5				√	
PCNB	82-68-8				√	
Permethrin (*cis* & *trans*)	52645-53-1				√	
cis-Permethrin	61949-76-6	√				
trans-Permethrin	61949-77-7	√				
Perthane	72-56-0				√	
Propachlor	1918-16-7	√			√	
Simazine	122-34-9	√				
Strobane	8001-50-1				√	
Technical chlordane*	57-74-9		√	√		
Toxaphene*	8001-35-2	√	√	√		
Trifluralin	1582-09-8	√			√	

*Multi-component standards.

Establishing and Maintaining Method Performance

The instrument used for the analysis of chlorinated pesticides and PCBs must be calibrated prior to performing quantitative analysis. The calibration should be linear over a 16- to 100-fold concentration range. The calibration of three to five points includes analyzing a low standard to meet the required reporting limit, as well as a high standard to minimize the need for dilutions. The linearity check should contain all the pesticides being reported. When using phenyl phase or cyanopropyl phase columns, not all analytes are resolved; therefore, two separate mixtures of pesticides must be used in order to ensure accurate calibration. This doubles the amount of time needed to calibrate for all the chlorinated pesticides. Because no coelution problems occur with the Rtx®-CLPesticides and Rtx®-CLPesticides2 columns, the calibrations can be completed with a single mixture, which reduces the overall time required to complete the calibration. Verifying linearity for all target compounds is important because different classes of pesticides (e.g., α-BHC vs. methoxychlor) will differ in injection port discrimination, chromatographic peak shape, and detector linearity. There are some chlorinated pesticides, such as technical chlordane and toxaphene, that are multi-component mixtures; due to their complexity, accurate calibrations are difficult to achieve. Typically, a few of the dominant peaks in each of the multi-component analytes are used to establish response factors. PCB calibration is handled the same way as the multi-component pesticides. Preferably, five peaks are chosen for the identification of each Aroclor. Initially, a five-point calibration of Aroclor 1016 and Aroclor 1260 is used. Single points at the midrange are then analyzed for the remaining Aroclor compounds.

Tips for Ensuring Optimum System Performance

Start With an Inert Injection Port

Active sites in the injection port can cause analyte breakdown and reduce recoveries. We recommend using deactivated Restek Topaz inlet liners and gold-plated dual Vespel® ring inlet seals to maximize inertness and prevent the loss of active compounds, such as endrin, DDT and 2,4-dinitrophenol.

Use Deactivated Guard Columns and Connectors

Guard columns protect your analytical column from nonvolatile residue and are also used for analyte focusing. Choose a deactivated guard column and attach it with a Press-Tight® connector to create an inert sample path and prevent the loss of target compounds.

Make Clean, Square Cuts for Leak-Free Connections

Restek® scoring wafers make cutting columns properly a quick and easy task. Just draw the smooth edge of the wafer perpendicularly across the fused silica surface, tap the column tubing, and then check your cut for jagged edges. Square cuts ensure a leak-free seal with the connector.

Prevent Contaminants From Entering the System

Oxygen, water, and hydrocarbon contaminants in carrier gas and detector gas lines will damage GC columns and cause noisy baselines. Use Super Clean® gas filters from Restek to purify incoming gases and protect your GC system.

Check for Leaks at all Connections

Catch a leak before it becomes a costly problem! Leaks allow oxygen and moisture to enter your system, which can cause permanent damage to GC columns and also to some detectors, including ECDs. Checking for leaks with an electronic leak detector allows you to identify and fix leaks before they cause problems.

The injection port is where many of the analytical problems occur in the analysis of chlorinated pesticides. Breakdown of endrin and 4,4'-DDT is not uncommon and usually is indicative of a chemical reaction taking place in the injection port. This breakdown could be caused by active glass wool, a contaminated or non-deactivated liner, an active metal surface in the injection system, septa particles, or impurities in the carrier gas. The breakdown of 4,4'-DDT is generally indicative of a dirty injection port caused by the analysis of oily or "dirty" sample extracts. GPC or carbon column cleanup can be useful in removing some of the matrix interferences that cause 4,4'-DDT breakdown (see Extract Cleanup for Chlorinated Pesticides and PCBs section). Frequent replacement of the inlet liner and inlet seal may be necessary to maintain low breakdown levels of endrin and 4,4'-DDT. It may also be necessary to cut 6 to 12 inches off the front of the analytical column or guard column to reduce the breakdown of endrin and 4,4'-DDT. Trimming the analytical or guard column removes any nonvolatile residue buildup in the column. Using a guard column will extend the lifetime of the analytical column and minimize the loss of separation that will result from trimming the analytical column.

Best practices and EPA methodology dictate that the inertness of the chromatographic system be confirmed on a regular basis. This is accomplished by analyzing a system evaluation mix that contains endrin and 4,4'-DDT. Endrin decomposes to endrin aldehyde and endrin ketone. 4,4'-DDT decomposes to 4,4'-DDD and 4,4'-DDE. The percentage of decomposition can be measured and must be below 15% for both compounds. If the breakdown is above 15%, instrument maintenance must be performed. Restek provides a complete range of system evaluation test mixes, surrogate standards, and calibration standards for chlorinated pesticides and PCBs. Visit **www.restek.com** for a complete product listing.

If replacing the inlet liner and trimming the front of the analytical column do not reduce breakdown, it may be necessary to perform additional inlet maintenance. When samples are injected into the hot injection port, the vaporized extract can backflash and escape from the top and bottom of the liner, causing analytes to come into contact with metal surfaces. This will leave nonvolatile residue on the metal surfaces of the injection port and create active sites. Keeping the injection port body clean requires frequent cleaning or replacement of the inlet seal. Periodic swabbing of the inside of the injection port with solvent may also be necessary if other maintenance does not reduce endrin and 4,4'-DDT breakdown. It may even be necessary to rinse the carrier gas lines coming into the injector to remove contamination that has backflashed into these lines. Do not flush solvent through any actuator valves or rubber parts and be certain the injection port is at room temperature prior to rinsing.

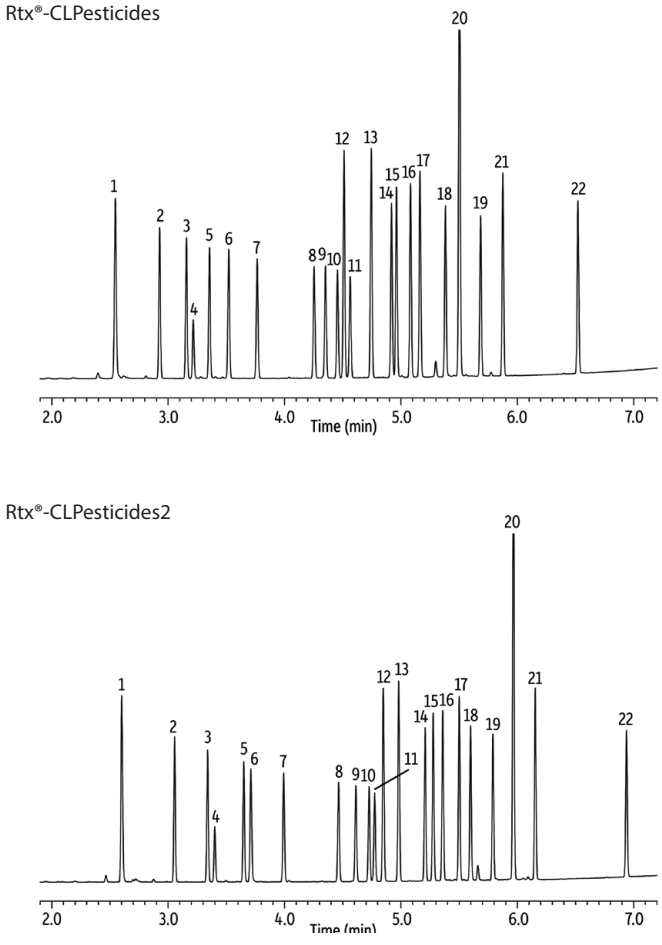
Some labs choose to clean and reuse inlet liners. When this is done, it is critical to use proper deactivation procedures to minimize endrin breakdown. There are two approaches to liner deactivation: perform the operation in-house or send liners out to be deactivated. Sending injection port liners to a company like Restek for cleaning and deactivating is inexpensive and allows analysts to spend their time more productively. There is a standard procedure for deactivating liners that includes a process of cleaning the liners and deactivating them. Contact Restek's Technical Service group at support@restek.com; 1-800-356-1688, ext. 4; or 1-814-353-1300, ext. 4 for more information.

Septa particles are another major cause of endrin breakdown. Over time, the septum will core and septum particles will sit on top of a glass wool plug or at the bottom of the liner. Changing septa frequently and using high-quality septa can reduce coring and particle generation. Ensuring that the syringe needle is free of burrs will also help reduce this problem. Alternatively, Merlin Microseal septa can eliminate particles from collecting in the liner. Information on Merlin Microseal septa is available at www.restek.com

Carrier gas contamination is another potential cause of endrin breakdown. Endrin can react with contaminants that are carried into the injection port by the carrier gas. Using in-line gas purifiers for the carrier gas will help remove contaminants so they are not introduced into the analytical system.

Figures 11 through 16 show example chromatograms of chlorinated pesticides and PCBs on the Rtx®-CLPesticides column set using optimized analysis conditions.

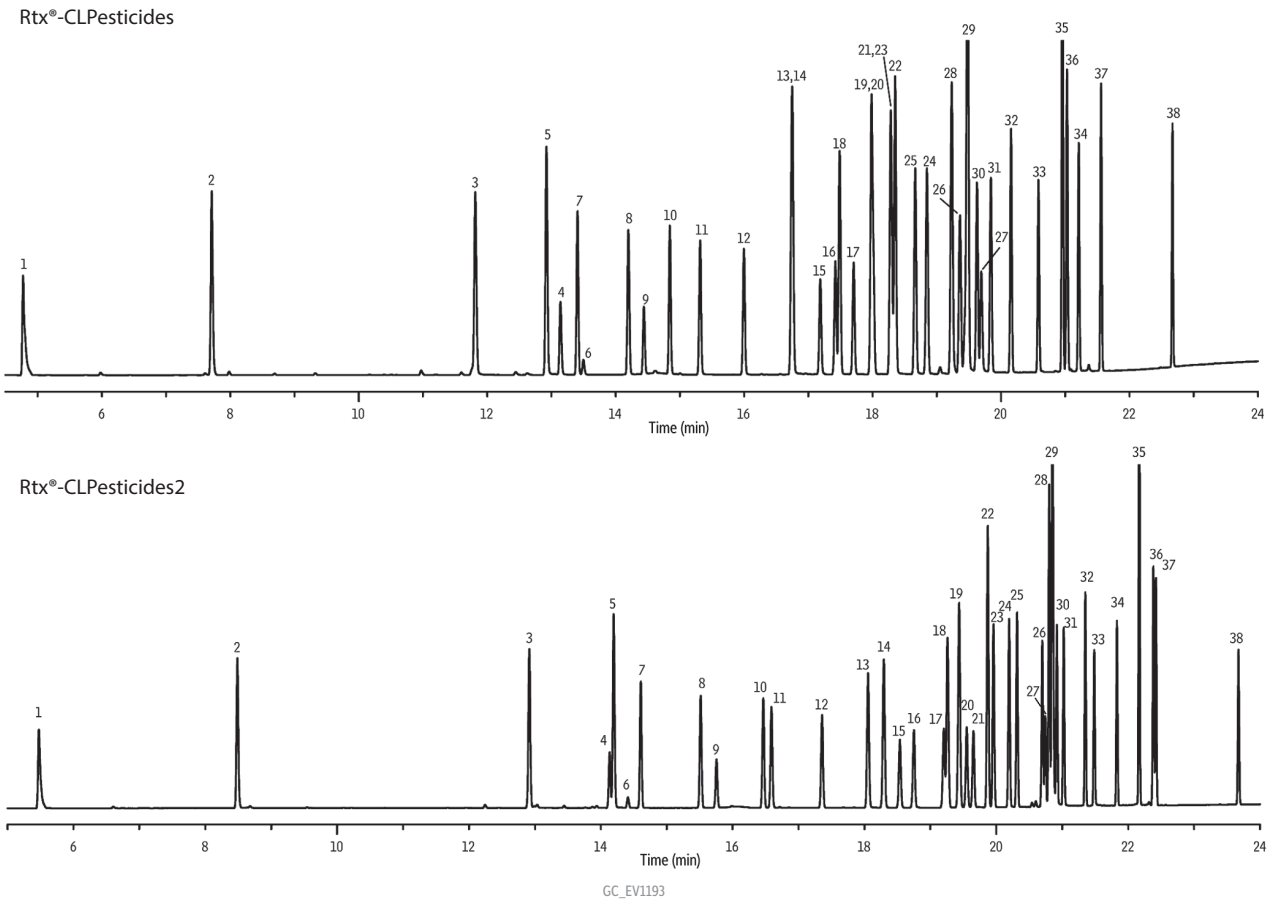
Figure 11: For commonly analyzed chlorinated pesticides, 0.32 mm ID columns provide the best balance of capacity and resolution.



GC_EV00933

Columns	Rtx®-CLPesticides 30 m, 0.32 mm ID, 0.32 µm (cat.# t) and Rtx®-CLPesticides2 30 m, 0.32 mm ID, 0.25 µm (cat.# 11324) using Rxi® guard column 5 m, 0.32 mm ID (cat.# 10039) with deactivated universal "Y" Press-Tight® connector (cat.# 20405-261)
Sample	Organochlorine pesticide mix AB #2 (cat.# 32292) Pesticide surrogate mix, EPA 8080, 8081 (cat.# 32000)
Injection	
Inj. Vol.:	1 µL splitless (hold 0.3 min)
Liner:	Splitless taper (4 mm) (cat.# 20799)
Inj. Temp.:	250 °C
Oven	
Oven Temp:	120 °C to 200 °C at 45 °C/min to 230 °C at 15 °C/min to 330 °C at 30 °C/min (hold 2 min)
Carrier Gas	He
Detector	µ-ECD @ 330 °C
Notes	Instrument was operated in constant flow mode. Linear velocity: 60 cm/sec @ 120 °C. This chromatogram was obtained using an Agilent µ-ECD. To obtain comparable results, you will need to employ a µ-ECD in addition to confirmational dual columns connected to a 5-meter guard column using a "Y" Press-Tight® connector.

Figure 12: Analysis of an extended list of organochlorine pesticides (EPA Method 8081B) on the Rtx®-CLPesticides and Rtx®-CLPesticides2 columns.



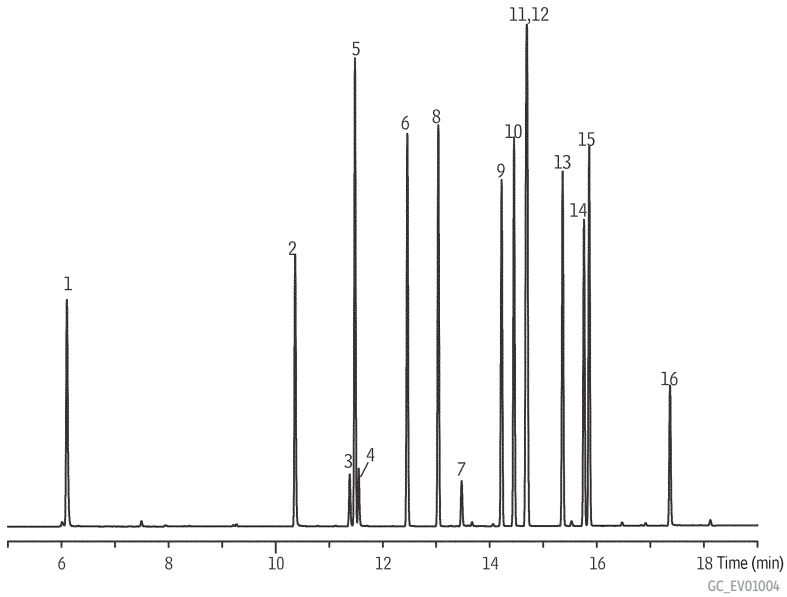
Columns	Rtx®-CLPesticides2 30 m, 0.32 mm ID, 0.25 µm (cat.# 11324) and Rtx®-CLPesticides 30 m, 0.32 mm ID, 0.32 µm (cat.# 11141) using Rxi® guard column 5 m, 0.32 mm ID (cat.# 10039) with deactivated universal "Y" Press-Tight® connector (cat.# 20405-261)
Sample	Organochlorine pesticide mix AB #2 (cat.# 32292) Organochlorine pesticide mix C #1 (cat.# 32296) 2,4,5,6-Tetrachloro-*m*-xylene (cat.# 32027) Decachlorobiphenyl (BZ #209) (cat.# 32029) Chlorpyrifos (cat.# 32212) 2,4'-DDE (cat.# 32099) 2,4'-DDD (cat.# 32098) Chlorobenzilate (cat.# 32211) Captan (cat.# custom) *cis*-Nonachlor (cat.# custom) Mirex (cat.# custom) Oxychlordone (cat.# custom) Diallate (cat.# custom)
Diluent:	Hexane
Injection	
Inj. Vol.:	1 µL splitless (hold 0.3 min)
Liner:	Cyclo double taper (4 mm) (cat.# 20896)
Inj. Temp.:	250 °C
Oven	
Oven Temp:	80 °C to 175 °C at 8 °C/min to 195 °C at 11 °C/min to 235 °C at 7 °C/min to 320 °C at 25 °C/min (hold 3 min)
Carrier Gas	He, constant flow
Linear Velocity:	28 cm/sec
Detector	µ-ECD @ 330 °C
Make-up Gas Flow Rate:	60 mL/min
Make-up Gas Type:	N_2
Instrument	Agilent/HP6890 GC
Notes	This chromatogram was obtained using an Agilent µ-ECD. To obtain comparable results, you will need to employ a µ-ECD in addition to confirmational dual columns connected to a 5-meter guard column using a "Y" Press-Tight® connector.

	Peaks	Conc. (ng/mL)		Peaks	Conc. (ng/mL)
1.	1,2-Dibromo-3-chloropropane	100	20.	*cis*-Chlordane*	40
2.	Hexachlorocyclopentadiene	100	21.	Endosulfan I	40
3.	Tetrachloro-*m*-xylene (SS)	100	22.	Captan	100
4.	*cis*-Diallate	500	23.	4,4'-DDE	100
5.	Hexachlorobenzene	100	24.	Dieldrin	80
6.	*trans*-Diallate	500	25.	2,4'-DDD	100
7.	α-BHC	40	26.	Endrin	80
8.	γ-BHC	40	27.	Chlorobenzilate	500
9.	β-BHC	40	28.	2,4'-DDT	100
10.	δ-BHC	40	29.	*cis*-Nonachlor	100
11.	Heptachlor	40	30.	4,4'-DDD	80
12.	Aldrin	40	31.	Endosulfan II	80
13.	Chlorpyrifos	100	32.	4,4'-DDT	80
14.	Isodrin	100	33.	Endrin aldehyde	80
15.	Oxychlordane	50	34.	Endosulfan sulfate	80
16.	Heptachlor epoxide	40	35.	Methoxychlor	400
17.	*trans*-Chlordane*	40	36.	Mirex	100
18.	2,4'-DDE	100	37.	Endrin ketone	80
19.	*trans*-Nonachlor	100	38.	Decachlorobiphenyl	100

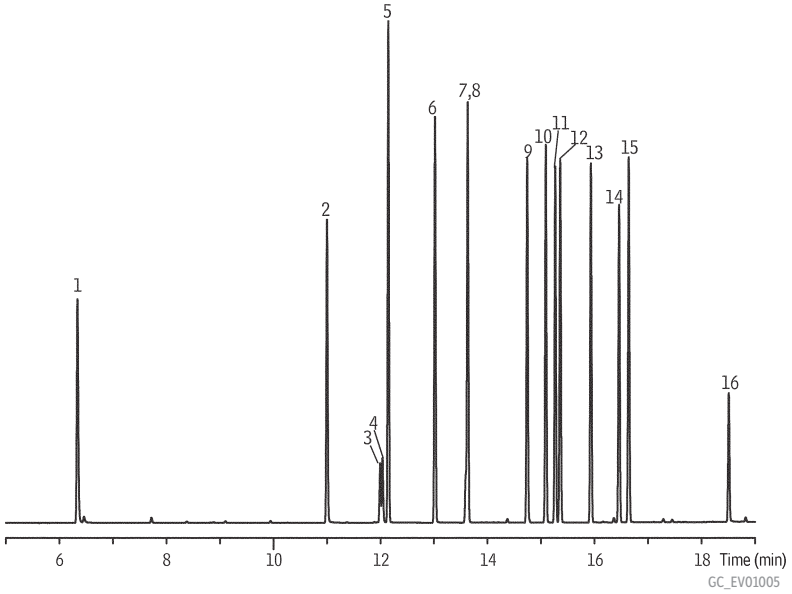
* For information regarding the nomenclature used for *cis*-chlordane and *trans*-chlordane, visit **www.restek.com/chlordane-notice**

Figure 13: Common pesticides monitored for in drinking water and groundwater.

Rtx®-CLPesticides



Rtx®-CLPesticides2



Peaks

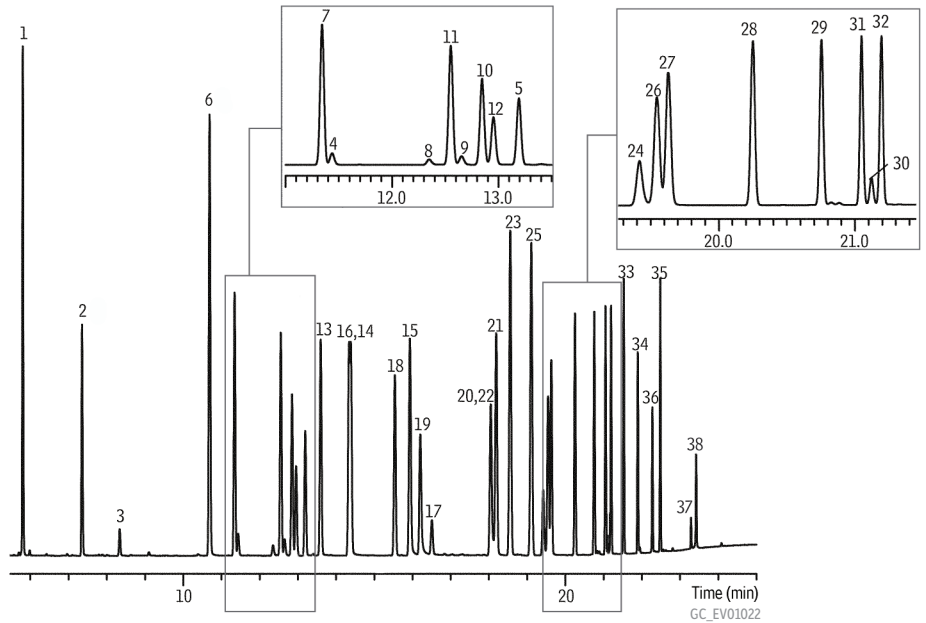
1. Hexachlorocyclopentadiene
2. Hexachlorobenzene
3. Simazine
4. Atrazine
5. γ-BHC
6. Heptachlor
7. Alachlor
8. Aldrin
9. Heptachlor epoxide
10. *trans*-Chlordane*
11. *trans*-Nonachlor
12. *cis*-Chlordane*
13. Dieldrin
14. Endrin
15. *cis*-Nonachlor
16. Methoxychlor

* For information regarding the nomenclature used for *cis*-chlordane and *trans*-chlordane, visit **www.restek.com/chlordane-notice**

Columns	Rtx®-CLPesticides 30 m, 0.32 mm ID, 0.32 µm (cat.# 11141) and Rtx®-CLPesticides2 30 m, 0.32 mm ID, 0.25 µm (cat.# 11324) using Rxi® guard column 5 m, 0.32 mm ID (cat.# 10039) with deactivated universal "Y" Press-Tight® connector (cat.# 20405-261)
Sample	200 ng/mL 505 organohalide pesticide mix (cat.# 32024) 4.2 µg/mL simazine (cat.# 32236) 4.2 µg/mL atrazine (cat.# 32208)
Diluent:	Methanol
Injection	
Inj. Vol.:	2 µL splitless (hold 0.75 min)
Liner:	Cyclo double taper (4 mm) (cat.# 20896)
Inj. Temp.:	250 °C
Oven	
Oven Temp:	90 °C (hold 1 min) to 310 °C at 10 °C/min (hold 5 min)
Carrier Gas	He, constant flow
Linear Velocity:	40 cm/sec
Detector	µ-ECD @ 325 °C
Instrument	Agilent/HP6890 GC
Notes	This chromatogram was obtained using an Agilent µ-ECD. To obtain comparable results, you will need to employ a µ-ECD in addition to confirmational dual columns connected to a 5-meter guard column using a "Y" Press-Tight® connector.

Figure 14: Pesticides and herbicides monitored for in drinking water and groundwater on the Rtx®-CLPesticides and Rtx®-CLPesticides2 columns.

Rtx®-CLPesticides

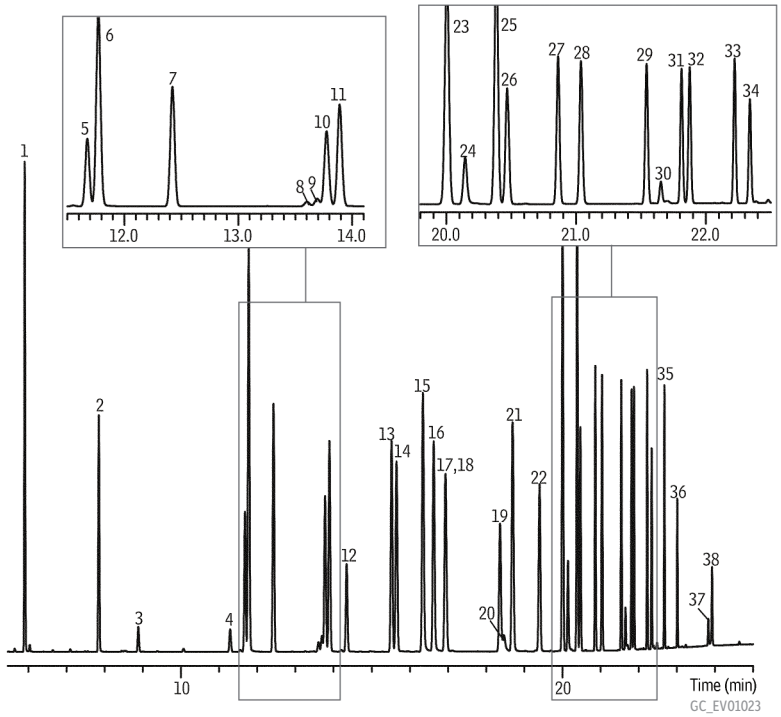


Peaks

1. Hexachlorocyclopentadiene
2. Etridiazole
3. Chlorneb
4. Propachlor
5. Trifluralin
6. Hexachlorobenzene
7. α-BHC
8. Simazine
9. Atrazine
10. Pentachloronitrobenzene (IS)
11. γ-BHC
12. β-BHC
13. δ-BHC
14. Heptachlor
15. Chlorothalonil
16. Metribuzin
17. Alachlor
18. Aldrin
19. 4,4'-Dibromobiphenyl (SS)
20. Metachlor
21. DCPA
22. Heptachlor epoxide
23. *trans*-Chlordane*
24. Cyanazine
25. *cis*-Chlordane*
26. Endosulfan I
27. 4,4'-DDE
28. Dieldrin
29. Endrin
30. Chlorobenzilate
31. 4,4'-DDD
32. Endosulfan II
33. 4,4'-DDT
34. Endrin aldehyde
35. Endosulfan sulfate
36. Methoxychlor
37. *cis*-Permethrin
38. *trans*-Permethrin

* For information regarding the nomenclature used for *cis*-chlordane and *trans*-chlordane, visit **www.restek.com/chlordane-notice**

Rtx®-CLPesticides2

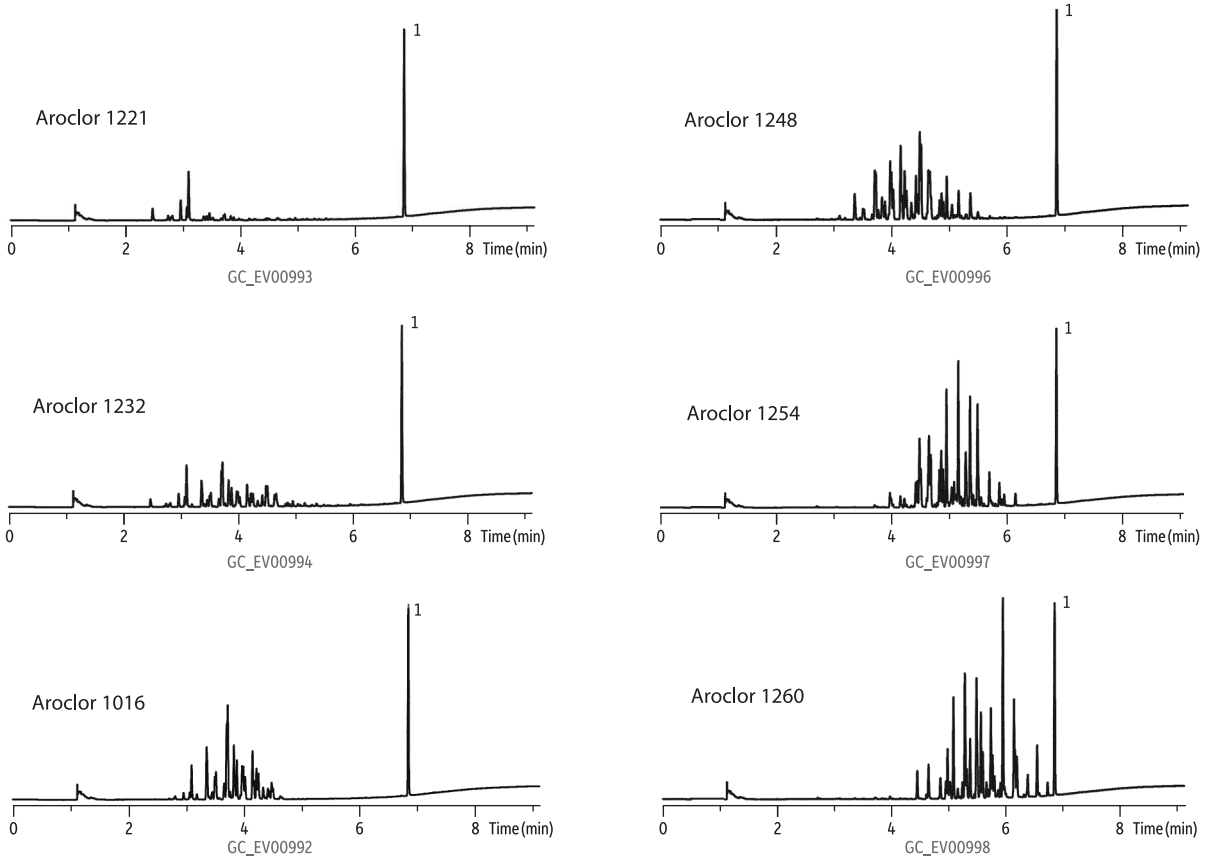


Columns	Rtx®-CLPesticides2 30 m, 0.32 mm ID, 0.25 µm (cat.# 11324) and Rtx®-CLPesticides 30 m, 0.32 mm ID, 0.32 µm (cat.# 11141) using Rxi® guard column 5 m, 0.32 mm ID (cat.# 10039) with deactivated universal "Y" Press-Tight® connector (cat.# 20405-261)
Sample	50 ng/mL 508.1 calibration mix #1 (cat.# 32094) 100 ng/mL 508.1 calibration mix #2 (cat.# 32095) 100 ng/mL 508.1 calibration mix #3 (cat.# 32096) 50 ng/mL pentachloronitrobenzene (cat.# 32091) 250 ng/mL 4,4'-dibromobiphenyl (cat.# 32092) 500 ng/mL atrazine (cat.# 32208) 500 ng/mL simazine (cat.# 32236)
Diluent:	Ethyl acetate
Injection	
Inj. Vol.:	2 µL splitless (hold 0.75 min)
Liner:	Cyclo double taper (4 mm) (cat.# 20896)
Inj. Temp.:	250 °C
Oven	
Oven Temp:	80 °C (hold 0.5 min) to 155 °C at 19 °C/min (hold 1 min) to 210 °C at 4 °C/min to 310 °C at 25 °C/min (hold 0.5 min)
Carrier Gas	He, constant flow
Linear Velocity:	26 cm/sec
Detector	µ-ECD @ 325 °C
Notes	This chromatogram was obtained using an Agilent µ-ECD. To obtain comparable results, you will need to employ a µ-ECD in addition to confirmational dual columns connected to a 5-meter guard column using a "Y" Press-Tight® connector.

Figure 15: Aroclor compounds on Rtx®-CLPesticides columns.

Rtx®-CLPesticides
30 m, 0.32 mm ID, 0.32 µm (cat.# 11141)

Peaks
1. Decachlorobiphenyl (DCB)

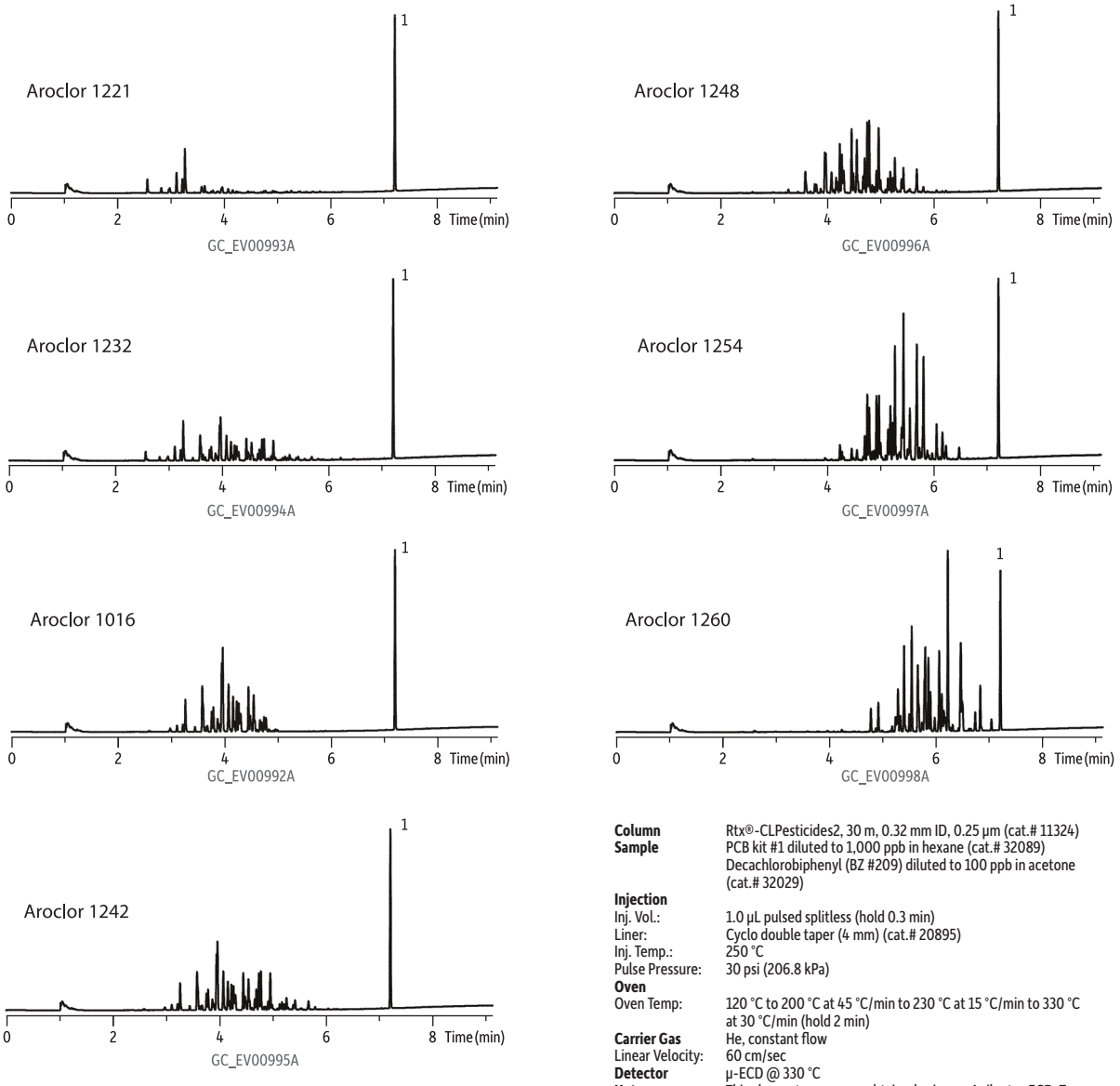


Column	Rtx®-CLPesticides, 30 m, 0.32 mm ID, 0.32 µm (cat.# 11141)
Sample	PCB kit #1 diluted to 1,000 ppb in hexane (cat.# 32089) Decachlorobiphenyl (BZ #209) diluted to 100 ppb in acetone (cat.# 32029)
Injection	
Inj. Vol.:	1.0 µL pulsed splitless (hold 0.3 min)
Liner:	Cyclo double taper (4 mm) (cat.# 20895)
Inj. Temp.:	250 °C
Pulse Pressure:	30 psi (206.8 kPa)
Oven	
Oven Temp:	120 °C to 200 °C at 45 °C/min to 230 °C at 15 °C/min to 330 °C at 30 °C/min (hold 2 min)
Carrier Gas	He, constant flow
Linear Velocity:	60 cm/sec
Detector	µ-ECD @ 330 °C
Notes	This chromatogram was obtained using an Agilent µ-ECD. To obtain comparable results, you will need to employ a µ-ECD in addition to confirmational dual columns connected to a 5-meter guard column using a "Y" Press-Tight® connector.

Figure 16: Aroclor compounds on Rtx®-CLPesticides2 columns.

Rtx®-CLPesticides2
30 m, 0.32 mm ID, 0.25 µm (cat.# 11324)

Peaks
1. Decachlorobiphenyl (DCB)



Column	Rtx®-CLPesticides2, 30 m, 0.32 mm ID, 0.25 µm (cat.# 11324)
Sample	PCB kit #1 diluted to 1,000 ppb in hexane (cat.# 32089) Decachlorobiphenyl (BZ #209) diluted to 100 ppb in acetone (cat.# 32029)
Injection	
Inj. Vol.:	1.0 µL pulsed splitless (hold 0.3 min)
Liner:	Cyclo double taper (4 mm) (cat.# 20895)
Inj. Temp.:	250 °C
Pulse Pressure:	30 psi (206.8 kPa)
Oven	
Oven Temp:	120 °C to 200 °C at 45 °C/min to 230 °C at 15 °C/min to 330 °C at 30 °C/min (hold 2 min)
Carrier Gas	He, constant flow
Linear Velocity:	60 cm/sec
Detector	µ-ECD @ 330 °C
Notes	This chromatogram was obtained using an Agilent µ-ECD. To obtain comparable results, you will need to employ a µ-ECD in addition to confirmational dual columns connected to a 5-meter guard column using a "Y" Press-Tight® connector.

Chlorinated Herbicides

Several methods have been developed for the analysis of chlorinated herbicides, including the methods listed in Table II. Since these compounds are used in different forms (free acids, salts, and esters) the methods require a hydrolysis step to convert all compounds to the free acid form. However, the free acid form is not amenable to gas chromatography and must be derivatized to the methyl ester form before GC analysis. This esterification is accomplished using diazomethane. *Diazomethane is a highly toxic, reactive, and potentially explosive material that should only be handled by analysts experienced with this process.* Pentafluorobenzyl bromide has also been used for the derivatization of chlorinated acid herbicides, but interferences from phenols and chlorinated phenols can be problematic for this derivatization reagent. Since hydrolyzed acids can readily react with basic surfaces, it is critical to acid rinse all glassware used for sample extraction and transfer. It is also recommended to acid rinse the sodium sulfate used for extract drying.

Table II: Target analyte lists for chlorinated herbicides by EPA method.

Compound	CAS #	Method 515.1	Method 615	Method 8151
Acifluorfen	50594-66-6	√		
Bentazon	25057-89-0	√		
Chloramben	133-90-4	√		
2,4-D	94-75-7	√	√	√
Dalapon	75-99-0	√	√	√
2,4-DB	94-82-6	√	√	√
DCPA acid metabolites	-	√		
Dicamba	1918-00-9	√	√	√
3,5-Dichlorobenzoic acid	51-36-5	√		
Dichlorprop	120-36-5	√	√	√
Dinoseb	88-85-7	√	√	√
5-Hydroxydicamba	7600-50-2	√		
MCPA	94-74-6		√	√
MCPP	7085-19-0		√	√
4-Nitrophenol	100-02-7	√		√
Pentachlorophenol (PCP)	87-86-5	√		√
Picloram	1918-02-1	√		
2,4,5-T	93-76-5	√	√	√
2,4,5-TP	93-72-1	√	√	√

Once esterified, the chlorinated herbicides can be analyzed by GC and Rtx®-CLPesticides and Rtx®-CLPesticides2 columns can be used to separate these derivatized compounds. Both internal and surrogate standards are specified for these methods to ensure accurate quantitative results. Restek provides a complete range of internal and surrogate standards, as well as calibration standards, for chlorinated herbicides as both free acids and methyl esters. Figure 17 shows example chromatograms of chlorinated herbicides on the Rtx®-CLPesticides column set using optimized analysis conditions.

Table III: Target analyte lists for haloacetic acids (EPA Method 552.2).

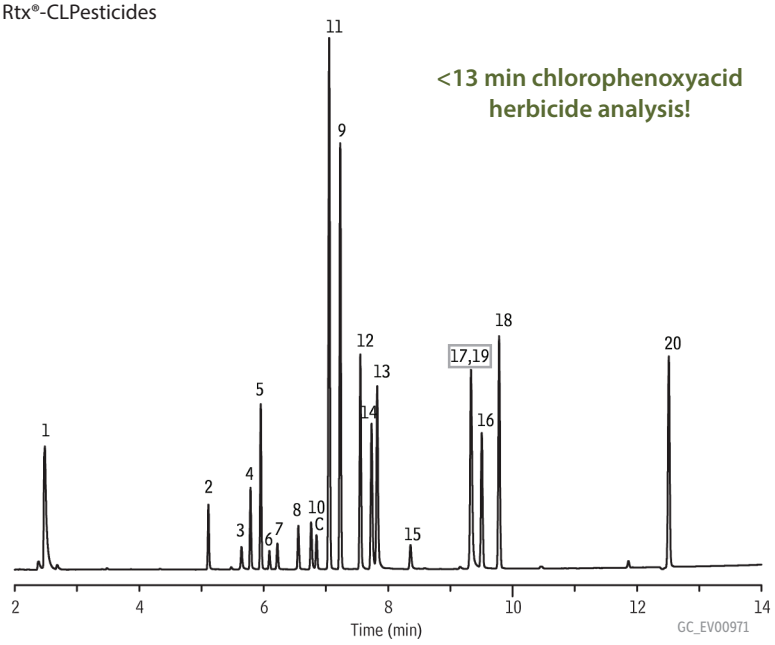
Compound	CAS #
Bromochloroacetic acid (BCAA)	5589-96-8
Bromodichloroacetic acid (BDCAA)	7113-314-7
Chlorodibromoacetic acid (CDBAA)	5278-95-5
Dalapon	75-99-0
Dibromoacetic acid (DBAA)	631-64-1
Dichloroacetic acid (DCAA)	79-43-6
Monobromoacetic acid (MBAA)	79-08-3
Monochloroacetic acid (MCAA)	79-11-8
Tribromoacetic acid (TBAA)	75-96-7
Trichloroacetic acid (TCAA)	76-03-9

Haloacetic Acids

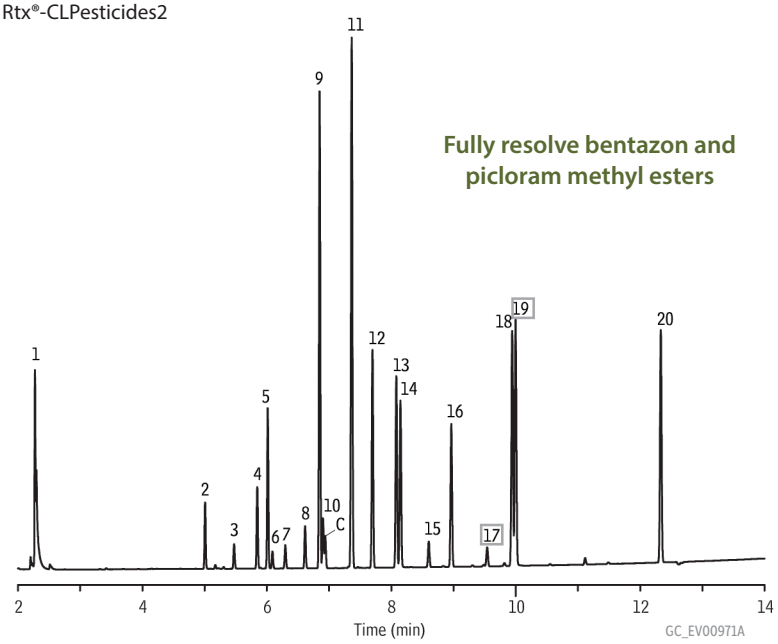
Haloacetic acids are byproducts created when drinking water is chlorinated. Analytical methods (such as EPA 552.2) were developed for the analysis of haloacetic acids in drinking water. Table III shows the target list of compounds included in this method. A 100 mL sample of water is adjusted to a pH of 11.5 and extracted with methyl *tert*-butyl ether (MTBE) to remove basic and neutral compounds from the sample. The sample is then acidified to pH 0.5 and the haloacetic acids are extracted into MTBE. The acids are then converted to methyl esters using diazomethane and analyzed by capillary GC using an ECD detector. *Diazomethane is a highly toxic, reactive, and potentially explosive material that should only be handled by analysts experienced with this process.* Note that derivatization can also be accomplished using acidic methanol and heat.

Figure 17: Analysis of chlorinated herbicides on the Rtx®-CLPesticides column set.

Rtx®-CLPesticides



Rtx®-CLPesticides2



Peaks

1. Dalapon methyl ester
2. 3,5-Dichlorobenzoic acid methyl ester (SS)
3. 4-Nitroanisole
4. DCAA methyl ester (SS)
5. Dicamba methyl ester
6. MCPP methyl ester
7. MCPA Methyl ester
8. Dichlorprop, methyl ester
9. 4,4'-DBOB (IS)
10. 2,4-D methyl ester
11. Pentachloroanisole
12. 2,4,5-TP, methyl ester
13. 2,4,5-T methyl ester
14. Chloramben, methyl ester
15. 2,4-DB methyl ester
16. Dinoseb methyl ester
17. Bentazon methyl ester
18. DCPA
19. Pichloram methyl ester
20. Acifluorfen methyl ester

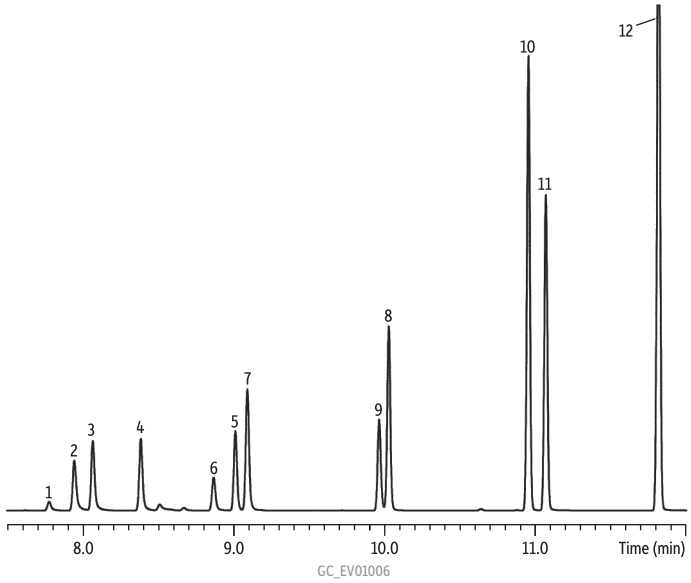
C. contaminant

Columns	Rtx®-CLPesticides2 30 m, 0.32 mm ID, 0.25 µm (cat.# 11324) and Rtx®-CLPesticides 30 m, 0.32 mm ID, 0.32 µm (cat.# 11141) using Rxi® deactivated guard column 5 m, 0.32 mm ID (cat.# 10039) with universal "Y" Press-Tight® connector (cat.# 20405-261)
Sample	200 ng/mL herbicide mix #1 (cat.# 32055) 1,000 ng/mL dalapon methyl ester (cat.# 32057) 20,000 ng/mL herbicide mix #3 (cat.# 32059) 200 ng/mL herbicide mix #4 (cat.# 32062) 250 ng/mL 4,4'-dibromooctafluorobiphenyl (cat.# 32053) 400 ng/mL 2,4-dichlorophenyl acetic acid methyl ester (cat.# 32050)
Diluent:	Hexane
Injection	
Inj. Vol.:	1.0 µL splitless (hold 0.75 min)
Liner:	Cyclo double taper (4 mm) (cat.# 20895)
Inj. Temp.:	250 °C
Oven	
Oven Temp:	70 °C (hold 0.5 min) to 190 °C at 25 °C/min (hold 1 min) to 300 °C at 11 °C/min (hold 5 min)
Carrier Gas	He, constant pressure
Linear Velocity:	36 cm/sec @ 70 °C
Detector	µ-ECD @ 325 °C
Instrument	Agilent/HP6890 GC
Notes	This chromatogram was obtained using an Agilent µ-ECD. To obtain comparable results, you will need to employ a µ-ECD in addition to confirmational dual columns connected to a 5-meter guard column using a "Y" Press-Tight® connector.

Figure 18 shows example chromatograms of derivatized haloacetic acids on the Rtx®-CLPesticides column set using optimized analysis conditions. Both internal and surrogate standards are specified for this method to ensure accurate quantitative results. Restek provides a complete range of internal and surrogate standards, as well as calibration standards, for haloacetic acids as both free acids and methyl esters.

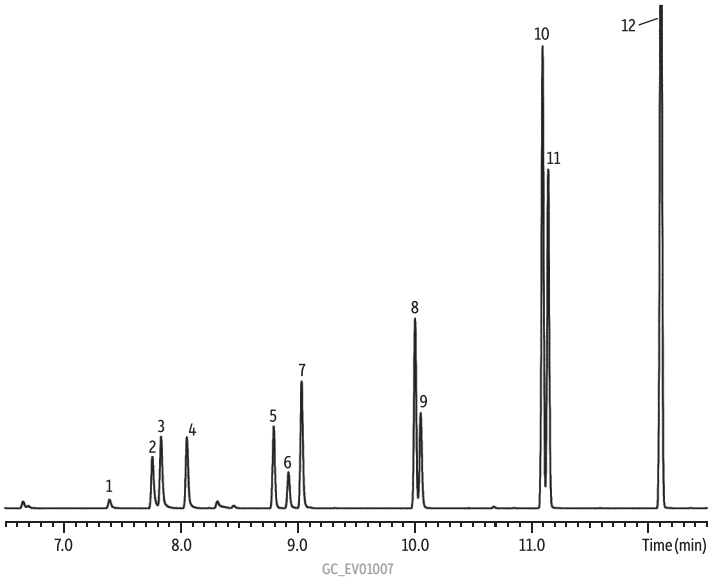
Figure 18: Analysis of derivatized haloacetic acids on Rtx®-CLPesticides and Rtx®-CLPesticides2 columns.

Rtx®-CLPesticides



	Peaks	Conc. (µg/mL)
1.	Methyl monochloroacetate	1.2
2.	Methyl monobromoacetate	0.8
3.	Methyl dichloroacetate	1.2
4.	Dalapon methyl ester	2
5.	Methyl trichloroacetate	0.4
6.	1,2,3-Trichloropropane (IS)	4
7.	Methyl bromo(chloro)acetate	0.8
8.	Methyl bromodichloroacetate	0.8
9.	Methyl dibromoacetate	0.4
10.	Methyl chlorodibromoacetate	2
11.	Methyl 2,3-dibromopropionate (SS)	2
12.	Methyl tribromoacetate	4

Rtx®-CLPesticides2



Columns	Rtx®-CLPesticides2 30 m, 0.32 mm ID, 0.25 µm (cat.# 11324) and Rtx®-CLPesticides 30 m, 0.32 mm ID, 0.32 µm (cat.# 11141) using Rxi® guard column 5 m, 0.32 mm ID (cat.# 10039) with deactivated universal "Y" Press-Tight® connector (cat.# 20405-261)
Sample	Haloacetic acid methyl ester mix #2 (cat.# 31647) Dalapon methyl ester (cat.# 32057) Methyl-2,3-dibromopropionate (cat.# 31656) 1,2,3-Trichloropropane (cat.# 31648)
Diluent:	Methyl *tert*-butyl ether (MTBE)
Injection	
Inj. Vol.:	1.0 µL splitless (hold 0.75 min)
Liner:	Cyclo double taper (4 mm) (cat.# 20896)
Inj. Temp.:	250 °C
Oven	
Oven Temp:	35 °C (hold 4 min) to 250 °C at 15 °C/min (hold 5 min)
Carrier Gas	He, constant flow
Linear Velocity:	25 cm/sec
Detector	µ-ECD @ 300 °C
Notes	This chromatogram was obtained using an Agilent µ-ECD. To obtain comparable results, you will need to employ a µ-ECD in addition to confirmational dual columns connected to a 5-meter guard column using a "Y" Press-Tight® connector.

EDB, DBCP, and TCP

1,2-Dibromoethane (EBD), 1,2-dibromo-3-chloropropane (DBCP), and 1,2,3-trichloropropane (TCP) are common byproducts created when drinking water is chlorinated. EPA Methods 504.1 and 8011 were developed for the analysis of EBD, DBCP, and TCP in drinking water. A 35 mL sample of drinking water is extracted with 2 mL of hexane and analyzed by capillary gas chromatography using ECD detection. Figure 19 shows example chromatograms of these compounds on the Rtx®-CLPesticides columns using optimized analysis conditions. Restek provides a complete range of calibration standards for these compounds.

Certified Reference Materials

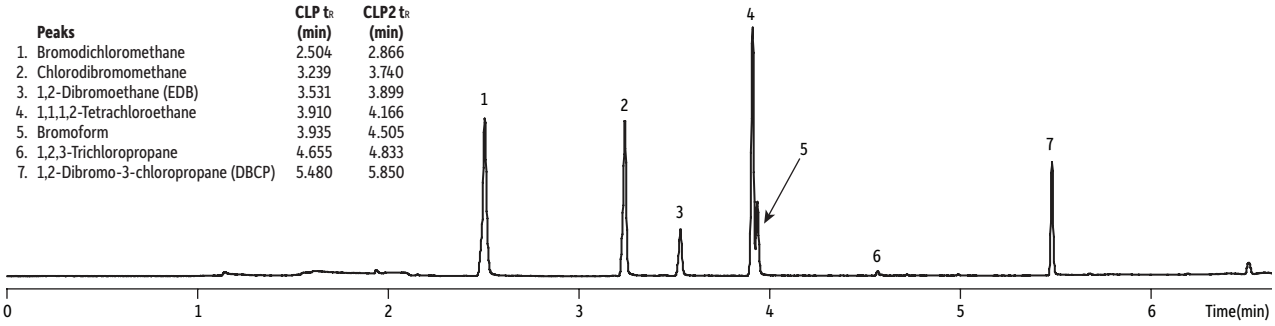
See **page 28** or visit

www.restek.com/**reference**

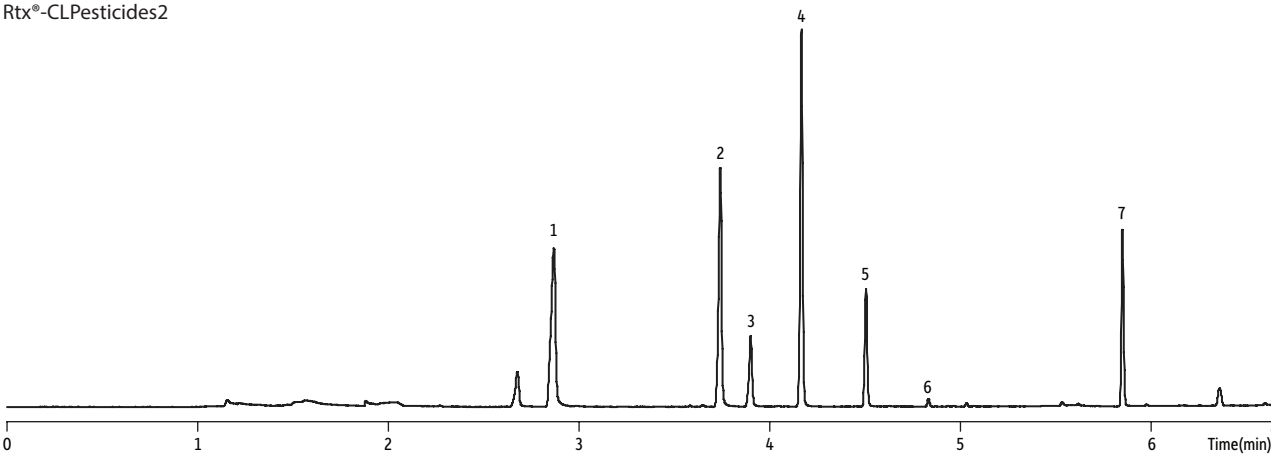
Figure 19: Analysis of EDB, DBCP, and TCP on the Rtx®-CLPesticides column set.

Rtx®-CLPesticides

Peaks	CLP t_R (min)	CLP2 t_R (min)
1. Bromodichloromethane	2.504	2.866
2. Chlorodibromomethane	3.239	3.740
3. 1,2-Dibromoethane (EDB)	3.531	3.899
4. 1,1,1,2-Tetrachloroethane	3.910	4.166
5. Bromoform	3.935	4.505
6. 1,2,3-Trichloropropane	4.655	4.833
7. 1,2-Dibromo-3-chloropropane (DBCP)	5.480	5.850



Rtx®-CLPesticides2



GC_EV1227

Columns	Rtx®-CLPesticides2 30 m, 0.32 mm ID, 0.25 µm (cat.# 11324) and Rtx®-CLPesticides 30 m, 0.32 mm ID, 0.32 µm (cat.# 11141) using Rxi® guard column 5 m, 0.32 mm ID (cat.# 10039) with Universal "Y" Press-Tight® connector (cat.# 20405)
Sample	Dibromochloromethane (chlorodibromochloromethane) (cat.# 30271) Bromodichloromethane (cat.# 30251) 504.1 calibration mix (cat.# 30239) 1,1,1,2-tetrachloroethane (cat.# 30411) Bromoform (cat.# 30252)
Diluent:	*n*-Hexane
Conc.:	10 ng/mL
Injection	
Inj. Vol.:	2 µL splitless (hold 0.50 min)
Liner:	Restek Premium 4 mm single taper inlet liner w/wool (cat.# 23303.1)
Inj. Temp.:	200 °C
Purge Flow:	50 mL/min

Oven	
Oven Temp:	30 °C (hold 2.0 min) to 220 °C at 30 °C/min
Carrier Gas	He, constant flow
Linear Velocity:	60 cm/sec
Detector	µ-ECD @ 220 °C
Make-up Gas Flow Rate:	50 mL/min
Make-up Gas Type:	He
Data Rate:	50 Hz
Instrument	Agilent/HP6890 GC
Notes	This chromatogram was obtained using an Agilent µ-ECD. To obtain comparable results, you will need to employ a µ-ECD in addition to confirmational dual-columns connected to a 5-meter guard column using a "Y" Press-Tight® connector.

Summary

Although the analysis of chlorinated pesticides and herbicides historically has been one of the more difficult tests performed by environmental testing laboratories, using Restek's Rtx®-CLPesticides column set, coupled with the methods and recommendations presented in this guide, will make your analyses easier and allow you to perform other environmental testing, such as PCB analysis, using the same columns and GC setup. Careful sample preparation and extract cleanup, proper injection technique, and suitable analytical columns and standards will improve your results and increase your lab's throughput.

When problems occur, using proper troubleshooting and maintenance techniques can quickly re-establish system integrity. When faced with difficulties in your pesticide or herbicide analysis, remember that the majority of problems occur during the sample preparation and cleanup steps, or at the injection port of the GC. If you are still having difficulties with your analysis after following the steps in this guide, please contact Restek's technical service at support@restek.com; 1-800-356-1688, ext. 4; or 1-814-353-1300, ext. 4 and we will be happy to help you.

Using Restek's Rtx®-CLPesticides column set, coupled with the methods and recommendations presented in this guide, will make your analyses easier and allow you to perform other environmental testing, such as PCB analysis, using the same columns and GC setup.

Sample Preparation

Glassware

Soxhlet Extraction Apparatus

Soxhlet extraction is used for the continuous solvent extraction of organic analytes from a solid matrix. All parts are connected with ST joints to reduce any risk of contamination. All flask joints are ST 24/40 joints.

Description	ID	Volume	Taper Size	qty.	cat.#
Extraction Apparatus	30 mm	125 mL	34/45	kit	23342
Extraction Apparatus	40 mm	250 mL	45/50	kit	23343
Extraction Apparatus	50 mm	300 mL	55/50	kit	23344

Separatory Funnels, Squibb Type with PTFE Stopcock

With solid ST stopper.

Description	Volume	Stopcock	qty.	cat.#
Separatory Funnel, Squibb Type	30 mL	2 PTFE	kit	23381
Separatory Funnel, Squibb Type	60 mL	2 PTFE	kit	23382
Separatory Funnel, Squibb Type	125 mL	2 PTFE	kit	23383
Separatory Funnel, Squibb Type	250 mL	4 PTFE	kit	23384
Separatory Funnel, Squibb Type	500 mL	4 PTFE	kit	23385
Separatory Funnel, Squibb Type	1 L	4 PTFE	kit	23386
Separatory Funnel, Squibb Type	2 L	6 PTFE	kit	23387

Kuderna-Danish Evaporator Concentrator

This apparatus is used to concentrate analytes from volatile solvents. Apparatus consist of a 3-ball Snyder distilling column, flask and concentrator tube. The flask and receivers are held together by ST joints and the included poly joint clamp. Concentrator tube is graduated.

Description	Volume	Receiver Capacity	qty.	cat.#
Kuderna-Danish Evaporator	250 mL	10 mL	kit	23339
Kuderna-Danish Evaporator	500 mL	15 mL	kit	23340

Concentrator Tube

Description	Volume	Taper Size	qty.	cat.#
Graduated Concentrator Tube	10 mL	19/22	ea.	23341

Solid Phase Extraction

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).

	3 mL/500 mg (50-pk.)	6 mL/500 mg (30-pk.)	6 mL/1,000 mg (30-pk.)	15 mL/2 g (15-pk.)
Florisil (EPA SW 846 methods and CLP protocols)	24031 24032*	26086**	24034 26085**	26228

*PTFE frits **Glass tubes with PTFE frits

All cartridges are manufactured using high density polypropylene and have polyethylene frits unless otherwise noted.

Cartridges may be processed by any one or all of these techniques: positive pressure, sidearm flask, centrifuge, or vacuum manifold.

▶ Visit **www.restek.com** for additional products and services

Excellent for Pesticide Residue Cleanup!

Resprep® CarboPrep® SPE Cartridges

- Improved recovery of sulfonylurea herbicides, phenols, carbamates, and triazine herbicides, compared to C18 and C8 cartridges.
- Wide range of selectivity for both analytes and their metabolites or degradation products.
- Rapid sampling flow rates; uncompromised recoveries.
- Maximum capacity for contaminant cleanup.
- Controlled manufacturing improves cleanliness, ensures reproducible performance.

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

Resprep® disks & flow filters extract analytes of interest at high flow rates and significantly reduce clogging.

Resprep®-C18 SPE Disks

- Glass fiber disks embedded with C18 bonded silica.
- Extract semivolatile organic compounds.
- Deep-pore design reduces clogging and allows faster flow rates.
- Meet requirements for EPA Methods 525.1, 506, 550.1, and 549.1.
- Lower cost than PTFE disks.

Description	Diameter	qty.	cat.#
Resprep-C18	47 mm	20-pk.	24004
Resprep-C18	90 mm	12-pk.	25988

Resprep® SPE Flow Filters

- Designed specifically to improve flow when filtering oil and grease samples.
- Use with Resprep® Diskcover-47 reservoir, or any 47 mm glass sample reservoir.

Description	qty.	cat.#
Resprep SPE Flow Filters	20-pk.	26024

Sodium Sulfate (Bulk Adsorbent)

- Ideal for removing water from sample extracts.
- Activate by heating to 400 °C for four hours before use.
- Packaged in recloseable 5 kg buckets.

Anhydrous sodium sulfate is the most common drying agent used to remove moisture from sample extracts. We package our 60 mesh material in recloseable buckets.

Description	qty.	cat.#
Sodium Sulfate	5 kg	26204

Florisil® PR (Bulk Adsorbent)

- Pesticide residue grade.
- Packaged in glass containers.

Florisil® PR is commonly used to remove polar interferences from pesticide residues. This bulk material is ideal for labs packing their own extraction cartridges for pesticide residue extractions.

Description	qty.	cat.#
Florisil PR, 60/100 mesh	500 g	26135

Granulated Activated Copper (Bulk Adsorbent)

- Convenient form for removing sulfur from environmental extracts.
- Acidified and activated—ready for use.

Activated copper effectively removes elemental sulfur from environmental extracts. Our acid-washed and activated material can be used right out of the package. The 30 mesh granular material eliminates the potential for fine copper particles in filtered extracts.

Description	qty.	cat.#
Granulated Activated Copper, 30 mesh	1 kg	26136

Accelerated Solvent Extraction

Extraction Cell Bodies for ASE® 200 Systems

- Cell bodies are serialized for easy sample identification.
- Inner surfaces polished for easier cleaning.

Extraction Cell Body	Similar to Dionex part #	Stainless Steel qty.	cat.#
1 mL for ASE 200	054973	ea.	26110
5 mL for ASE 200	054974	ea.	26112
11 mL for ASE 200	048820	ea.	26114
22 mL for ASE 200	048821	ea.	26098
33 mL for ASE 200	048822	ea.	26116

Cell bodies are serialized for easy sample identification.

Extraction Cell Caps & Replacement Parts for ASE® 200 Systems

- Inner surfaces polished for easier cleaning.
- Caps include frit, washer, PTFE O-ring, and threaded insert.

Description	Similar to Dionex part #	Stainless Steel qty.	cat.#
Replacement Extraction Cell End Caps for ASE 200	049450	2-pk.	26096
Cap Inserts for ASE 200		2-pk.	26166
Replacement Frits for ASE 200	049453	10-pk.	26100
Replacement Frits for ASE 200	049453	100-pk.	25959

Description	Similar to Dionex part #	qty.	cat.#
Snap Rings for Caps for ASE 200	049456	10-pk.	26184
Funnel for ASE 200	056958	ea.	26180
PTFE O-Rings for ASE 200 & ASE 300 Caps	049457	100-pk.	26187
Viton O-Rings for ASE 200 & ASE 300 Caps	056325	50-pk.	26188

▶ Other ASE® 200 and ASE® 300 replacement parts are available at **www.restek.com/ase**

Reference Standards

▶ Additional reference standards for chlorinated pesticides, PCBs, and chlorinated herbicides are available at **www.restek.com**

Organochlorine Pesticide Mix AB #1 (20 components)

aldrin	*cis*-chlordane	dieldrin	endrin aldehyde
α-BHC	*trans*-chlordane	endosulfan I	endrin ketone
β-BHC	4,4'-DDD	endosulfan II	heptachlor
δ-BHC	4,4'-DDE	endosulfan sulfate	heptachlor epoxide (isomer B)
γ-BHC (lindane)	4,4'-DDT	endrin	methoxychlor

200 µg/mL each in hexane:toluene (1:1), 1 mL/ampul — cat.# 32291

Organochlorine Pesticide Mix AB #2 (20 components)

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

In hexane:toluene (1:1), 1 mL/ampul — cat.# 32292

Pesticide Surrogate Mix (2 components)

decachlorobiphenyl 2,4,5,6-tetrachloro-*m*-xylene

200 µg/mL each in acetone, 1 mL/ampul — cat.# 32000

200 µg/mL each in acetone, 5 mL/ampul — cat.# 32457

Organochlorine Pesticide System Evaluation Mix (2 components)

- Designed for daily assessment of system performance.
- Reveals active sites in the injection port and/or GC column.
- Prepared in MTBE—low expansion volume helps minimize backflash.

4,4'-DDT 200 µg/mL endrin 100 µg/mL

In methyl *tert*-butyl ether, 1 mL/ampul — cat.# 32417

Instrument Supplies

Guard Columns and Connectors

Deactivated guard columns minimize breakdown and improve recovery of analytes!

Rxi® Guard/Retention Gap Columns (fused silica)

- Extend column lifetime.
- Excellent inertness—obtain lower detection limits for active compounds.
- Sharper chromatographic peaks by utilizing retention gap technology.
- Maximum temperature: 360 °C.

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

Intermediate-Polarity Deactivated Guard/Retention Gap Columns/ Transfer Lines (fused silica)

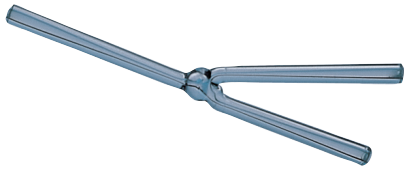
- Tested with a comprehensive test mix to ensure high inertness.
- Useful for a wide range of applications.
- Use with most common solvents.
- Maximum temperature: 325 °C

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.
0.53 mm	0.69 ± 0.05 mm	10045	10045-600

Guard Columns and Connectors, cont.

Press-Tight® Connectors

- Deactivated Press-Tight® connectors maintain complete inertness along the GC flow path.
- Fit 0.33–0.74 mm OD columns (Restek 0.1–0.53 mm ID).



Universal Angled "Y" Press-Tight® Connectors

- Perform confirmation analysis with a single injection.
- Inlet and outlet ends conform to the column curvature—alleviates column-end connection strain.

Description	ea.	3-pk.
Universal Angled "Y" Press-Tight Connector	20403	20404
Universal Angled "Y" Press-Tight Connector, Deactivated	20403-261	20404-261

Columns and Kits

Rtx®-CLPesticides/Rtx®-CLPesticides2

- Application-specific columns for organochlorine pesticides and herbicides.
- Low bleed—ideal for GC-ECD or GC-MS analyses.
- Baseline separations in less than 10 minutes.
- Stable to 340 °C.
- Analyze EPA Method 8081B, 8082A, 8151A, 504.1, 515, 508.1, and 552.2 compounds without time-consuming column change.

Rtx®-CLPesticides Columns (fused silica)
(proprietary Crossbond® phases)

ID	df	temp. limits	15-Meter	20-Meter	30-Meter	60-Meter
0.18 mm	0.18 µm	-60 to 320/340 °C		42102		
0.25 mm	0.25 µm	-60 to 320/340 °C	11120		11123	11126
0.32 mm	0.32 µm	-60 to 320/340 °C			11141	
	0.50 µm	-60 to 320/340 °C	11136		11139	
0.53 mm	0.50 µm	-60 to 300/320 °C	11137		11140	

Rtx®-CLPesticides2 Columns (fused silica)
(proprietary Crossbond® phases)

ID	df	temp. limits	10-Meter	15-Meter	20-Meter	30-Meter	60-Meter
0.18 mm	0.14 µm	-60 to 320/330 °C	42301		42302		
0.25 mm	0.20 µm	-60 to 320/340 °C				11323	11326
0.32 mm	0.25 µm	-60 to 320/340 °C		11321		11324	
	0.50 µm	-60 to 320/340 °C				11325	
0.53 mm	0.42 µm	-60 to 300/320 °C		11337		11340	

▶ Visit **www.restek.com** for additional products and services

Inlet Supplies and Accessories

Restek Super Clean® Gas Filter Kits and Replacements

- High-purity output ensures 99.9999% pure gas (at max. flow of 2 L/min).
- "Quick connect" fittings for easy, leak-tight cartridge changes.
- Glass inside to prevent diffusion; polycarbonate housing outside for safety.
- All traps measure 10 5/8" x 1 3/4" (27 x 4.4 cm).
- Each base plate unit measures 4" x 4" x 1 7/8" (10.2 x 10.2 x 4.8 cm).

Description	qty.	cat.#
Carrier Gas Cleaning Kit Includes: mounting base plate, 1/8" inlet/outlet fittings, and oxygen/moisture/hydrocarbon triple gas filter	kit	22019
Replacement Triple Gas Filter (removes oxygen, moisture, and hydrocarbons)	ea.	22020
Helium-Specific Carrier Gas Cleaning Kit Includes: mounting base plate, 1/8" inlet/outlet fittings, and oxygen/moisture/hydrocarbon helium-specific filter	kit	21983
Replacement Helium-Specific Gas Filter (removes oxygen, moisture, and hydrocarbons)	ea.	21982

Extend column lifetime!

Dual Vespel® Ring Inlet Seals Washerless, leak-tight seals for Agilent® GCs

- Does not require a separate washer.
- Requires less torque to seal.
- Does not require retightening of reducing nut after several oven cycles.
- Extends column lifetime by preventing oxygen from reaching the column.
- Same price as the regular inlet seals with washers.

0.8 mm ID Dual Vespel Ring Inlet Seal	2-pk.	10-pk.	50-pk.
Gold-Plated	21240	21241	23418

Restek Premium Inlet Liners for Agilent GCs

Splitless Liners for Agilent GCs	ID OD x Length	Similar to Agilent part #	ea.	cat.# 5-pk.	25-pk.
 4 mm Single Taper	4.0 mm 6.5 mm x 78.5 mm	5181-3316 (ea.) 5183-4695 (5-pk.) 5183-4696 (25-pk.)	23302.1	23302.5	23302.25
 4 mm Single Taper w/Wool	4.0 mm 6.5 mm x 78.5 mm	5062-3587 (ea.) 5183-4693 (5-pk.) 5183-4694 (25-pk.)	23303.1	23303.5	23303.25
 4 mm Double Taper	4.0 mm 6.5 mm x 78.5 mm	5181-3315 (ea.) 5183-4705 (5-pk.) 5183-4706 (25-pk.)	23308.1	23308.5	23308.25
 4 mm Cyclo Double Taper	4.0 mm 6.5 mm x 78.5 mm		23310.1	23310.5	23310.25
Direct Injection Liners for Agilent GCs (for 0.25/0.32/0.53mm ID Columns)	**ID OD x Length**	**Similar to Agilent part #**	**ea.**	**cat.# 5-pk.**	**25-pk.**
 Drilled Uniliner (hole near top)	4.0 mm 6.3 mm x 78.5 mm		23311.1	23311.5	23311.25

Patent pending

Restek Electronic Leak Detector

Don't let a small leak turn into a costly repair—protect your analytical column by using a Restek leak detector.

Features & benefits include:

- Audible tone indicates the severity of a leak.
- Redesigned circuitry offers 12 hours of operation between charges.
- Detects a broad range of gases; EX rated for use with hydrogen and other explosive gases.*
- Ergonomic, handheld design.
- Rugged side grips for added durability.
- Handy probe storage for cleanliness and convenience.
- Long-lasting battery; up to 12 hours of continuous use.
- Automatic shutoff.
- A convenient hard-sided carrying and storage case.
- Easy-to-clean probe assembly.
- A universal charger set (U.S., European, UK, and Australian plugs included).

Backed by a one-year warranty, the Restek leak detector is the industry standard for performance and affordability in handheld leak detectors.

22655

Leak Detector Specifications

Detectable Gases:	Helium, nitrogen, argon, carbon dioxide, hydrogen
Battery:	Rechargeable lithium ion internal battery pack (12 hours normal operation)
Operating Temperature Range:	32–120 °F (0–48 °C)
Humidity Range:	0–97%
Warranty:	One year
Certifications:	CE, Ex, Japan
Compliance:	WEEE, RoHS

Limits of Detection

These gases can be detected with the Restek electronic leak detector at the following leak rates:

Minimum Detectable Gas Limits and Indicating LED Color:
Helium, 1.0 x 10^{-5}, red LED
Hydrogen*, 1.0 x 10^{-5}, red LED
Nitrogen, 1.4 x 10^{-3}, yellow LED
Argon, 1.0 x 10^{-4}, yellow LED
Carbon dioxide, 1.0 x 10^{-4}, yellow LED

Gas detection limits measured in atm cc/sec.

22657

Optional soft-side storage case is ideal for storing your leak detector or flowmeter in smaller spaces, such as your toolbox.

Description	qty.	cat.#
Leak Detector With Hard-Sided Carrying Case and Universal Charger Set (U.S., UK, European, Australian)	ea.	22655
Small Probe Adaptor for Leak Detector	ea.	22658
Dynamic Duo Combo Pack (Restek Leak Detector and ProFLOW 6000 Flowmeter)	kit	22654
Soft-Sided Storage Case for Leak Detector or ProFLOW 6000 Flowmeter	ea.	22657

22658

Verify hard-to-reach leaks using the small probe adaptor (sold separately).

Avoid using liquid leak detectors on a GC! Liquids can be drawn into the system and/or into the leak detector.

*Caution: The Restek electronic leak detector is designed to detect trace amounts of hydrogen in a noncombustible environment. It is NOT designed for determining leaks in a combustible environment. A combustible gas detector should be used for determining combustible gas leaks under any condition. When using it to detect hydrogen, the Restek electronic leak detector may only be used for determining trace amounts in a GC environment.

▶ Visit **www.restek.com** for additional products and services

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