

Optimizing the Analysis of Volatile Organic Compounds



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Introduction

Optimizing the Analysis of Volatile Organic Compounds

One of our standing goals is to provide you with practical technical information to help you obtain reliable data from your chromatographic and peripheral systems. This guide presents information on the common US Environmental Protection Agency (EPA) gas chromatography (GC) methods and procedures used to analyze volatile organic compounds (VOCs). It is a compilation of information based on our experience and that of experts in this field. Much of this guide is dedicated to discussing purge and trap techniques, and showing applications using a variety of configurations and conditions.

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We hope you enjoy reading this guide and find it useful in your work. If you have any questions, or have input for future editions, please don't hesitate to contact Restek Corporation - we'll be happy to hear from you.



Christopher English
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EPA Method Definitions

Many EPA methods have been developed for the analysis of VOCs. Virtually all VOC methods employ purge and trap techniques to concentrate the volatiles from the sample matrix. The type of sample matrix being analyzed determines which method is used. We will discuss drinking water methods (500 series), wastewater methods (600 series), hazardous waste methods (8000 series), and Contract Laboratory Program (CLP) methods. In addition, we will discuss state gasoline range organic (GRO) methods.

Drinking Water Methods (500 Series)

Proposed in 1973 by the EPA and passed by Congress a year later, the Safe Drinking Water Act (SDWA) establishes national standards for drinking water from surface and ground water sources. These methods regulate the analysis of trace-level organic pollutants in drinking water. Enforcement of the SDWA provides that states shall have the primary authority, while the EPA will oversee activities pertaining to the public water supply system. These methods have evolved over the years, which has resulted in a growing list of compounds of interest in the subsequent revisions.

Method 502.2: This capillary column GC method is used to monitor 60 regulated volatile contaminants in drinking water. It employs a purge and trap concentrator, combined with a photoionization detector (PID) and an electrolytic conductivity detector (ELCD) in series. The PID detects aromatic and double-bond compounds, and the ELCD detects halogenated compounds.

Method 504: This capillary column GC method is used to monitor ethylene dibromide (EDB) and dibromochloropropane (DBCP) in drinking water. It employs microextraction, using hexane, and analysis using an electron capture detector (ECD).

Method 524.2: This capillary column GC/mass spectroscopy (GC/MS) method is used to monitor the same 60 drinking water contaminants listed in Method 502.2. It also employs purge and trap concentration, but uses the MS to determine both aromatic and halogenated compounds.

Method 524.2, Revision IV: This capillary column GC/MS method is used to monitor the 60 compounds listed in Methods 524.2 and 502.2, plus 24 additional compounds. As of Fall 2001, revisions were proposed to replace hydrochloric acid sample preservation with sodium thiosulfate. These revisions, however, were not promulgated at the time of this printing.

Wastewater Methods (600 Series)

In 1977, President Carter signed the Clean Water Act (CWA) allowing the EPA to study and, if necessary, regulate 65 priority wastewater pollutants. A cooperative effort between environmental laboratories and the EPA resulted in the final version of what are now known as the 600 series methods. These methods regulate the analysis of organic pollutants in industrial and municipal wastewater discharges. They were written for packed GC columns, but most environmental laboratories now use capillary column technology.

Method 601: This GC method was developed to monitor 29 halogenated volatile pollutants in wastewater. It employs purge and trap concentration combined with an ELCD.

Method 602: This GC method was developed to monitor seven aromatic volatile pollutants in wastewater. It employs purge and trap concentration combined with a PID. Many laboratories combine Methods 601 and 602 by using a PID and an ELCD connected in series.

Method 624: This GC/MS method uses purge and trap concentration to monitor 35 halogenated and aromatic volatile pollutants in wastewater.

Method 1624: This isotope dilution GC/MS method uses purge and trap concentration to monitor 58 volatile pollutants in wastewater. Stable, isotopically labeled analogs of the target compounds are added to correct for analyte recoveries that might vary due to matrix interference in the analyzed samples.

Drinking Water Disinfection Byproducts

1996 amendments to the SDWA require the EPA to review and revise existing National Primary Drinking Water Regulations (NPDWR) at least once every six years. Much of this renewed interest in changes to drinking water regulation standards stems from studies suggesting negative reproductive effects, such as spontaneous abortions, resulting from trihalomethanes (THMs) in water. Current studies using compliant levels of THMs in water have revealed adverse reproductive effects, therefore method detection limits (MDLs) will continue to be lowered in methods that address THMs.¹

1. S. Richardson, *Anal. Chem.* 73 (2001) 2719-2734.

Hazardous Waste Methods (8000 Series)

The Resource Conservation and Recovery Act (RCRA) of 1976 was enforced shortly after front-page headlines revealed the presence of serious hazardous waste sites like Love Canal, NY and Times Beach, MO. The analytical methods for determining hazardous waste, known as the 8000 series methods, fall under US EPA SW-846. These methods were designed for monitoring organic pollutants in waste samples prior to disposal at hazardous waste facilities. They also can be used for monitoring groundwater at these facilities.

Method 8010B: This packed column GC method is used to monitor 50 halogenated volatile pollutants in hazardous waste samples. It employs purge and trap concentration and an ELCD.

Method 8011: This capillary column GC method is used to monitor 1,2-dibromoethane (EDB) and 1,2-dibromo-3-chloropropane (DBCP) in hazardous waste samples. It employs microextraction, using hexane, and analysis using an ECD.

Method 8015A: This packed column GC method is used to monitor non-halogenated volatile pollutants in hazardous waste samples. It employs purge and trap concentration and an FID. Total petroleum hydrocarbon analysis, commonly referred to as 8015-TPH, also falls under this method. Method 8015-TPH uses an FID to match a known pattern of gasoline with an unknown sample containing peaks that fall within the gasoline pattern range. If a pattern falls within the gasoline window it may be reported as gasoline.

Method 8020A: This packed column GC method is used to monitor ten aromatic volatile pollutants in hazardous waste samples. It employs purge and trap concentration and a PID. It is common for analysts to combine Methods 8010 and 8020, by using a PID and an ELCD in series.

Method 8021A: This capillary column GC method is used to monitor 60 volatile contaminants in hazardous waste samples. It employs purge and trap concentration, combined with a PID and an ELCD in series. The PID detects aromatic compounds and double-bond compounds, and the ELCD detects halogenated compounds.

Method 8021B: Using the same analytical technique as Method 8021A, the compound list for Method 8021B includes ten additional compounds but does not require the analysis of several branched aromatics and halogenated compounds.

Method 8240B: This packed column GC/MS method is used to monitor 79 volatile pollutants in hazardous waste samples. It employs purge and trap concentration for most analytes, but direct injection can be used for some limited applications.

Method 8260B: This capillary column GC/MS method is used to monitor 98 volatile pollutants in hazardous waste samples. It employs purge and trap concentration for most analytes, but direct injection can be used for some limited applications.

State GRO Methods

Leaking underground storage tanks (LUST) pose significant environmental risks throughout the country. States have the responsibility to develop LUST testing methods. State gasoline range organics (GRO) methods are based on EPA methods such as 602, 8020 and 8015. The most common EPA method used is 8015, which relies on baseline-integrating the total area of the gasoline fingerprint, using marker compounds such as hexane (C6) and dodecane (C12). The 8015-TPH Method analysis uses an FID and pattern recognition—the specific ratio of peaks that make up a particular fuel—to identify the type of fuel. If a pattern falls within the window markers it may be reported as gasoline, then quantified. Difficult matrices can result in misidentification or poor quantitation of the sample, and deterioration in the environment (weathering) further complicates the analysis. Therefore, many states have combined EPA methods, using a PID/FID in series (e.g., Methods 8020/8015-TPH). Specific aromatic compounds are analyzed using PID (Methods 602, 8020), which is connected to the FID (Method 8015-TPH). The common target compounds are benzene, toluene, ethylbenzene, and *m*-, *o*-, and *p*-xylene (BTEX), however many states also have added other compounds to their methods (Table I).

Table I.

State gasoline methods include specific compounds.

State	Method-Specific Compounds
Alaska (AK101AA)	BTEX, branched aromatics
Arizona	BTEX, C6-C12
California	BTEX, MTBE
California (WIP)	Method 8020, MTBE
Connecticut	GRO
Florida	PVOC
Georgia	GRO Method 8015B
Iowa (OA-1)	GRO, BTEX, MTBE
Louisiana	GRO (C6-C12)
Maryland	GRO Method 8015B
Massachusetts (VPH)	BTEX, <i>m</i> -naphthalene, MTBE, etc.
Michigan (GRO)	BTEX, <i>m</i> -naphthalene, MTBE, etc.
Mississippi	GRO
Missouri (OA-1)	GRO, BTEX, MTBE
Montana	Method 8015
New York	GRO Method 8015B
North Carolina	Massachusetts VPH
Oklahoma	GRO
Oregon	C5, C6, C8, C10, C12, BTEX, MTBE, etc.
Pennsylvania (DEP)	BTEX, MTBE, 1,2-dibromoethane, 1,2-dichloroethane
South Carolina	GRO Method 8015B
Tennessee	GRO
Texas (TNRCC 1005)	hexane, decane (locator mix)
Utah	BTEX, MTBE, naphthalene
Virginia	GRO Method 8015B
Washington (VPH)	C5, C6, C8, C10, C12, BTEX, MTBE, etc.
West Virginia	Method 8015B
Wisconsin	PVOC/GRO BTEX, MTBE, naphthalene, TMB, 1,3,5-TMB

Acronyms:

BTEX - benzene, toluene, ethylbenzene, xylenes

MTBE - methyl-*tert*-butylether

GRO - gasoline range organics

PVOC - petroleum volatile organic compounds

VPH - volatile petroleum hydrocarbons

TMB - trimethylbenzene.



Where can EPA methods be obtained?

Drinking Water Methods (500 Series)

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
703-487-4600

Wastewater Methods (600 Series)

Environmental Monitoring and Support Laboratory
U.S. EPA
Cincinnati, OH 05268
513-569-7562

Hazardous Waste Methods (8000 Series)

U.S. Government Printing Office
Washington, DC 20402
202-783-3238

Websites:

U.S. EPA Homepage
www.epa.gov

Federal Register Link
www.epa.gov/fedrgstr/

Ground Water/ Drinking Water (500 Series)
www.epa.gov/safewater/

Wastewater Office (600 Series)
www.epa.gov/OWM/

Solid and Hazardous Waste (8000 Series)
www.epa.gov/epaoswer/osw/

Updated List of EPA Methods and Web Locations

www.epa.gov/region01/oarm/testmeth.pdf

Contract Laboratory Program (CLP)

In 1980 the US Congress addressed the cleaning of the most contaminated abandoned and inactive dumpsites. This new legislation was known as the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) and the Superfund Amendments and Reauthorization Act (SARA). These acts required cleanup of the sites and the prosecution of those responsible for the contamination. The methods monitor volatile pollutants at Superfund sites.

Method OLM04.1 (04.2): The US EPA has awarded contracts for organic low-medium (OLM) concentration samples within the Superfund program under the 04.2 revision Statement of Work (SOW). This is a capillary column GC/MS method used to monitor in hazardous waste 50 volatile pollutants that fall under CERCLA and SARA guidelines. While this method employs purge and trap concentration, direct injection can be used for higher concentration samples that require extraction with methanol.

Method OLC03.2: This new EPA Statement of Work (SOW) describes analytical methods for aqueous low concentration organics. This capillary GC/MS method adds nine new volatile compounds to the OLC03.1 target compound list (TCL), for a total of 52 compounds. Deuterated Monitoring Compounds (DMC) are introduced as a sample-by-sample accuracy indicator.

The Love Canal Scandal

In the early 1900s William T. Love started work on his dream—to build a canal between the upper and lower Niagara Rivers to generate power for a planned model city. Before the canal was a mile long, the economy failed—and with it, Love's dream. Hooker Chemical purchased the land in 1920 and for the next three decades the City of Niagara, the US Army, and Hooker dumped waste into the canal. Eventually, the dump was filled and a clay cap was placed over the waste site. Soon after, the city persuaded Hooker to sell the property for \$1 with the threat of the Constitution's imminent domain clause. Although Hooker added a lengthy disclaimer to the property deed detailing the toxic nature of the site, within two years sewer lines were dug into the clay cap that had sealed the waste from leaching to the surface. In the late 1950s, about 100 homes and a school were built near the 20,000 tons of waste (Figure 1). Heavy snow and rainfall in 1975 and 1976 caused high water levels, which exposed the 55-gallon drums (Figure 2).

Figure 2.

Four decades after dumping, toxic waste drums like these were exposed at Love Canal, NY.



Niagara Gazette reporter Michael Brown broke the story, explaining that many residents were living on a toxic waste dump. From the time the families moved in during the '50s they had noticed strange odors, and in the early '70s a tar-like substance was reported in many basements. Analysis using the 8000 series methods, and later the 600 series and CLP methods, identified 248 chemicals, including 2,3,7,8-tetrachlorodibenzo-*p*-dioxin, which is believed to be the most toxic substance known to man. Many VOCs were discovered in the ground, water, and air—most notably benzene—a known carcinogen. There were no toxicological data available for 100 of the 248 compounds. On August 2, 1978 state health officials ordered all pregnant women and children under the age of two to leave the area. A week later, with headlines across the country detailing the Love Canal disaster, President Carter approved the immediate evacuation of 221 families. That number would soar to nearly 900 families by the time this tragedy completely unfolded.

This was the first environmental disaster given daily front-line media coverage. It was a turning point for environmental awareness and ultimately helped to shape the environmental testing methods that are used today for the identification of VOCs in air, water, and soil. The combined efforts of environmental laboratories, engineering firms, and regulatory agencies have evolved since Love Canal to protect the public and ultimately save lives.

Figure 1.



Infrared aerial photo of Love Canal area (spring 1978) showing 99th Street elementary school (center), two rings of homes bordering the landfill, and LaSalle Housing Development (upper right). White patchy areas are barren sections where vegetation will not grow, presumably due to leaching chemicals.

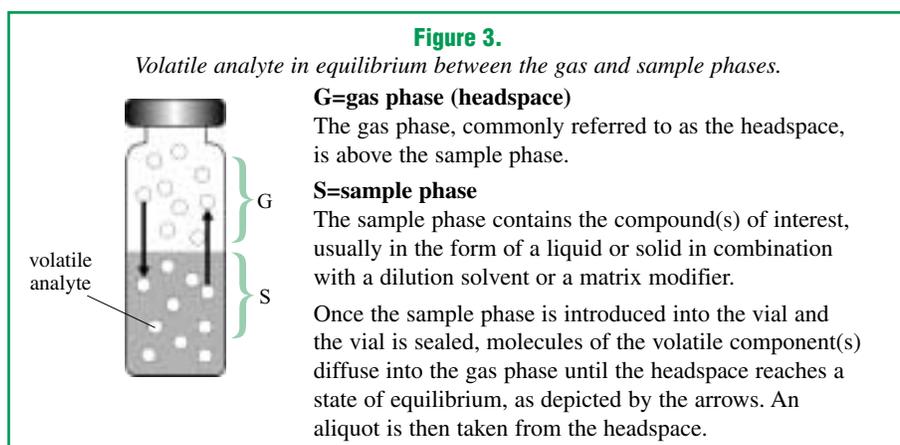
Image courtesy of State University of New York at Buffalo University Archives.

We thank Dan Di Landro, Visiting Assistant Librarian, for help with obtaining the photograph.

Purge and Trap Theory

Concentration of Volatile Organics

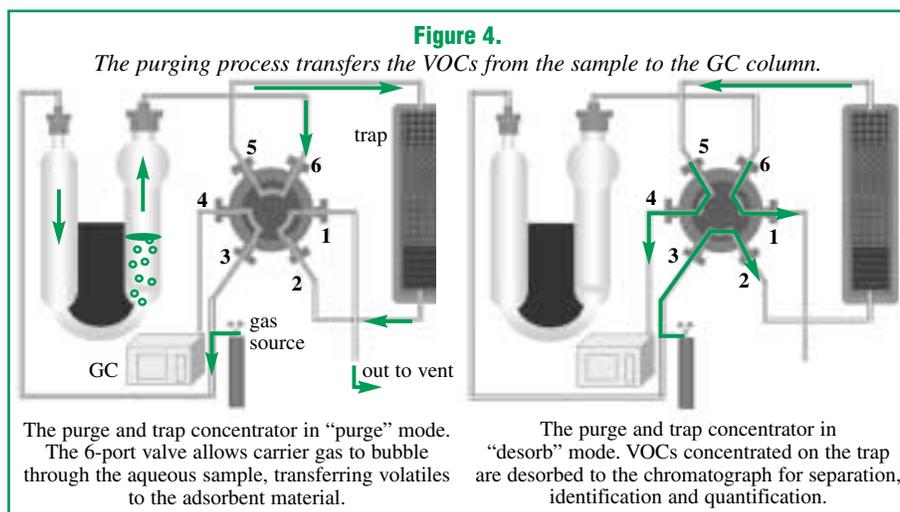
Volatile organic compounds can be concentrated by either static headspace or dynamic headspace (i.e., purge and trap) sampling. In static headspace concentration, a sample is placed in a closed sample chamber. Molecules of the volatile compounds in the sample migrate to the headspace above the sample and equilibrium is established between the concentration of the compounds in the vapor phase and in the liquid phase (Figure 3). Once equilibrium is reached, an aliquot of the headspace above the sample is injected onto the GC column. A major problem with static headspace techniques is that the sample matrix significantly affects equilibrium. Analyses for compounds that show high solubility in the sample matrix often yield low sensitivity as a result of matrix effects. Further, static headspace analysis only samples an aliquot of the volatiles (i.e., 1mL, 2mL, or whatever the size of the sample loop), which also affects sensitivity.



Purge and trap concentration is a dynamic headspace technique that reduces matrix effects and increases sensitivity, relative to static headspace techniques. Samples containing VOCs are introduced into a purge vessel and a flow of inert gas is passed through the sample at a constant flow rate for a fixed time. Volatile compounds are purged from the sample into the headspace above the sample and are transferred to and concentrated on an adsorbent trap (Figure 4). After the purging process is complete, the trap is rapidly heated and backflushed with carrier gas to desorb and transfer the analytes to the GC column.



Sample purging in progress in a Tekmar 3100 concentrator.



Sequences and Flow Paths of the Purge and Trap Unit

Purge and trap units are designed to have separate flow rates for the purge gas and the desorb (carrier) gas. The recommended gas for both purging and desorption is helium. The purge gas flow typically is set at 30-50mL/min. The desorb gas flow ranges from 10-80mL/min., depending on the column type and GC equipment used (see the Applications section of this guide for example chromatograms). The desorb gas should be controlled using a flow controller. The flow controller from the injection port of the GC commonly is used, but a separate flow controller can be connected to the desorb gas bulkhead fitting on the back of the purge and trap system. Hydrocarbon traps should be installed on the carrier gas line prior to the purge and trap system. This will prevent trace hydrocarbon or solvent "ghost peak" contamination from interfering with the analyses.

Purge and trap techniques involve the following series of steps that must be followed to ensure accurate and reproducible results:

Step 1. Standby

During the standby mode, the purge gas flow is stopped, the trap is cooled, and the system is readied for the start of an analysis. The desorb gas bypasses the trap and is directed onto the column as the carrier gas flow. The gas flow rate through the column can be measured.

Step 2. Purge (wet)

During the wet purge, the purge gas flow passes through the purge vessel, removes volatile analytes from the sample, and sweeps the analytes through the heated valve onto the adsorbent trap. The analytes are collected on the trap and the purge gas exits through the purge vent. The purge gas flow typically is set at 30-50mL/min. and can be measured at the purge vent. Samples usually are purged for 10-15 minutes. During the purge mode, the desorb (carrier) gas is directed onto the column.

Step 3. Purge (dry)

During the wet purge, a large amount of water is removed from the sample and collects on the trap. The dry purge removes the excess water that accumulated. During the dry purge, the purge gas bypasses the purge vessel and is directed to the trap. The dry purge gas removes water and carries it out the exit vent. The desorb (carrier) gas is directed onto the column. Only traps that incorporate hydrophobic adsorbents can be dry purged.

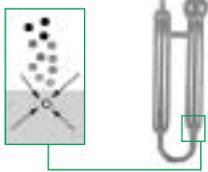
Step 4. Desorb Preheat

Once the analytes have been trapped and excess water removed, the purge gas flow is stopped. During this static period, the trap is rapidly heated to ~5°C below the desorb temperature of the adsorbent materials used. The desorb preheat step uniformly volatilizes the sample to create a narrow sample band and a more efficient sample transfer onto the GC column. Without a desorb preheat step the peaks would tail, resulting in poor chromatography. During the desorb preheat step the desorb (carrier) gas is directed onto the column.

Step 2.

Purge (wet)

- Volatiles in matrix diffuse into carrier gas as gas is bubbled (purged) through the matrix. Volatiles are transferred to the trap.
- Typical flow: 30–50mL/min. for 10–15 min.



Step 3.

Purge (dry)

- Trap is dried by purging with gas only.
- Typical time: 1-4min.



Step 4.

Desorb Preheat

- Trap is heated without flow, to minimize analyte desorb time from packing material.
- Typical temp.: 5°C below desorb temp.

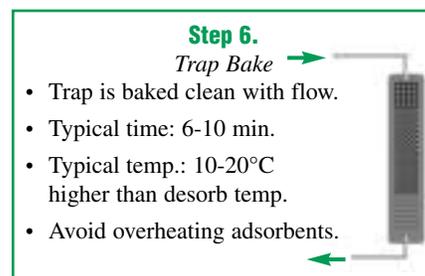
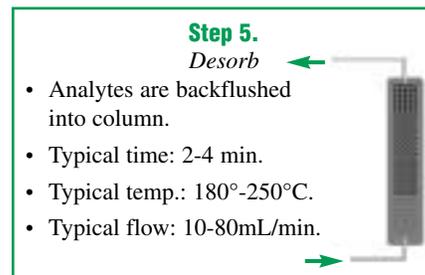


Step 5. Desorb

Once the desorb preheat temperature is reached, the purge and trap unit valve is rotated. This directs the desorb (carrier) gas flow to backflush the adsorbent trap and carry the analytes in a narrow band to the GC system. Figure 4 (page 7) shows the flow path of the desorb mode. While the sample transfer occurs, the trap is heated to its final desorb temperature. Desorb temperatures range from 180°C-250°C, determined by the adsorbent materials and the model of concentrator. The desorb flow rate is extremely important; it must be high enough to ensure that the sample remains in a narrow band during the transfer to the GC column. The optimum desorb flow rate for a purge and trap system is >20mL/min.; however, this flow rate is too high to use with capillary columns and must be reduced to retain column efficiency. The optimum flow rate for 0.53mm ID columns is 8-10mL/min. For narrow bore capillary columns (0.18-0.32mm ID), the desorb flow rate usually is 1-2mL/min. when direct interface is used. This low flow rate requires a longer desorb time due to the slow transfer of the sample from the trap, which, in turn, creates a wide sample bandwidth resulting in broad peak shapes for all early eluting compounds. Cryofocusing (i.e., cold trapping) can be used to reduce band broadening, by installing a secondary cold trap or by cooling the GC column to subambient temperatures. The desorb time is inversely proportional to flow rate and trap temperature, so that as the flow rate/trap temperature increases, the desorb time decreases, due to the analytes flushing off the trap at a higher rate. Also, it is possible to desorb at higher flow rates (25-80mL/min.), when using narrow bore capillary columns, by using a split injector to split the flow prior to the column (for more details on this technique see the GC System Configurations section, page 18).

Step 6. Trap Bake

After the desorb step the trap is baked, with gas flow, to remove any remaining sample components and contaminants from the trap in preparation for its next use. This step generally lasts 6-10 minutes; typical temperatures are 10-20°C above the desorb temperature. To prevent damage to the adsorbent materials, do not exceed the maximum temperature of the trap.



Purge and Trap Components

Purge Vessel

Three types of purge vessels (i.e., spargers) commonly are used in purge and trap systems. Frit spargers (Figure 5) are used for most water samples. The frit creates many small bubbles that travel through the sample to increase purging efficiency. Fritless spargers are used for samples that have high particulate content, or for industrial wastewater samples that may foam. They create fewer bubbles, which decreases purging efficiency but eliminates plugged frits and reduces foaming problems. Needle spargers are used when purging soil, sludge or solid samples. A narrow gauge needle is inserted into the sample and used to release a small stream of purge gas. The two common sizes of spargers are 5mL and 25mL.

Figure 5.

Purge and trap frit spargers.



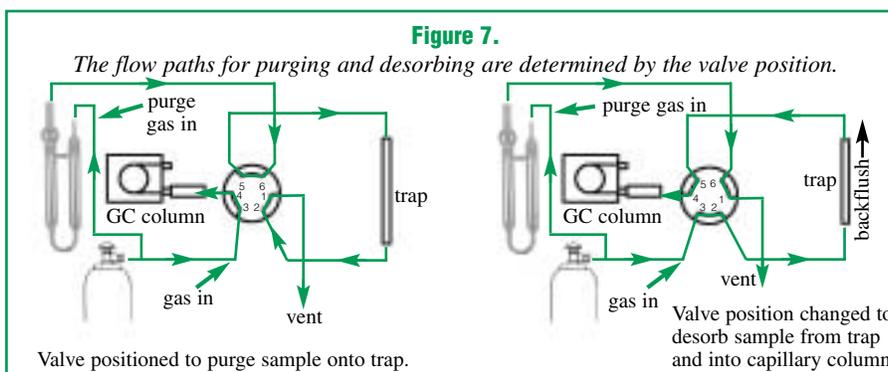
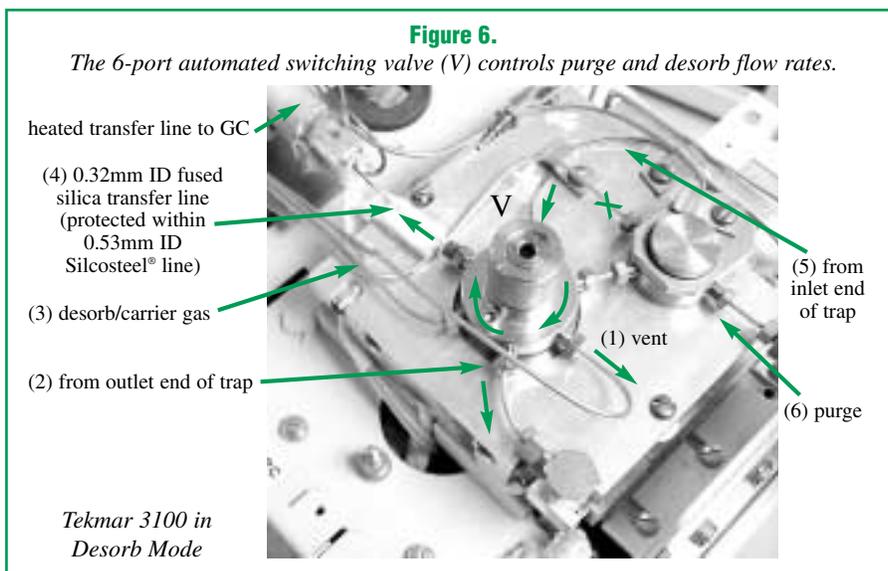
- For Tekmar 2000, 3000, or 3100.
- Available in 5mL and 25mL sizes.
- Uniform frits ensure maximum purge efficiency for water samples.
- Manufactured to tight tolerances to ensure a leak-free seal.

Description	qty.	cat.#
5mL Fritted Sparger, 1/2-inch mount	ea.	21150
25mL Fritted Sparger, 1/2-inch mount	ea.	21151

Not recommended for wastewater samples because the sample might foam or the frit might become plugged.

Valves

The purge and desorb flows are controlled by an automated switching valve (Figure 6). The valve is contained in a heated compartment to prevent sample condensation inside. By rotating the valve, the purge and desorb flow paths can be changed during the purge and trap sequence (Figure 7).



Adsorbent Materials and Traps

Adsorbent materials are used to trap the VOCs that have been purged from the sample. The adsorbent must be able to retain compounds during the entire purging sequence and then rapidly release them during the desorption step. Each adsorbent has a unique trapping capability for a specific class or classes of compounds. Therefore, a trap may have several different beds of adsorbents. The weakest adsorbent material is placed at the inlet end of the trap, then the next strongest adsorbent, and so on. The more volatile compounds pass through the weaker adsorbents and are retained by the stronger adsorbents, while the less volatile compounds are retained on the weaker adsorbents and never reach the stronger adsorbents (from which they would be difficult to desorb). Once the compounds are collected, the trap is rapidly heated and backflushed with carrier gas to drive the sample components into the GC system. Ideally, the adsorbents in the trap retain polar and non-polar analytes without retaining water or methanol, efficiently release the trapped compounds onto the analytical column, and withstand the temperatures required to desorb (i.e., “bake off”) the higher molecular weight contaminants. A list of common traps used in purge and trap concentration can help you choose the best one for your application (Table II, page 13).

Adsorbent Materials

Tenax® Adsorbent (surface area: 50m²/g): Tenax® adsorbent is excellent for trapping non-polar compounds and is hydrophobic so it does not retain water; however, it does have some disadvantages. Very volatile compounds are not retained well and must be trapped on a stronger adsorbent material. In addition, polar compounds like alcohols are poorly retained on this adsorbent. Tenax® adsorbent also has limited thermal stability; the 2,6-diphenyleneoxide polymer thermally decomposes into toluene, benzene, and other aromatics. The particles melt together and permanently adhere to the trap; this then restricts carrier gas flow. As the adsorbent degrades, there often is a loss in response for brominated compounds.

There are two grades of Tenax® adsorbent used as a trapping material: Tenax® GC and Tenax® TA (Trapping Agent) adsorbents. Common background contaminants in Tenax® GC adsorbent include benzene and toluene. Tenax® TA adsorbent is a purer form and is more commonly recommended for thermal desorption applications. The manufacturer's recommended operating temperature is 230°C but, realistically, the material performs best when kept below 200°C. Samples that contain organic acids can degrade Tenax® adsorbent. This effect is more pronounced at higher temperatures; for longer trap life and better consistency do not use traps containing this adsorbent at temperatures above 200°C.

Silica Gel (surface area: 200-800m²/g): Silica gel is a stronger adsorbent than Tenax® adsorbent. Silica gel is commonly used in conjunction with Tenax® adsorbent as a trap for volatile organic pollutants. It is an excellent trapping material for polar and highly volatile compounds that are gases at room temperature; however, silica gel is extremely hydrophilic and will retain large amounts of water. *Be aware that if a trap contains silica gel, dry purging will not reduce the water content.*

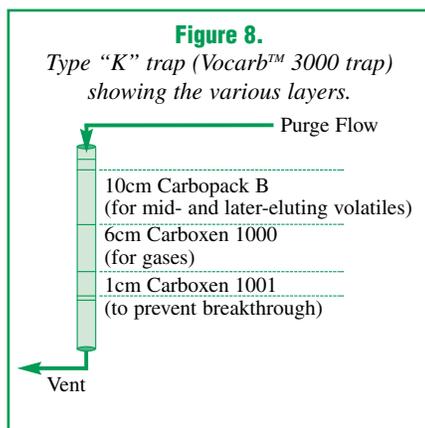
Coconut Charcoal (surface area: 900m²/g): Coconut charcoal is another strong adsorbent material. It is commonly used in series after silica gel for trapping very volatile compounds that might break through the gel. Coconut charcoal is hydrophobic, and does not retain significant amounts of water. It does, however, trap carbon dioxide (CO₂) purged from the sample, and it has been reported that charcoal is a source of CO₂, which can interfere with the quantitation of early-eluting compounds when using GC/MS systems.

Graphitized Carbon Black or Carboxen® Adsorbent (surface area: 10-100m²/g): Graphitized carbon black (GCB) is an alternative to Tenax® adsorbent. GCB is available in many pore sizes and is effective in trapping volatile organics in the same range as Tenax® adsorbent. GCB is hydrophobic and has excellent thermal stability, making it ideal for purge and trap techniques. Highly volatile compounds are not retained well on GCB and must be trapped on stronger adsorbent materials such as carbon molecular sieves.

Carbon Molecular Sieves (surface area: 50-800m²/g): Carbon molecular sieves such as Carbosieve™-SIII are alternatives to silica gel and charcoal. High surface areas make these materials ideal for trapping highly volatile compounds. They are commonly used in series after GCB because they retain compounds that break through the GCB. Carbon molecular sieves are hydrophobic and have excellent thermal stability.

Carboxen®-1000 Adsorbent (surface area: 1200m²/g): Carboxen®-1000 adsorbent is a strong adsorbent designed to be used as the innermost adsorbent bed in the trap (Figure 8, page 12). This material traps Freon® compounds, permanent gases, and light hydrocarbons. It has characteristics very similar to those of Carbosieve® S-III packing material. Carboxen®-1000 adsorbent is stable to temperatures of 300°C. Its only shortcoming is the adsorption of CO₂, which can interfere with early-eluting compounds.² Carboxen®-1001 and Carboxen®-1002 are similar materials.

2. Mosesman, N.H., W.R. Betz, and S.D. Corman. "Alternate Trapping Materials for Purge-and-Trap Analysis of Volatile Compounds." *Proc.-Water Qual. Technol. Conf. Adv. Water Anal. Treat.* 14 (1987): 245-50.



Choosing the Right Trap for Your Analysis

Type “K” Trap (Vocarb™ 3000 Trap): The most effective trap on the market is the Vocarb™ 3000 or type “K” trap (Figure 8). This trap has exceptional ability to retain highly volatile compounds like difluorodichloromethane with minimal bleed, activity, or break-down, yet it works well for trapping higher boiling compounds like naphthalene and trichlorobenzene. The trap resists adsorption of water and methanol, and virtually eliminates the need for moisture control systems (MCS) and the dry purge step on the concentrator. Because this trap contains Carboxen™ 1000 adsorbent (described on page 11), which has a surface area of over 1200m²/g, a desorb temperature of 245°C is required when using Tekmar purge and trap instruments. For OI Analytical sample concentrators, such as the Model 4560, the desorb temperature should be 220°C or lower. The lower temperature will prevent overshooting the maximum temperature of the trap, which would damage the packing materials (caused by the rapid trap heating rate, 800°C/min., of the OI system).³ When using this trap be sure to verify performance. Non-linear response for chloromethane is a sign of breakthrough and an indication that the trap must be changed. Another indication of a defective “K” trap is loss in response for acrolein.

Type “J” Trap (BTEXTRAP™ Trap): The “J” trap is excellent for concentrating gasoline range organics (GRO) because it retains less water and methanol compared to the “K” trap, and can withstand higher temperatures than the Tenax®/silica gel trap. Because many GRO samples have high concentrations of gasoline components, it is necessary to dilute the sample in methanol, and this trap can accept a heavy sample load with percent levels of methanol while still passing continuing calibration check criteria. The disadvantage of the “J” trap is its limited ability to retain more polar analytes like the ethers and alcohols. Laboratories analyzing for *tert*-butyl alcohol will attain lower detection limits by using the “K” trap, compared to the “J” trap. For GRO samples containing methyl-*tert*-butyl ether (MTBE), trap selection will depend on the sample matrix. When analyzing highly contaminated soils for MTBE, it is best to use the “J” trap. For cleaner samples, the “K” trap provides better sensitivity.

Type “B” Trap (Tenax®/Silica Gel Traps): Tenax®/silica gel traps are used for a variety of VOC methods. These traps exhibit better recoveries of polar analytes than the “K” trap, but the silica gel layer adsorbs water, methanol, and carbon dioxide. The Tenax®/silica gel trap also has better lot-to-lot reproducibility compared to the “K” or “I” traps. For laboratories that are not trying to achieve MDLs for gaseous VOCs at concentrations above 10ppb, these traps will work well. To achieve detection limits for gases at concentrations below 10ppb, the lower water and methanol retention of the “K” trap is recommended.

Type “F” Trap (OV®-1 /Tenax®/Silica Gel Traps): Although these traps are recommended in many EPA methods, they exhibit more bleed and activity than the Tenax®/silica gel trap, with no significant improvement in performance. This suggests the bleed originates from the OV-1 (methyl silicone) material.⁴ Therefore, laboratories wishing to adhere more closely to the EPA Method Protocol should choose Tenax®/silica gel traps without OV®-1.

Type “I” trap (Vocarb 4000™): The “I” trap is used for increased response for less volatile compounds such as the chloronaphthalenes and methylnaphthalenes. Generally, it is used only for applications involving analytes of larger molecular size and is not the first choice for ketones or alcohols. Common desorb times of two to four minutes should be increased with the “I” trap, to optimize sensitivity for compounds having high boiling points.

3. OI Analytical, “Volatile Organics Analysis: Building a State-of-the-Art Purge and Trap GC/MS system” Application Note 02971294.

4. OI Analytical, “Proper Trap Selection for the OI Analytical Model 4460A Purge and Trap Sample Concentrator” Application Note 12851098.

Table II.
Compositions and characteristics of common types of traps.

Description	Trap Designation	Dry Purge	Preheat (°C)	Desorb (°C)	Bake (°C)
24cm Tenax®	A	yes	175	180	200
15cm Tenax®/8cm silica gel	B	no	175	180	200
8cm Tenax®/7.7cm silica gel/7.7cm charcoal	C	no	175	180	200
16cm Tenax®/7.7cm charcoal	D	yes	175	180	200
1cm OV®-1/7.7cm Tenax®/7.7cm silica gel/ 7.7cm charcoal	E	no	175	180	200
1cm OV®-1/15cm Tenax®/7.7cm silica gel	F	no	175	180	200
1cm OV®-1/ 23cm Tenax®	G	yes	245	250	260
7.6cm Carbopack® B/1.3cm Carbosieve® S-III	H	yes	245	250	260
8.5cm Carbopack® C/10cm Carbopack® B/ 6cm Carboxen® 1000/1cm Carboxen® 1001	I (Vocarb™ 4000)	yes	245	250	260
7.7cm Carbopack® C/1.2cm Carbopack® B	J (BTEXTRAP™)	yes	245	250	260
10cm Carbopack® B/6cm Carboxen® 1000/ 1cm Carboxen® 1001	K (Vocarb™ 3000)	yes	245	250	260

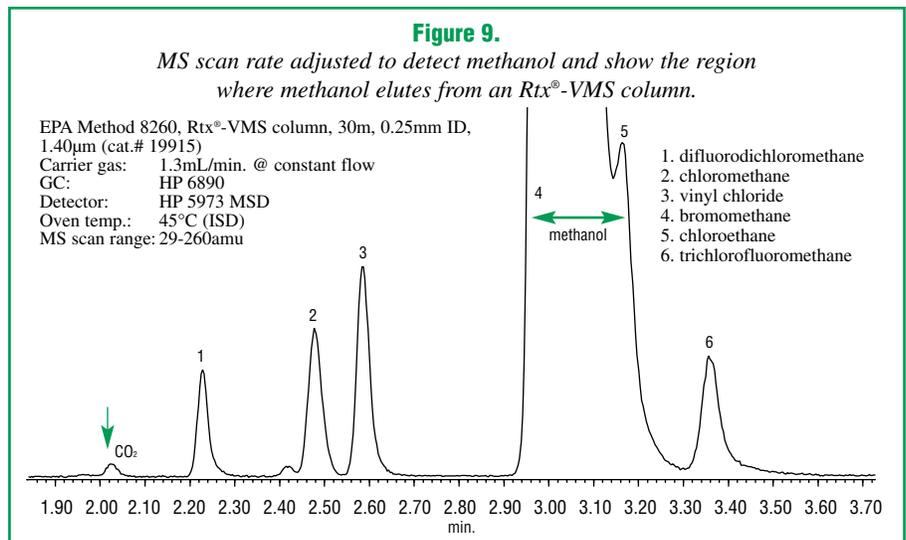
Moisture Control Systems—Water and Methanol Management

Water and methanol can cause the biggest problems in purge and trap concentration. During the desorb step, water and methanol that accumulated on the trap are released into the chromatographic system. As much as 10µL of water can accumulate on a trap containing silica gel during a purge; this expands to 12mL of water vapor during desorption.⁵ Interference caused by excess water is a problem during detection. For example, water vapor passing through a PID can cause a negative dip in the baseline. Water also can saturate a PID, decreasing its sensitivity and interfering with the identification of compounds that coelute with water. Detector saturation also can occur with MS systems. Although the lower end of the scan range typically is adjusted above the molecular weight of water, interference can still occur. If the water plug is very large, the peaks for analytes that elute in the water/methanol region will broaden and sensitivity will be reduced.

In similar fashion, methanol also causes interferences with target analytes. The PID gives a positive signal for methanol as a broad, flat-topped peak that usually interferes with 2-methylpentane, 3-methylpentane, and vinyl chloride. Adjusting the MS scan range to start above 35amu can minimize the effects of methanol (mass/charge ratio of 31amu). When using an Rtx®-VMS column, or a cyanopropylphenyl “624”-type column, methanol and chloroethane will elute simultaneously. This can affect sensitivity and linearity for chloroethane (Figure 9). When using a “502.2” phase column, methanol coelutes with bromomethane. Spiking higher concentration intermediate standards into the purge vessel or autosampler vials can minimize methanol interference. Also note that an increase in methanol added to the purge standard also inevitably increases the amount of water that purges into the system.

In recent years, much work has gone into developing hydrophobic adsorbents that minimize water collection on the trap. Extensive studies recommend incorporating a dry purge cycle to remove water from the trap prior to desorption. Current designs of purge and trap systems have added features to eliminate water prior to delivering the sample to the chromatographic system. Moisture control systems (MCS) remove water by condensation, prior to the desorb step. Such systems typically are composed of a piece of metal tubing that is heated during purge and then cooled to 30°C. The sample, desorbed from the heated trap, travels through the MCS, where a large portion of the water is condensed from the saturated carrier gas. These systems are very effective for GC methods that do not have polar/active compounds, such as ketones, in the analyte list. An older purge and trap system that does not have an MCS can be retrofitted with one. Restek offers an MCS bypass line for Tekmar 3000 and 3100 purge and trap concentrators, to increase response and maintain linearity for ketones, alcohols, and acetates (Figure 10, page 14). When analyzing samples for ketones or other polar compounds, the MCS should be bypassed to maintain linear calibration for these compounds.

5. OI Analytical, “OI Analytical Model 4560 Sample Concentrator Rapid Trap Heating” Application Note 04521297.



Transfer Line

Once the sample is desorbed from the trap, it travels through the heated transfer line to the GC. This line can be made of nickel, fused silica, or Silcosteel®-treated tubing. A heating jacket surrounds the transfer line to keep it between 120-125°C, which prevents water and analyte condensation in the line. For direct connection, we recommend matching the inside diameter of the transfer line to the inside diameter of the capillary GC column, or use of tubing of a slightly smaller inner diameter than the capillary column. This helps minimize band broadening and poor peak symmetry for sample components. Because transfer lines can be a source of active sites, use deactivated fused silica or Silcosteel®-treated tubing to reduce analyte adsorption. When using a fused silica transfer line, insert the line into a metal tube before installing it into the heated jacket. This will protect the fused silica tubing from nicks and scratches that could cause the line to break. Be sure to use the correct Valco® ferrules to minimize dead volume (see Direct Connection, page 20).

Figure 10.

An MCS bypass line can increase response and maintain linearity for ketones, alcohols, and acetates.

Moisture Control Bypass Line for Tekmar 3000 Purge & Trap

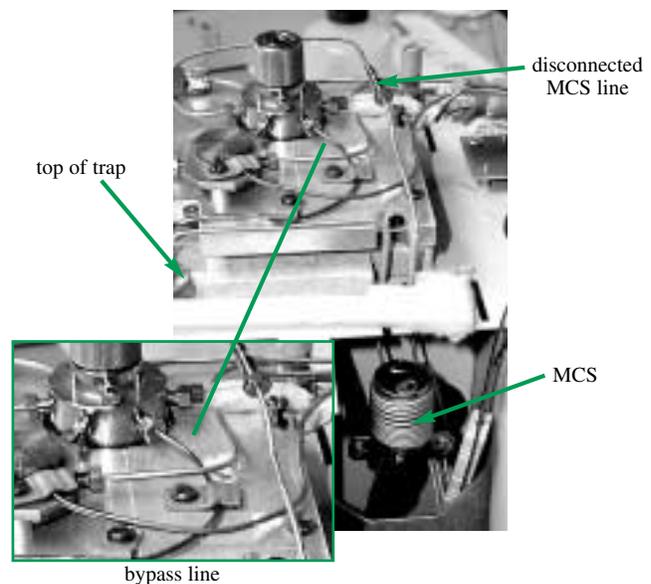
- Increases response for ketones, alcohols, and acetates.
- Suitable for US EPA Methods 8260, 524.2, and OLM4.1.
- Silcosteel® tubing for increased inertness.
- Easily attaches in minutes.



Description

Moisture Control Bypass Line

qty. cat.#
ea. 21035



Troubleshooting Common Problems Associated with Purge and Trap Units

Water: The most common problem with VOC analysis is water in the sample. Water does not create problems with the purge and trap system, but it will create problems with the GC system. A large quantity of water can quench the PID response, causing a negative dip in the baseline. Water also can saturate the MS and create interference with early eluting gases. Analysts using an MS can observe the water band eluting from the column as a large baseline rise. Since VOC samples typically are aqueous or contain large amounts of water, water vapor will be purged along with the target compounds. Although water cannot be completely prevented from collecting on the trap, the amount transferred to the GC system can be minimized by using a trap that contains hydrophobic adsorbents (see Adsorbent Materials and Traps, page 10). A dry purge step also may remove water from the adsorbent surface (see Sequences and Flow Paths, page 8). Many new purge and trap systems employ water management to minimize the amount of water reaching the GC system, but be aware that these systems can have adverse effects on the recovery of polar compounds such as ketones (page 13). Many laboratories purge 5mL-10mL of sample in 25mL purge vessels (see photo); water condenses on the inner wall of the vessel, reducing the amount of moisture that ultimately gets onto the trap.

Leaks and Active Sites: Another common problem in purge and trap systems is reduced sensitivity caused by leaks or active sites in the system. Reduced sensitivity for all compounds normally indicates a leak. To test for leaks in the purge and trap system, perform a pressure decay test by capping off the purge vent during the purge cycle. The bubbles passing through the purge vessel should stop within 2 to 10 minutes. If the bubbles do not stop, there is a leak in the purge system. To locate the leak, use a leak detector. Start checking for leaks at the purge vessel and work back to the inlet line on the back of the instrument. Leaks most commonly occur at the purge vessel and at the trap fittings.

Reduced sensitivity for specific compounds usually indicates the presence of active sites in the system. Poor response for bromoform or other brominated compounds is a good indicator of active sites in the purge and trap unit or transfer line. However, poor bromoform response also can be caused by high transfer line temperatures (>130°C). Reduce the transfer line temperature and determine if bromoform recoveries improve. Another component that decomposes due to active sites is 1,1,2,2-tetrachloroethane (Figure 11).⁶ To reduce or eliminate sources of activity, clean or replace sources of contamination, including internal gas lines and the transfer line. Inert Silcosteel[®]-treated tubing is an excellent choice for re-plumbing purge and trap gas lines. Tekmar's newest purge and trap concentrator, Model 3100, incorporates Silcosteel[®] treatment on all tubing and internal surfaces.

Ghost Peaks: Ghost peaks typically are caused by carryover from sample components that collect within the purge and trap system. This problem is most common when performing total petroleum hydrocarbon (TPH) analysis because these samples often contain high molecular weight components. If the valve oven and transfer line temperatures are set too low, high molecular weight compounds can condense in the line, then bleed onto the column. To eliminate ghost peaks, temporarily increase the purge and trap valve oven and transfer line temperatures to bake out the contaminants. The heated mount feature on some purge and trap instruments can reduce carryover by up to 50%, but this also will increase the amount of moisture entering the trap. The standard mount temperature is 40°C; increasing the temperature to 70°C significantly reduces sample carryover. For severe contamination, steam cleaning or methanol rinsing can be performed (see instructions on page 11). Unlike in other cleaning procedures, here we do not recommend using water after methanol rinsing because it is very difficult to remove water from the purge and trap system. Ghost peaks also are caused by adsorbent contamination or degradation. Tenax[®] can break down to toluene, benzene, styrene, naphthalene, and other aromatic compounds (see Adsorbent Materials and Traps, page 10). This normally is an indication of trap overheating. To prevent this problem, do not expose a trap containing Tenax[®] adsorbent to temperatures above 200°C.



Purging a 5mL sample in a 25mL purge vessel, to reduce water transfer to the trap.

6. Tekmar-Dohrmann, *Purge and Trap Concentrator Course*, 1989. Cincinnati, Ohio.

EPA Update

The US EPA promulgated update III of Test Methods for Evaluating Solid Waste (SW-846). This 1997 update deleted the previous EPA purge and trap Method 5030A, "Sample Preparation of Volatile Organic Compounds for Purge and Trap Analysis" and replaced it with Method 5035, "Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples." Method 5035 involves extensive fieldwork and raises MDLs for soil samples; however, accuracy is improved.

Previously, soil samples were collected using Teflon[®]-lined screw-cap containers and stored at 4°C, with a 14-day maximum holding time. Once the samples were in the laboratory, 5g aliquots of soil were added to 5mL of reverse osmosis (RO) water. The volatiles in these samples exit the soil matrix and leak from the container. Method 5035 requires samples to be collected and preserved in the field at the time of sampling, using methanol and a stir-bar. Volatiles dissolved in the methanol are less likely to escape. The seal is not broken until the time of analysis, thus minimizing analyte loss through evaporative mechanisms. Sodium bisulfate is used to prevent biodegradation of VOCs. Unlike HCl preservation, sodium bisulfate does not break down 2-chloroethyl-vinyl-ether. This greatly improves the accuracy of analytical results from soil samples because evaporative loss occurs almost immediately in soils that are not preserved in methanol. Disadvantages include the higher detection limits and the problems associated with purging higher percentages of methanol.



Flushing the trap attachment area with methanol. Repeat several times.

Figure 11.

No measurable response for bromoform (9), combined with a greatly diminished response for 1,1,2,2-tetrachloroethane (10), strongly indicates a contaminated transfer line.

20m, 0.18mm ID, 1.0µm Rtx[®]-502.2 column (cat.# 40914), 4ppb of VOA standards.

Oven temp.: 35°C (hold 5 min.) to 180°C @ 6°C/min. to 210°C @ 20°C/min. (hold 5 min.)

Inj. / det. temp.: 100°C / 280°C

Linear velocity: 20cm/sec. set @ 35°C

Purge & trap: Tekmar 3000

Purge: 11 min.

Trap pressure control: 6psi

Desorb preheat: 250°C

Desorb time: 2 min.

Detector: MS

Split ratio: 40:1

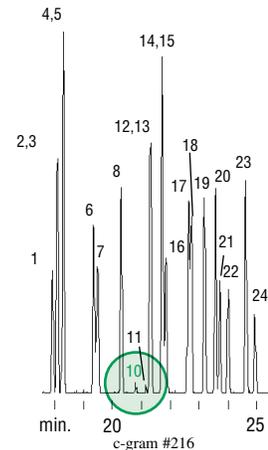
Scan range: 35-260AMU

Trap: Vocar[™] 3000

Desorb temp.: 260°C

Desorb flow rate: 20mL/min.

1. chlorobenzene	13. bromobenzene
2. ethylbenzene	14. 1,3,5-trimethylbenzene
3. 1,1,1,2-tetrachloroethane	15. 2-chlorotoluene
4. <i>m</i> -xylene	16. 4-chlorotoluene
5. <i>p</i> -xylene	17. <i>tert</i> -butylbenzene
6. <i>o</i> -xylene	18. 1,2,4-trimethylbenzene
7. styrene	19. <i>sec</i> -butylbenzene
8. isopropylbenzene	20. <i>p</i> -isopropyltoluene
9. bromoform (not detected)	21. 1,3-dichlorobenzene
10. 1,1,2,2-tetrachloroethane	22. 1,4-dichlorobenzene
11. 1,2,3-trichloropropane	23. <i>n</i> -butylbenzene
12. propylbenzene	24. 1,2-dichlorobenzene



Permission to publish this chromatogram granted by Anne Williams, Tekmar Company.

Instructions for Cleaning Purge and Trap Concentrators

We developed these instructions using Tekmar LSC 2000 and 3000 concentrators. Always remember to use safety glasses when working in the laboratory.

1. Keep the instrument power on and turn the line heaters off. Set all temperatures to the off position. WAIT UNTIL HEATED ZONES HAVE COOLED.
2. Make sure the unit is in standby mode.
3. Disconnect the purge and trap vessel.
4. Flush methanol into the area where the top of the trap attaches, using a 5mL syringe without a needle (i.e., a Luer-lock syringe - see photo). This is the area where the purge vessel attaches to the purge and trap. You should see methanol coming out of the mount.
5. Clean the mount, using a tissue. The mount is either nickel- or gold-plated, so be careful not to scratch the surface. If you cannot clean the mount, it may need to be replaced.
6. Clean the purge vessel with methanol, then with ultra-pure water. Do not use soap. You may use a brush.
7. Increase purge and trap temperatures to normal operating conditions and hold for two hours, with no trap or purge vessel installed.
8. Install an empty trap. Do not use an old trap with the packing removed; particles of trapping material may end up in the concentrator. If an empty trap is not available, refer to the next paragraph. With the empty trap in place, attach all lines, including the line to the purge vessel. Desorb for at least one hour with the transfer line disconnected from the column. This will help to drive any methanol remaining from step 6 out of the system.

If you do not have an empty trap, disconnect the transfer line from the column, connect the purge vessel and all lines, and install an old trap. Desorb for one hour.
9. Install and condition a new trap and run blanks until a clean baseline is achieved.

If you are still having activity problems after following this procedure, please contact the Restek Technical Service Team via email at support@restekcorp.com or via phone at 800-356-1688 or 814-353-1300, ext. 4.

Broad Peaks: Peak broadening is another problem often experienced when analyzing VOCs by purge and trap methods. Broad peaks are caused either by poor sample transfer from the purge and trap to the GC or by dead volume within either system. Broad peaks frequently result from dead volume in the connection between the purge and trap unit and the GC system. (See pages 18-21 for connection methods.) Because trap desorption is a relatively slow process, the sample band transferred to the GC can be very wide. To reduce this bandwidth, the sample must be transferred quickly and/or refocused at the inlet of the GC column through a secondary cold trapping technique such as cryofocusing or subambient cooling. The transfer time can be reduced by using the desorb preheat feature. During this step, the trap is heated to 5°C below the desorption temperature, and the valve is positioned so no flow passes through the trap. This helps the compounds trapped on the adsorbents to rapidly migrate from the trap when backflushing begins.

The desorb flow rate also will affect the bandwidth. If the desorb flow is too low (<9mL/min.), the band becomes broad (Figure 12) and must be refocused at the column inlet. If faster flow rates are used (>9mL/min.) in conjunction with long, thick-film columns, the bandwidth can be reduced enough so that secondary trapping is not required. Ideally, desorbing at a flow rate of 20-30mL/min. yields a very narrow bandwidth. However, when using narrow bore columns, it might be necessary to split the flow at the injection port to maintain column efficiency.

Foaming Samples: Analysts deal with foaming samples in two primary ways: by dilution or by addition of an anti-foaming agent. Diluting the sample compromises the detection limit, but in the end may save instrument downtime. Anti-foaming agents such as polydimethylsiloxane and silicon dioxide methylcellulose are designed to reduce foaming of surfactants in a liquid matrix. These are effective at preventing a sample from foaming, but they generally produce artifact peaks that can interfere with the target analytes. An anti-foam blank must be run prior to samples to determine the contribution of artifact peaks from the anti-foaming agent. If dilution or anti-foaming agents do not reduce foaming or if samples have not been screened for surfactants, use a 5 or 10mL sample in a 25mL purge vessel to prevent the bubbles from entering the fittings and, ultimately, the trap. If you are running an unattended autosampler, you can insert a plug of deactivated fused silica or glass wool into the top of the purge vessel to prevent foam from entering the purge and trap lines. If all else fails consider switching to a fritless sparge tube and increasing the purge time to effectively remove the volatile analytes.



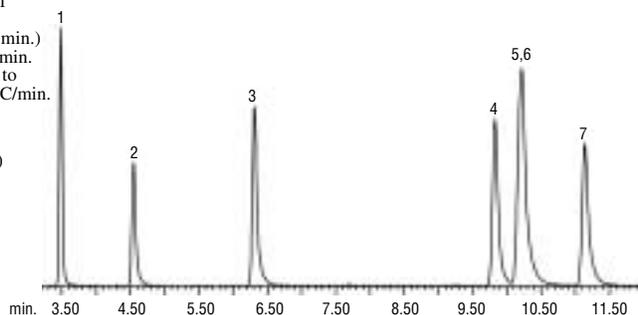
Wastewater samples commonly contain surfactants and other material that can contaminate the concentrator.

Figure 12.

A low desorb flow can produce tailing peaks, as in this example, desorbed at 9mL/min.

EPA Method 8020, Rtx®-5Si1 MS column, 40m, 0.45mm ID, 1.5µm (cat. #12798)
 Carrier gas: 9mL/min. @ constant pressure
 GC: Finnigan 9001
 Detector: FID
 Oven temp.: 40°C (hold 2 min.)
 85°C @ 4°C/min.
 (hold 1 min.) to
 225°C @ 40°C/min.
 (hold 2 min.)

1. benzene
2. α,α,α -trifluorotoluene (SS)
3. toluene
4. ethylbenzene
5. m-xylene
6. p-xylene
7. o-xylene



GC System Configurations

Wide-bore Systems (0.45mm ID and 0.53mm ID columns)

Wide-bore capillary columns are operated at faster flow rates than narrow-bore columns, and can be connected directly to the purge and trap system with a 10mL/min. desorb flow. Wide-bore columns used for VOCs analyses usually are coated with a thick film of stationary phase to increase retention and separation of the highly volatile analytes (e.g., chloromethane and vinyl chloride, bromomethane and chloroethane), or other closely-eluting sample components.

Wide-bore columns range from 30-105 meters in length. A longer column can refocus early-eluting volatile compounds and greatly improve separation of the gases (see Applications, page 37). Shorter columns require sub-ambient cooling for separating the gases; this increases the cost of the analysis and adds laboratory time associated with handling tanks of liquid nitrogen. For best overall results, we recommend using a 75m, 0.45mm ID capillary column for analyzing the volatile compounds listed in US EPA Methods 502.2 and 8021B (see Applications, page 37).

Resolution of the early-eluting gaseous analytes increases significantly with a decrease in temperature. Use a starting temperature of 35°C-50°C, depending on the target list and the purge and trap conditions (see Applications, page 37). A longer column can be used to increase the pressure within the column, which, in turn, will increase the solubility of the analytes in the stationary phase. Using optimized temperature programs and narrower bandwidths, reasonably fast analysis times can be achieved (see page 37). However, the higher flow rates through wide-bore columns prevent the analyst from directly connecting the column to the vacuum system of an MS. A jet separator or open split interface must be used to reduce the amount of carrier gas flowing into the MS (see Figure 31, page 32).

The method for connecting a purge and trap transfer line to a wide-bore GC column should be carefully considered. The three connection methods are: 1) through the existing GC injection port; 2) using a low volume injector; and 3) with a direct column connection. These alternatives are described below.

Injection Port Connection: In this connection option, the purge and trap transfer line is connected to the GC injection port that accepts the carrier gas line. The carrier gas line is cut close to the injection port body and a deactivated union (e.g., cat.# 20510, see our catalog) is used to connect the purge and trap transfer line to the injection port (Figures 13 through 16). This allows the analyst to make manual injections when troubleshooting, and to inject bromofluorobenzene when tuning the MS in accordance with EPA methods. The injection port can be a source of dead volume, however. Dead volume causes band broadening, resulting in poor peak shape and loss of resolution for the most volatile target compounds. The severity of the problem is determined by the inside diameter of the injection port liner and the total desorb flow through the port. To reduce the dead volume in the injection port, use a 1mm ID split liner (e.g., cat. #20972; see products section). If the injection port is designed for

Vu-Tight® Direct Injection Liner

- Visually observe the Press-Tight® connection between the column end and liner.
- 1/4-Inch OD: accepts 0.32 or 0.53mm ID capillary column (column OD from 0.5mm to 0.8mm).
- Slotted top prevents obstruction of carrier gas flow.
- Two designs available.*
- Operate in the direct injection mode.
- Can easily be packed with wool for dirty samples.



Description	qty.	cat.#
Vu-Tight® DI Liner	ea.	20342
Vu-Tight® DI Liner	5-pk.	20343
Vu-Tight® DI Liner	25-pk.	20344

* Refer to our catalog for information about Cyclo Vu-Tight® liners, for use with dirty samples.

Figure 13.

Plumbing a purge and trap interface to a GC injection port allows flow adjustment via the GC flow controller. This is the most common way of analyzing volatile compounds by MS because the flow can be split, allowing 1mL/min. into the MS source.

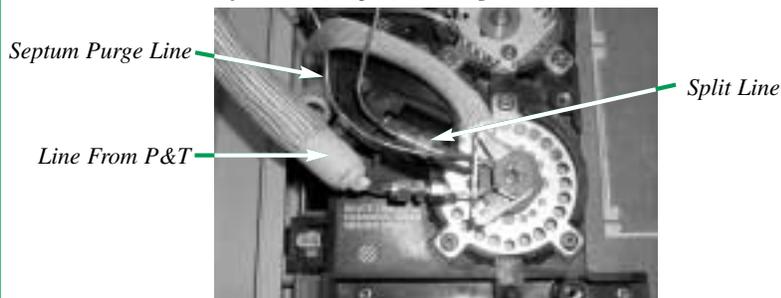
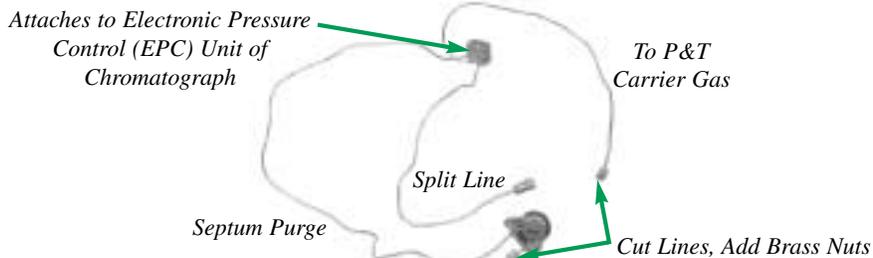


Figure 14.

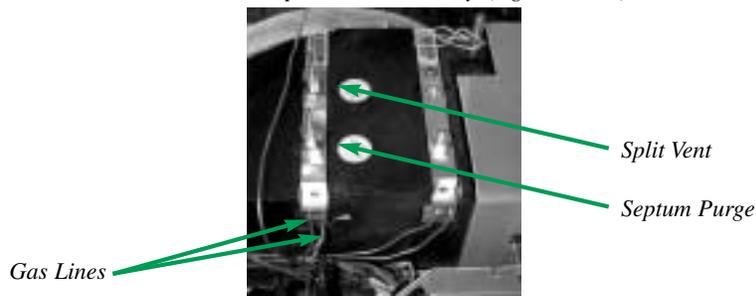
Configuring your GC is simple! Prepare the lines by cutting them as shown. (Agilent 6880)

**Figure 15.**

Carrier gas flow is adjusted through the injection port regulator. Carrier gas sweeps analytes from the trap, through the transfer line, onto the column. (Tekmar 3100)

**Figure 16.**

Reconnect the weldment lines to the GC and check for leaks. Be sure to set up the lines correctly. (Agilent 6890)



packed columns, we recommend using a Vu-Tight® inlet liner (cat. #20342, page 18). The wide-bore capillary column is sealed into the tapered restriction in the liner, ensuring direct transfer of the sample to the column. In addition, the Vu-Tight® inlet liner allows visual inspection of the column-to-liner seal.

Low-Volume Injectors: A low-volume injector (LVI) will reduce dead volume, yet allow limited manual injections. Such a system can be used to convert a packed column or a capillary split/splitless injection port for purge and trap applications. The internal volume of the injector is significantly reduced, compared to a conventional injection port, which improves sample transfer from the purge and trap system. The purge and trap transfer line is connected to the LVI, and the wide-bore column is connected at the base of the injector. A septum in the LVI allows manual injections if needed.

Low Volume Injectors



Description	cat.#
Low-Volume Injector for Agilent Split/Splitless GC Inlets	21692
Low-Volume Injector for Agilent 5890 Septum Packed Purge Port	21698
Low-Volume Injector for Varian Split/Splitless GC Inlets	21693

For descriptions of low-volume injectors, see page 63.

Direct Connection

A union between the purge and trap transfer line and the capillary column bypasses the injection port, eliminating the problems associated with the injection port: loss of sample through the septum purge, adsorption of active compounds, bleed from Viton® O-rings or septum, and — most important — dead volume. Two direct connections are described below. The disadvantage of a direct connection is it eliminates the ability to make manual injections when attempting to isolate a chromatographic problem. Therefore, this connection technique works best for experienced analysts and for instruments that undergo regular maintenance.

Metal Transfer Line: This is the easier of the two direct connection methods. Using the transfer line provided by the instrument manufacturer and an MXT® low dead volume connector (cat.# 20394, see our catalog), connect the trap to the capillary column. This configuration significantly improves peak shape, compared to injection port connections, especially with an electrolytic conductivity detector (ELCD).

Fused Silica Transfer Line: A fused silica transfer line further reduces dead volume, relative to the original equipment line. We recommend using Siltek® fused silica tubing for VOC or other sensitive analyses because it is not affected by moisture and is inert to active compounds. To configure the line, disconnect the metal transfer line from the Valco® six-port valve, then remove the metal ferrule and 1/16" nut by cutting the end of the tubing. While wearing insulated gloves, heat the line to 200°C to melt the glue that holds the line in place, then use pliers to pull the line out of the heater jacket. Cool the line, install a piece of metal tubing (cat. #21503, see our catalog) inside the line, then install the Siltek®-treated fused silica transfer line within the metal tubing (cat. #10027, page 63). The metal tubing will prevent the transfer line from being scratched or broken. Base the ID of the metal tubing on the OD of the transfer line: 0.02" ID for a 0.25 or 0.32mm ID fused silica line, 0.30" ID for a 0.45 or 0.53mm ID line. In turn, base the ID of the transfer line on the ID of the analytical column; we recommend using a transfer line with an ID equal to or slightly smaller than that of the column. A transfer line with an ID slightly smaller than that of the column will increase backpressure, enhancing the resolution of early-eluting compounds. Use a Press-Tight® connector (cat. #20400 or 20403, page 20) to connect the fused silica transfer line to the analytical column (Figure 17). Use the correct ferrule for connecting the column to the 6-port valve (Figure 18); we recommend a one-piece fused silica adaptor (cat. #20137, page 64).

Universal Press-Tight® Connectors



Description	cat.#
Universal Press-Tight® Connectors, 5-pk.	20400
Universal Press-Tight® Connectors, 25-pk.	20401
Universal Press-Tight® Connectors, 100-pk.	20402
Universal Angled "Y" Press-Tight® Connector, ea.	20403
Universal Angled "Y" Press-Tight® Connectors, 3-pk.	20404

For additional connectors, see page 64.

Figure 17.

A dual-column configuration splits the sample equally between separate detector systems.

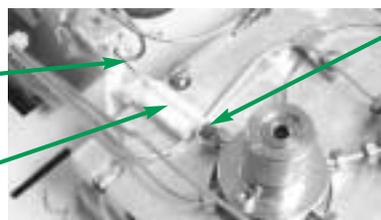


Figure 18.

Connect the fused silica line directly to the 6-port valve. Notice a small (5mm) septum helps determine how far the column is inserted into the valve, preventing breakage at the column end that could allow shards of fused silica to enter the valve.

MXT® Metal Tubing
(used to protect fused
silica column)

Fused Silica



Septum

Narrow-bore Systems (0.18mm ID - 0.32mm ID columns)

Narrow-bore columns (0.18mm ID-0.32mm ID) offer higher resolution, compared to 0.45mm ID or 0.53mm ID columns. Because these columns typically are operated at lower flow rates, they are not compatible with the fast desorb flow rates from common purge and trap systems. Splitting the sample at the injection port or cryofocusing (i.e., secondary trapping) will provide compatibility and help focus the sample at the column inlet.

Splitting the Sample: Many environmental laboratories analyzing VOCs by GC/MS use narrow-bore capillary columns and split the sample at the injection port. Higher sensitivity ion trap GC/MS systems (e.g., Varian Saturn 2000™ and Thermo Finnigan GCQplus™ systems)⁷ and recently developed quadrupole MS systems (e.g., the Agilent 5973 system) allow high split ratios in the injection port while maintaining sensitivity adequate to meet the requirements of EPA Method 524.2.⁸ Older quadrupole GC/MS systems require an increase in purge volume (25mL) to compensate for the sample lost due to splitting.

Using a standard split/splitless injection port to split the desorb flow allows a higher desorb flow rate while maintaining a lower column flow. With this technique, the trap is desorbed at a flow rate of 10-60mL/min. and the column flow rate is adjusted to 1.0-1.3mL/min., which is compatible with the vacuum system of an MS. The remaining flow exits through the split vent. The faster desorb flow rate produces a narrow sample bandwidth which, when combined with the high efficiency of a narrow-bore column, allows high split ratios without significant loss in sensitivity.⁸ Surprisingly, a 1:20 split ratio provides more sensitivity than a 1:10 split ratio, because the higher flow from the trap focuses the target compounds more efficiently.

Cryofocusing (secondary trapping): A cryofocusing unit refocuses the volatile compounds at the inlet of the narrow-bore column. This allows the trap to be desorbed at only 1-2mL/min., while improving peak shape and resolution by reducing sample bandwidth. Cryofocusing takes place on a short length of deactivated, uncoated, fused silica tubing that is cooled to -160°C using liquid nitrogen. To increase retention for very volatile gases, or when analyzing Freon® compounds, use tubing coated with a thick film of stationary phase.

While cryofocusing greatly improves peak shapes from narrow-bore columns, the approach consumes large amounts of liquid nitrogen, increasing operating expenses and requiring liquid nitrogen tanks in the lab. If the liquid nitrogen tank empties in the middle of a sample sequence, there can be significant downtime before the tank is replaced.

Capillary Column Phases

Many capillary columns have been designed for the analysis of VOCs. Column selection normally is based on the analytical method (e.g., US EPA method), compound list, and detection system used. This section serves as an overview of the different phases used for VOC analyses. See the Applications section (page 37) for examples of GC and GC/MS separations under specific conditions.

The first columns used for analyzing volatiles were based on diphenyl/dimethyl polysiloxane stationary phases. These include VOCOL®, Rtx®-Volatiles, HP®-VOC, and Rtx®-502.2 columns. The main advantages of these phases are their resistance to oxidative breakdown and their lower bleed, compared to cyanopropylphenyl polysiloxane (i.e., "624") phases. The major drawback of diphenyl/dimethyl polysiloxane phases is the incomplete resolution of bromomethane and chloroethane. Many environmental laboratories still use these columns, especially when analyzing samples for a limited set of compounds.

7. Jessie Crockett Butler, Meredith Conoley, "Analysis of Volatile Organics in Solid Wastes, Soils, and Water Using a Split Injection and the Polaris Q Ion Trap GC/MS." Application Note AN9167. Thermo Finnigan, GC and GC/MS Division, Austin, TX.

8. D.R. Decker, J.J. Harland, and M.J. Feeney, "Comparison of Detection Limits and Analysis Time Using Wide and Narrow Bore Capillary Columns for Purge-and-Trap GC/MS Analyses." *OI Analytical*. Application Note 02850896.

Another type of column used for VOC analysis is based on cyanopropylphenyl/dimethyl propyl polysiloxane phases, commonly known as the “624” phases. The Rtx[®]-624 column is designed for EPA Method 624, but also performs well for Methods 524.2, Revision IV, and 8260. The main advantage of the Rtx[®]-624 column is the complete separation of the highly volatile gases, including dichlorodifluoromethane, chloromethane, vinyl chloride, chloroethane, and bromomethane.

More recently, the Rtx[®]-VRX column was developed, using computer-assisted stationary phase design (CASPD), to address the expanded list of compounds in EPA Methods 8021 and 502.2. This unique column improves resolution and reduces overall analysis time compared to traditional columns. Like the Rtx[®]-624 column, the Rtx[®]-VRX column provides excellent separation of the highly volatile gases. Its only disadvantage is poor resolution of the most common trihalomethanes (THMs), chloroform and bromodichloromethane, from other target analytes. These analytes are frequently found in chlorinated drinking water samples. While the Rtx[®]-VRX column has been used for MS methods with favorable results, because of this poor resolution it is not recommended for drinking water analysis using PID/ELCD detection.

The most recent innovations for VOC analysis have been the development of the Rtx[®]-VGC and Rtx[®]-VMS columns. These columns also were designed using CASPD. Designed for PID/ELCD analyses, the Rtx[®]-VGC column resolves all compounds listed in EPA Methods 502.2 and 8021, with >80% resolution of each of the four trihalomethanes from the other target compounds, >30% resolution between 2-chlorotoluene/1,1,2,2-tetrachloroethane, and >60% resolution of all other volatile compounds in the two EPA methods. The column resolves the gases and early-eluting compounds well enough that the GC oven program can be started at 50°C.

The Rtx[®]-VMS column was designed to address the increasing number of analytes listed in EPA Method 8260, and also is a good choice for separating compounds listed in EPA Method 524.2, revision IV. The major difference between the Rtx[®]-VMS phase and others such as “502.2,” “624,” or “VRX” is its overall selectivity and the distance between members of isomeric pairs, like 2-/4-chlorotoluene. A faster final oven ramp rate is possible because these compounds elute farther apart on the Rtx[®]-VMS phase, eliminating partial co-elutions that would interfere with quantification. This column offers excellent separation of EPA Method 8260B compounds in less than 18 minutes - the normal cycle time for a purge and trap system. Using the EPA-suggested surrogates the analysis time can be less than 10 minutes with a narrow bore column. Even faster analyses are possible if you replace the internal standard chlorobenzene-d5 with another compound, such as 4-bromofluorobenzene. Sub-10-minute analysis times allow you to connect two purge & trap units to one GC/MS instrument, significantly increasing sample throughput (see page 37).

Metal Columns

In addition to the standard fused silica versions of the analytical columns discussed above, metal MXT[®] columns coated with the same stationary phases also are available from Restek. To eliminate the activity problems associated with metal tubing, we make these columns from Silcosteel[®]-treated stainless steel tubing, assuring excellent inertness. Because these columns are much more durable than fused silica columns, and can be coiled to less than 5-inch diameters, they are ideal for portable GC applications. Their durability makes them a popular choice for teaching laboratories at colleges and universities. Analyte resolution on MXT[®] columns is similar to that on fused silica columns.

Detection Systems

VOCs can be analyzed using a variety of detection systems, including GC/MS, GC/PID, and GC/ELCD. Here, we discuss consequences of using each of these systems, and present tips for maintenance and troubleshooting.

Column Configurations

Single Column: Environmental engineers characterize a contaminated site using MS or dual-column GC, or they might monitor the site solely with single-column GC methods. Injections of standards on a single column, delivered to the detector, can provide tentative identification and quantification. Retention times for analytes listed in a given method are established by injecting a check standard containing all of the target compounds. Retention times for analytes in site samples are compared against retention times for the standard, to verify if unknown compounds match known targets. A single column configuration works well with characterized samples, but retention times are not unique for every analyte, especially for early-eluting compounds that spend little time in the stationary phase (e.g., Freon® compounds). In environmental laboratories coelutions from non-target compounds also are very common, creating very complex chromatograms that are difficult to interpret using a single-column design.

Dual Columns: In a dual-column configuration, the sample passes through a fused silica guard column, then is split between two analytical columns of differing selectivity. Standards are injected to establish retention times on both columns simultaneously. One disadvantage to this configuration is the 50% loss in sensitivity resulting from splitting the sample. This loss can be overcome by increasing the sample volume or by optimizing the detector. Flow rates for the two columns should agree within 20% because uneven splitting will further affect sensitivity.

Detector Configurations

Detectors can be connected in parallel, in series, or in tandem, to double the amount of information about the sample.

Parallel System: In a parallel system the sample is split equally between the two detectors, allowing both detectors to be destructive (e.g., ELCD/FID). This detection system works well but is unsuitable for a dual-column analytical configuration because the sample already will have been split between the two columns.

Series System: Series detection involves connecting two detectors in sequence, using a short length of deactivated metal or fused silica tubing. The sample passes through the first detector, which must be non-destructive (e.g., PID), then through the second detector. This produces two sets of information about the sample with no loss in sensitivity because the sample volume does not change from the first detector to the second. The only disadvantage is dead volume, which can broaden the peaks. Minimize dead volume by minimizing the length and ID of the line connecting the detectors.

Tandem System: The tandem configuration connects two detectors without the dead volume associated with a series system. The non-destructive first detector is the base for the second detector. The units can be connected to a single detector port on one GC. This makes it possible to use a dual-column configuration, with each column connecting to tandem detectors, producing four sets of data per analysis. This approach is used in EPA Method 8021.⁹

Detectors: Method requirements determine the choice of detector(s). The current shift toward analysis by performance-based criteria makes it possible to use detection other than that listed in a method if it can be shown that performance is similar to, or better than, what would be attained by following the guidelines in the method. The most common GC detectors are the PID, the FID, and the ELCD. GC/MS eliminates the need for a confirmation column.

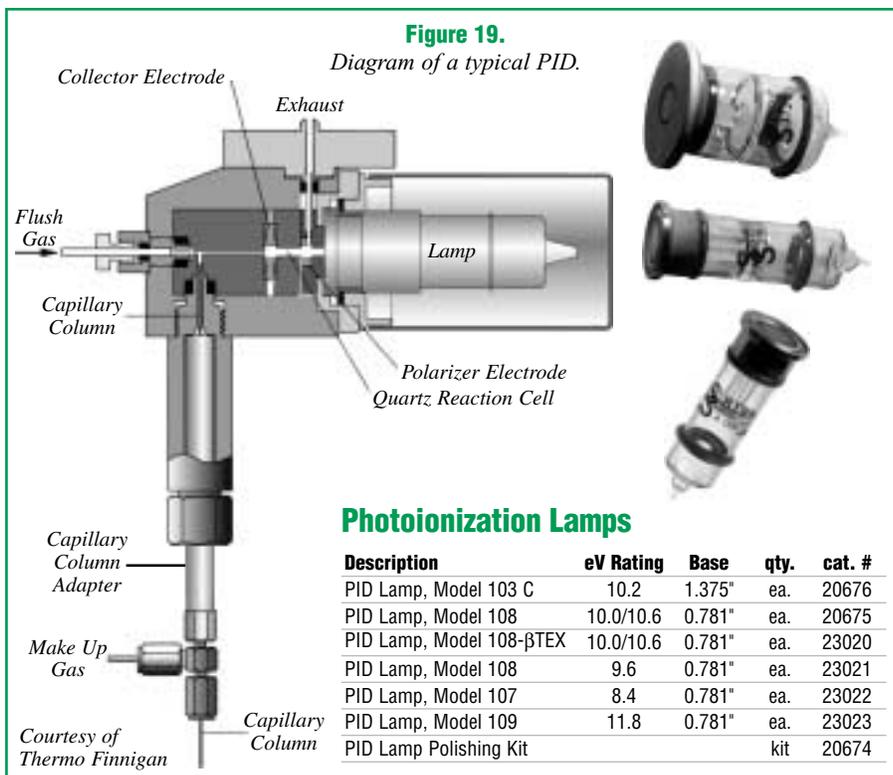
9. R.D. Braun. *Introduction to Instrumental Analysis*. McGraw-Hill Book Company. New York. 1987. pp. 915-916.

PID Operation

The photoionization detector, PID, is a selective, non-destructive detector most commonly used for characterizing aromatic compounds (Figure 19). It has excellent sensitivity (low pg detection) and provides a linear dynamic range of 3 orders of magnitude.

In a PID, a krypton lamp emits photons in the form of light energy at a wavelength of 116.6nm and 123.6nm. The photons excite compounds having an ionization potential of less than 10.2eV. Charged particles produced in this manner pass through a reaction cell with an electrical potential of 50 to 200 volts, producing an electrical charge that is measured as a signal. Sensitivity is a function of the chemical structure of the analyte, including the number of carbon atoms, the nature and position of functional groups, and the position of double or conjugated double bonds.¹⁰ For suitable analytes, a PID is 10-times more sensitive than an FID. Compounds such as benzene (9.3eV ionization potential) have ionization efficiencies of less than 0.1%, allowing the majority of the sample to pass through the detector unaffected. Even with this minute portion of the sample ionized, sensitivity for aromatic compounds is measured at the pg level.

PID Maintenance and Troubleshooting: It is very common for silicone from column bleed to collect on the PID window and reduce transmission from the lamp. Reduced sensitivity for all components is evidence of this condition. We recommend cleaning the window on a regular basis, using a mild abrasive material such as iron oxide. Alumina powder is more abrasive than iron oxide and can scratch the lens. This will reduce sensitivity. Create a slurry with the powder, scrub the window, then rinse with acetone or methanol. Avoid touching the clean window. If sensitivity is not restored, replace the lamp. Dead volume within a PID will produce broad peaks, and might cause peaks to tail. PIDs designed for packed column systems have a cell volume intended for high flow rates. When using these detectors with capillary columns, 20-30mL/min. of make-up gas is required to minimize dead volume and, in turn, reduce peak tailing. Newer PID designs have smaller cell volumes that are compatible with capillary columns.



10. Jessie Crockett Butler, "Tandem Detector Technology for Gas Chromatography." *Environmental Analysis Technical Report No. 9110, p3. Thermo Finnigan, GC and GC/MS Division, Austin, TX.*

FID Operation

The flame ionization detector, FID (Figure 20), is a selective detector because it only responds to materials that ionize in an air/hydrogen flame. This condition covers a very broad range of compounds, however. An FID / PID combination often is used for petroleum or volatile analyte applications.

In an FID, the combustion of hydrogen and air produces a flame. When an organic compound enters the flame; the large increase in ions produced is measured as a positive signal. Because response is proportional to the number of carbon atoms introduced into the flame, an FID is considered a quantitative counter of carbon atoms burned.¹⁰ Among the detectors discussed here, an FID has the largest linear dynamic range – nearly five orders of magnitude. The FID will detect most carbon-containing analytes at a sensitivity of approximately 0.5ng/ μ L.

Properly set gas flow rates are important to achieving maximum sensitivity with an FID, and preventing the flame from being extinguished (flame-outs). Generally, the total flow to the FID is 300mL/min. to 500mL/min., of which the hydrogen flow plus the carrier gas flow is approximately 30mL/min. The balance of the flow (make-up gas) typically is nitrogen.

The carrier gas and hydrogen gas mix in the FID jet. Capillary columns require a small jet (0.011 inch ID) whereas most packed column applications are compatible with a larger jet (0.018 inch ID). Jets with even larger ID are available for applications involving packed columns that exhibit higher bleed.

Capillary Adaptable FID Jet for Agilent 5890/6890/6850 GCs (0.011-inch ID tip)

(Similar to Agilent part # 19244-80560.)

Description	qty.	cat.#	qty.	cat.#
Standard	ea.	20670	3-pk.	20671
High-Performance (Silcosteel [®] -Passivated)	ea.	20672	3-pk.	20673

Capillary Dedicated FID Jet for Agilent 6890/6850 GCs

(Similar to Agilent part # G1531-80560.)

Description	qty.	cat.#	qty.	cat.#
Standard	ea.	21621	3-pk.	21682
High-Performance (Silcosteel [®] -Passivated)	ea.	21620	3-pk.	21683

Capillary FID Jet for Agilent 5880 GCs

(Similar to Agilent part # 19301-80500.)

Description	qty.	cat.#
Standard	ea.	21637
High-Performance (Silcosteel [®] -Passivated)	ea.	21638

Packed Column FID Jets for Agilent 5890/6890/6850 GCs

0.018-Inch ID

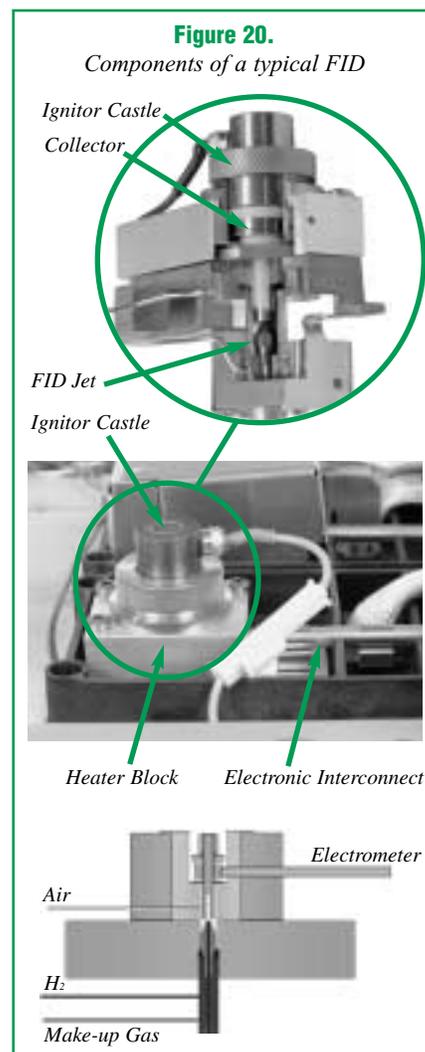
(Similar to Agilent part # 18710-20119.)

Description	qty.	cat.#	qty.	cat.#
Standard	ea.	21694	3-pk.	21695
High-Performance (Silcosteel [®] -Passivated)	ea.	21696	3-pk.	21697

0.030-Inch ID

(Similar to Agilent part # 18789-80070.)

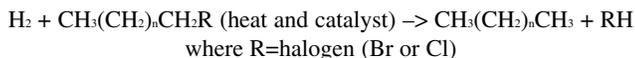
Description	qty.	cat.#	qty.	cat.#
Standard	ea.	21688	3-pk.	21689
High-Performance (Silcosteel [®] -Passivated)	ea.	21686	3-pk.	21687



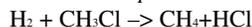
FID Maintenance and Troubleshooting: Contamination and a clogged jet are common problems associated with using an FID in analyses of volatile compounds, such as gasoline range organics (GRO) analyses that involve samples containing diesel fuel or oils. Flame-outs at the beginning of a VOCs analysis usually are the combined result of incorrect gas flows and excessive water from the purge trap. When performing maintenance on an FID always check the gas flows before calibrating the instrument. Water management is discussed on page 13.

ELCD (Hall® detector) Operation

In typical applications, an electrolytic conductivity detector, ELCD (Figure 21), is a chemical detector that catalytically reduces halogenated materials to haloacids, HCl and HBr, by mixing them with high-temperature hydrogen in a heated nickel reaction tube. In other words, this detector pyrolyzes these analytes in the presence of a catalyst and a reaction gas (hydrogen):



Example:



The haloacid molecules flow into the electrolytic conductivity cell via a Teflon® transfer line, and are dissolved in a stream of n-propanol. The conductivity of the alcohol is monitored because the concentration of hydrogen halide is directly proportional to the current. The signals thus produced characteristically have tailing peaks. Although the ELCD is most commonly used for halogenated compounds and, in the halogen mode, it is selective only for these species (Figure 21), it can be configured to detect sulfur, nitrogen, and nitrosamine compounds. Figures 22-25 and Figure 27 show various important parts of the ELCD system.

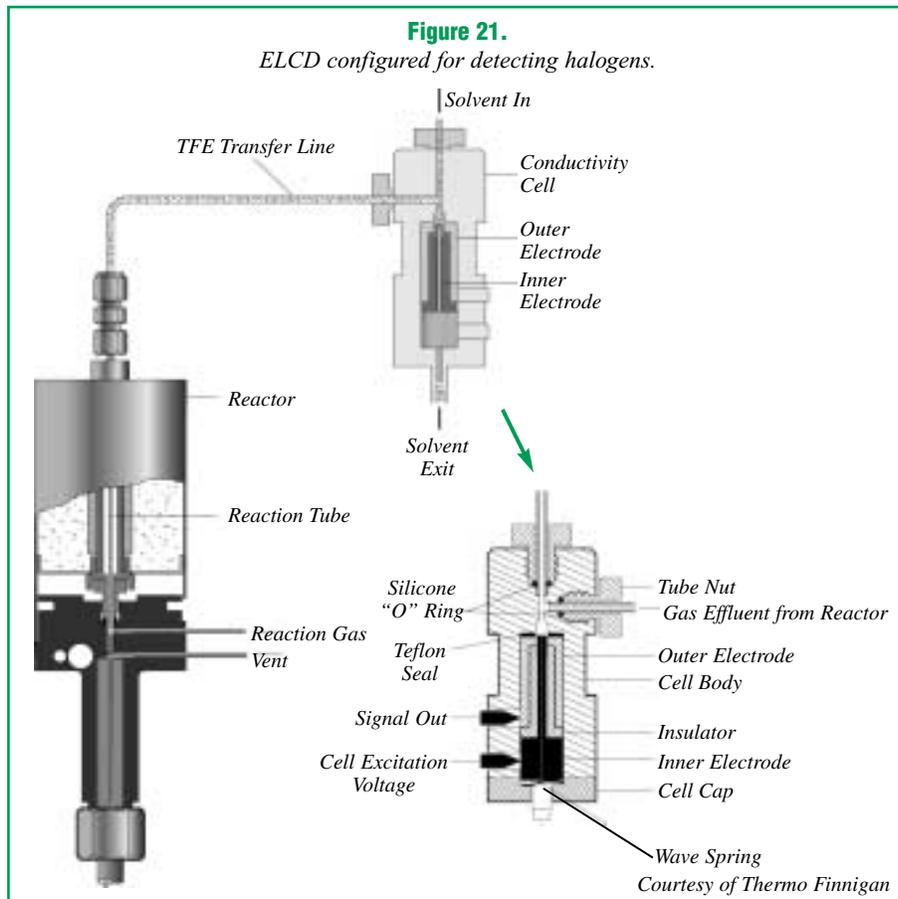
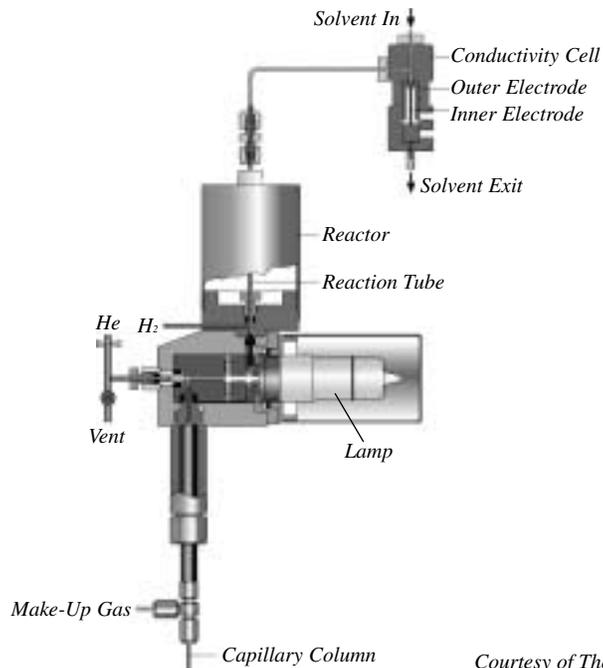


Figure 22.

Cross-section of the Thermo Finnigan PID/ELCD



Courtesy of Thermo Finnigan

Teflon® Transfer Lines for ELCDs

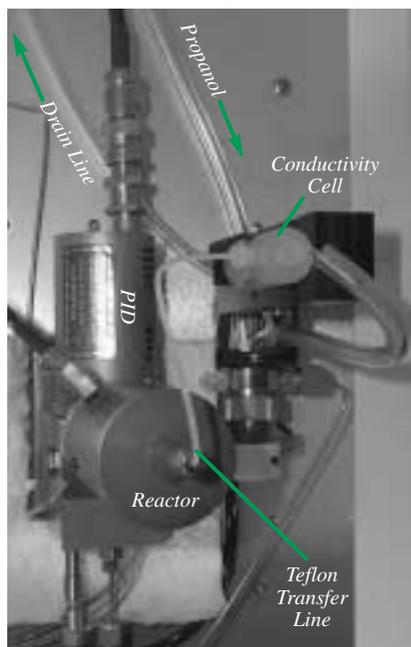


- Stringently cleaned with HCl.
- Convenient precut pieces.
- Fit Tracor, Tremetics, O.I., many other ELCDs.

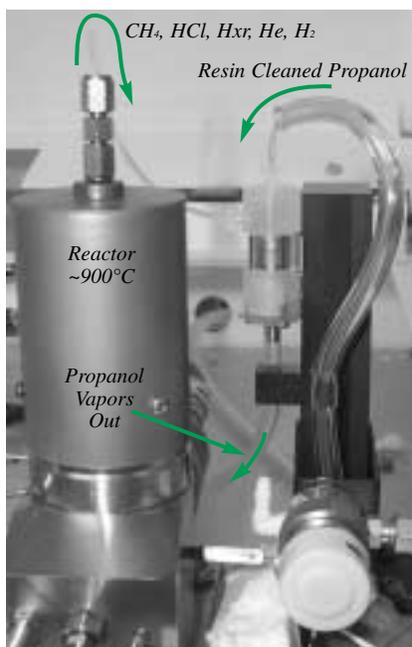
Description	cat.#
Teflon® Transfer Lines for ELCDs (five 6.5-inch lines), 5-pk.	20121

Figure 23.

Electrolytic conductivity detector: top view

**Figure 24.**

Side view of the tandem detectors.



Chromatographic Detectors: Design, Function, and Operation

Comprehensively covers the design, construction, and operation of gas chromatography, liquid chromatography and thin-layer chromatography detectors—all in one convenient, up-to-date source.

*R.P.W. Scott, Marcell Dekker, Inc.,
1996, 514pp., ISBN 0-8247-9779-5
cat.# 21090*



Ultra-High-Purity Brass Line Regulator

- Use wherever you need to reduce the line pressure by 20psi or more.
- Same purity level as high-pressure cylinder regulators.



Fitting	qty.	cat.#
1/4" female NPT ports*	ea.	21666

* Please order appropriate male connector, pipe-to-tube fittings; see our catalog.

Figure 25.

Electrolytic conductivity detector: propanol tank with pump.

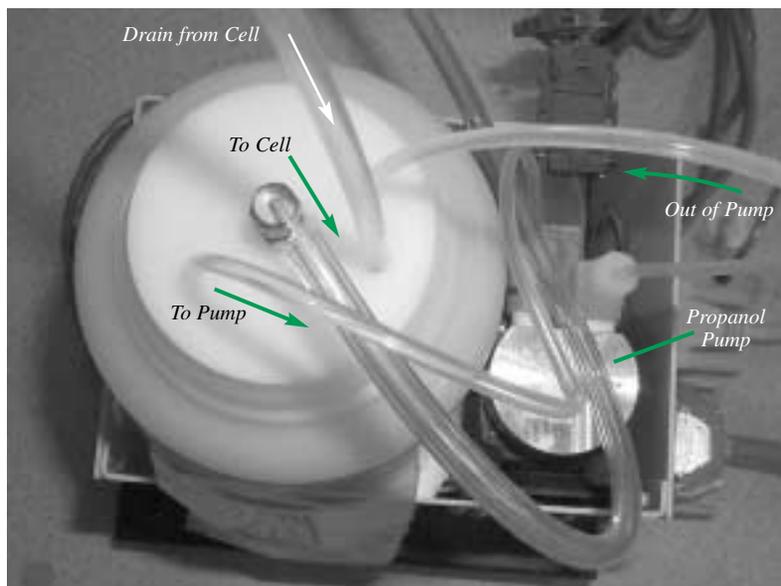


Figure 26.

With any detector, always be sure that gas flows are adjusted properly.



ELCD Maintenance and Troubleshooting: ELCD performance depends on the reactor temperature, the volume of the conductivity cell, the propanol flow rate, the hydrogen gas flow (Figure 26), and the purity of the solvent system. The goal is to minimize tailing as much as possible without losing sensitivity. Depending on the amount of use and the types of samples analyzed, the nickel reaction tube – the reaction catalyst – should be replaced as often as monthly. Hydrocarbons and certain other compounds can “poison” the reaction tube and reduce its catalytic activity. Oxygen and moisture can oxidize the reaction tube, affecting sensitivity. High-purity hydrogen gas is critical for a stable baseline. Use gas regulators with stainless steel diaphragms and the proper purifiers for reaction gases.

A drop in sensitivity (particularly for brominated compounds), baseline instability, or appearance of unknown peaks indicate it might be necessary to replace the reaction tube. Reconditioning the reaction tube might restore baseline stability: disconnect the Teflon® transfer line, then increase the reactor temperature to 1000°C for one hour, then reset the reaction temperature to 900°C for re-calibration.

Similarly, the Teflon® transfer line between the reactor and the conductivity cell requires frequent cleaning or replacement. Flushing the transfer line should remove most of the contamination. To do this, disconnect the line from the reaction tube and plug the drain line leading from the reaction cell. This will force propanol through the transfer line, flushing contamination out. If this does not improve response and peak shape, replace the transfer line (cat# 20121, page 27).

Use only high-purity solvents in the ELCD (only HPLC-grade for halogen mode). The solvent intake line is equipped with a scrubber resin cartridge that removes contaminants from the solvent. To maintain solvent purity and a stable baseline, change this cartridge every six months.

ELCD: Minimizing Peak Tailing: Peak tailing is a characteristic of the ELCD – the key to successful ELCD operation is regular maintenance to minimize the tailing. Most tailing problems are caused by contamination or leaks in the system. Peak tailing also can be caused by contamination in the Teflon® transfer line from the reaction tube to the conductivity cell. Table III lists factors that can contribute to tailing peaks. Reaction tube deterioration can be due to water and/or oxygen corroding the tube surface over time, or to carbon deposits left by the organic solvent. In purge and trap applications, water management can help slow this corrosion.

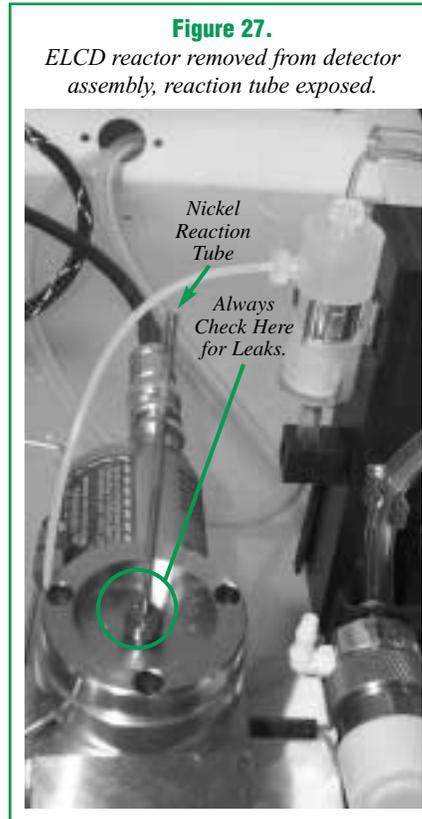
Poor responses for brominated compounds indicate active sites in the pathway. Isolate the purge and trap system by making a manual injection. If responses for brominated compounds still are poor, the reaction tube probably is deteriorating. A combination of tailing peaks and poor responses for brominated compounds also is an indication that the reaction tube must be replaced. Maintain detailed notes on instrument maintenance to minimize troubleshooting problems in the future.

ELCD performance also depends on the internal volume of the conductivity cell. Older ELCDs have larger cell volumes that cause more tailing. Smaller cells in newer ELCDs significantly reduce peak tailing.

Table III.

Common causes of excessive peak tailing from an ELCD.

Contaminated conductivity cell	Low makeup gas flow
Contaminated reaction tube	Low propanol flow
Contaminated resin cartridge	Low reaction gas flow (hydrogen)
Contaminated Teflon® transfer line	Low reaction temperature (below 850°C)
Dead volume between detectors in series	Unpure gas (carrier / makeup / reaction)
Leak at the base of the reaction tube	



Replacement Nickel Reaction Tubes

- Pretreated for maximum sensitivity.
- Quality-controlled for reliability.
- Available for different models.



To replace these instrument part numbers:

Order these Restek part numbers:

ELCD Model #	Tremetrics	Varian	PerkinElmer	Shimadzu	O.I. Analytical	qty.	cat.#
Hall 700A	115439-0003	00-996724-14	0330-2675	—	—	2-pk.	21580
Hall 1000	117459-0003	00-997625-12	N660-1072	220-90435-00	—	2-pk.	21581
O.I. 4420	—	—	—	—	183780	2-pk.	21582

GC/MS Operation

Mass spectrometry (MS) is the most common detection system used for VOC analysis. The MS provides unique spectral information for accurately identifying components eluting from the capillary column. As a compound exits the column it is bombarded with high-energy electrons and is broken into structurally significant charged fragments. These fragments are separated by their mass-to-charge ratios in the analyzer, to produce a spectral pattern (i.e., fingerprint) unique to the compound. To confirm the identity of the compound the spectral fingerprint is matched to a library of known spectra. By knowing the spectral patterns for compounds in the target list, the appropriate masses for quantification can be chosen.

For analyzing volatile compounds in environmental samples, the most common types of MS operating systems are the quadrupole system and the ion trap system.

Quadrupole Operation

A narrow bore (≤ 0.25 mm ID) capillary column can be inserted into the source of the quadrupole MS in electron impact mode (EI). The carrier gas flowing through the column, approximately 1mL/min., is quickly swept away under the high vacuum of the source while analytes exiting the column are bombarded with a stream of electrons at 70eV.

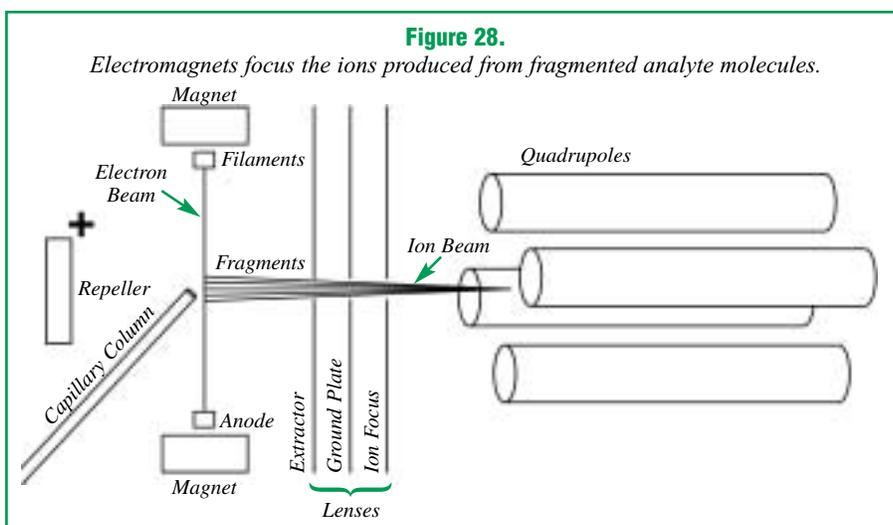
Electromagnets begin focusing the ions (Figure 28). Positively charged fragments are pushed away from the positively charged repeller, toward a series of focusing lenses. The first lens, the draw-out plate or extractor, accelerates the ions, then the ion focus lens further accelerates the ions and squeezes them into a tight beam of charged particles before they enter the mass analyzer – an array of four parallel rods, or quadrupoles. Other plates (i.e., a ground plate), if present, are connected to a ground that discharges the defocused ions, to prevent them from causing charge interference with the ion focus lens. In this way ions that are not correctly directed down the quadrupoles are discarded. Electromagnetic fields produced by a combination of direct current (DC) and an oscillating radio frequency (RF), enables ions that have a specific mass-to-charge ratio to pass through the quadrupoles to the detector, forcing these ions into a spiral, or corkscrew-shaped, three-dimensional sine wave that passes through the center of the quadrupole arrangement (Figure 29). As the DC/RF waves are swept up or down, specific mass-to-charge ions strike the electron multiplier (detector), which translates ions to electrons. The electrons bounce off the dynodes (walls) of the electron multiplier, generating a cascade of electrons. These electrons are exchanged to photons, which are measured as a current by the photomultiplier.¹¹

Gas Chromatography & Mass Spectrometry, A Practical Guide

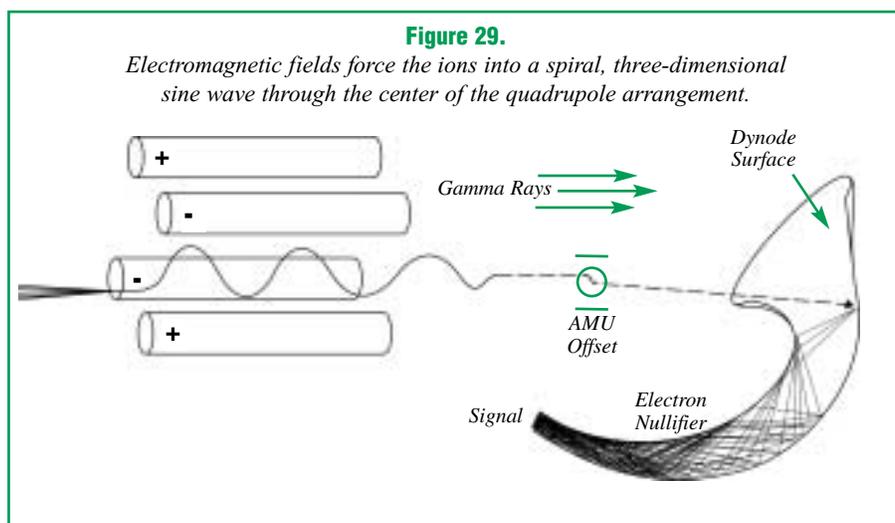
- Separation conditions for numerous compound types, derivatized and underivatized.
- How to interpret mass-spectral data, with examples.



F.G. Kitson, B.S. Larsen and C.N. McEwen, Academic Press, 1996, 381pp., ISBN 0-12-483385-3 cat.# 20497



11. F.G. Kitson, B.S. Larsen & C.N. McEwen. *Gas Chromatography and Mass Spectrometry: A Practical Guide*, Academic Press, New York. 1996.



Ion Trap Operation

The major difference between a quadrupole MS and an ion trap MS are the mechanisms of ion focus and scanning. Three hyperbolic electrodes, a ring and two endcaps, form the core of an ion trap MS (Figure 30). In electron impact mode the sample is ionized, fragmented, and introduced into the ion trap through a pulsing electronic gate that opens and closes, controlling the number of ions that enter the trap. Ions that enter the trap are stored in stable orbits. Adjusting the voltage around the ring electrode pushes some of these ions into unstable orbits, causing them to exit to the detector. Because all ions entering the trap are stored temporarily, only a finite amount of sample can be allowed to enter the trap area, otherwise the system would be overloaded.

Interfacing the Capillary Column to the MS

The ion source and analyzer of the MS are under vacuum. To enable the pumping system to maintain this vacuum, the volume of carrier gas entering the MS must be small. Regardless of the pumping capacity of the MS vacuum system, the best sensitivity is achieved if the carrier gas flow rate is approximately 1 mL/min. Because a narrow-bore capillary column routinely is operated at near 1 mL/min. flow rates, it can be connected directly to the MS without overwhelming the pumping system. Wide-bore capillary columns, however, usually are operated at flow rates that are too high for most MS systems. Consequently, an interface must be used to reduce the flow to a level that is compatible with the MS pumping system. Figure 31 shows the two most common interfaces – the open split and the jet separator.

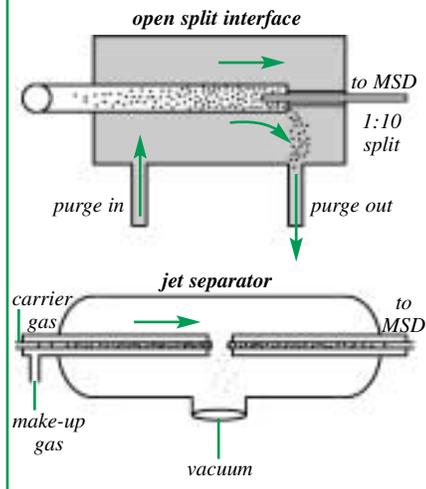
An open split interface (OSI) functions like an inlet splitter system in a chromatograph. It allows as much as 90% of the carrier gas to be vented away from the MS vacuum system. Correspondingly, this is reflected by a sample loss of up to 90%, which reduces sensitivity by an order of magnitude. Therefore, an OSI is not suitable for trace-level environmental analysis. Splitting the sample at the injection port, combined with analysis on a narrow-bore column, is favored over using an OSI because a high desorb flow rate can be used to ensure better sample transfer from the trap. Also, a 0.25 mm ID or narrower column increases efficiency and improves resolution of analytes.

Another alternative to an OSI, the jet separator, reduces the carrier gas flow without significant loss of sensitivity. A jet separator works on the principle of momentum. Very small molecules such as helium (or other carrier gas) do not have sufficient momentum to pass across a small gap in the jet separator and are routed away from the MS, using a vacuum pump. Larger molecules, such as most target components, have the necessary momentum to carry them across the gap and into the MS. Using this device, much of the carrier gas can be eliminated without significant loss of target compounds. Added momentum is required to carry very small analyte molecules, such as gases, across the gap, however. In these situations we recommend adding make-up gas to provide the extra momentum and improve responses for low molecular weight target compounds.



Figure 31.

An open split interface or a jet separator will reduce the gas flow entering a mass spectrometer vacuum system.



MS Calibration and Tuning

Calibration allows the correct identification of masses, whereas tuning adjusts the intensity and peak widths for masses. The MS is calibrated by adjusting the DC/RF frequency so that mass axis points are aligned with expected mass fragments of known spectra. Tuning ensures that target compounds analyzed on the MS will have the same distribution (pattern) of ions, and peak widths for ions will be narrow enough that adjacent mass peaks will not overlap. A compound widely used for calibrating and tuning MS systems is perfluorotributylamine (PFTBA or FC43). Modern instruments introduce PFTBA into the ion source during the autotune procedure. The instrument software adjusts the MS parameters to match the known fragmentation pattern for PFTBA. The ion of greatest abundance in the spectrum is mass 69; the relative abundances of masses 131 and 219 are roughly 50% of that for mass 69 (Figure 32). In analyses of volatiles, mass 502 is less important because its relative abundance is 1% of the mass 69 value. Low peak heights or a loss of mass 502 generally indicate a cleanliness problem at the source.

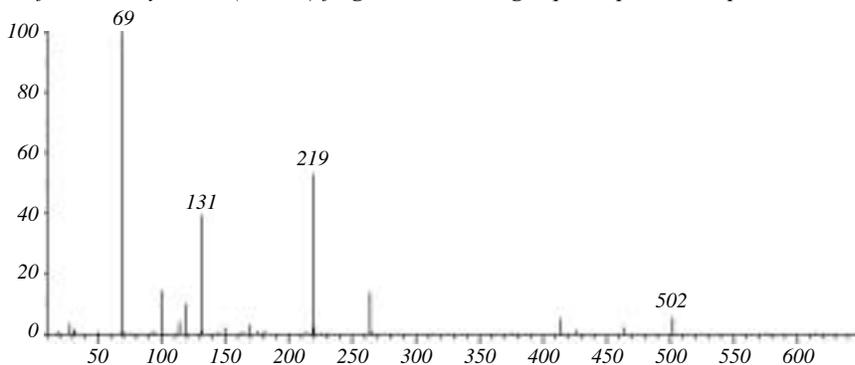
Specific Tuning Requirements: 4-Bromofluorobenzene: After the system is calibrated and tuned, using PFTBA, a 50ng solution of 4-bromofluorobenzene (BFB) is introduced. BFB usually is introduced by injection through the GC injection port but, alternatively, it can be purged from a water blank. Abundance criteria for BFB are listed in Table IV.

If tuning with BFB fails under criteria acceptable for PFTBA, decrease the relative abundance of masses 131 and 219 to 30% of mass 69 by adjusting the entrance lens. If necessary, slightly decrease the repeller voltage. This procedure targets the ions from mass 173 through mass 177. A second tuning failure with BFB may dictate recalibration and tuning with PFTBA. Ion ratios for BFB should be checked every 12 hours. As long as results meet the specifications in Table IV, no further calibration or tuning is required.

Poor tuning can significantly affect the sensitivity of the MS. Figure 33 shows spectra for a sample analyzed twice, first after a failing PFTBA tuning with mass 131 as the base peak (Figure 33, A), then after a passing tuning (Figure 33, B). The second analysis exhibits a three-fold increase in sensitivity.

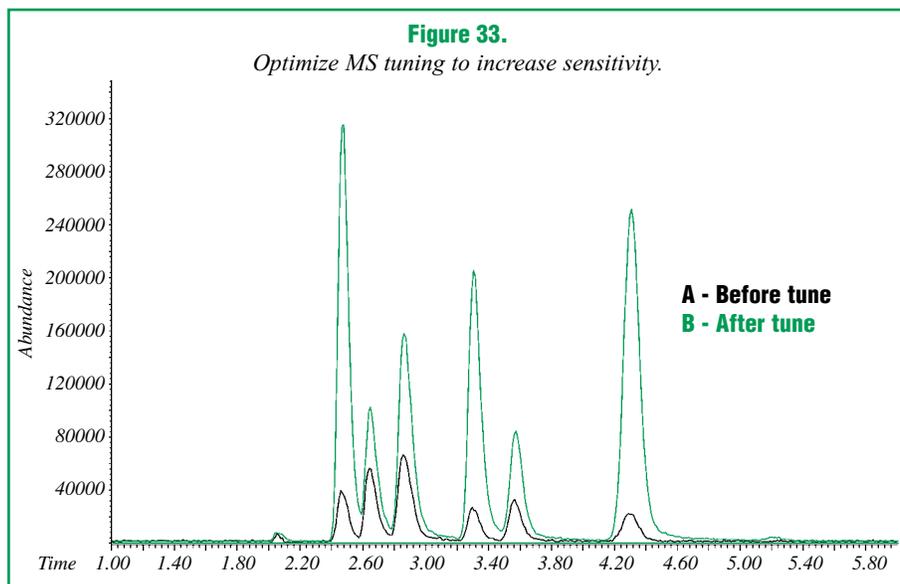
Figure 32.

Perfluorotributylamine (PFTBA) fragmentation, using a quadrupole mass spectrometer.

**Table IV.**

US EPA ion abundance criteria for 4-bromofluorobenzene (BFB).

Mass/Charge Ratio	Relative Abundance Criterion
50	15-50% of mass 95
75	30-80% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	<2% of mass 174
174	>50% of mass 95
175	5-9% of mass 174
176	>95% but <101% of mass 174
177	5-9% of mass 176



Leak Checks: The MS is a powerful tool for determining the presence of leaks in the GC/MS system because it is able to detect air and water. Check for leaks by turning on the PFTBA valve and scanning for m/z 69, 18, and 28. By using the base peak for PFTBA (m/z 69), a relative concentration of water/nitrogen can be determined. The combined relative abundance of 18/28 to 69 should be between 0.1% and 3%. Figure 34 shows an air/water value of 0.14% (0.05+0.09). If the value is below 0.1%, compare the current total abundance of ion 69 with its abundance at the last leak check. Instruments with large leaks have reduced sensitivity for 69 and may show abundances of 0 for lower ions, suggesting there is no leak. This is due to saturation of the detector. If a leak is present, the instrument will not tune. An MS with a diffusion pump should be allowed more time to equilibrate because it is less efficient at removing low molecular weight contamination. After this preliminary air/water check, begin tuning the instrument. After the instrument passes tuning, check again for air and water.

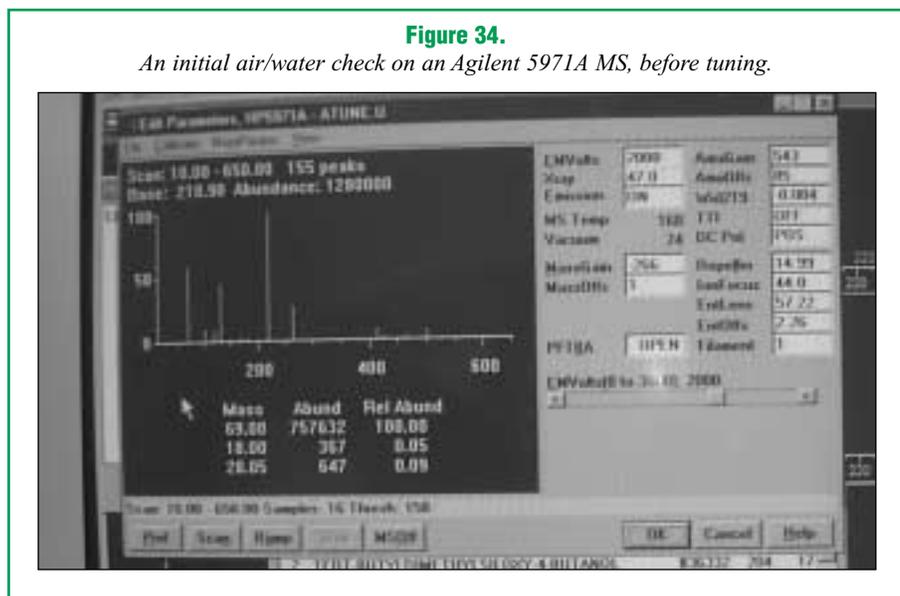
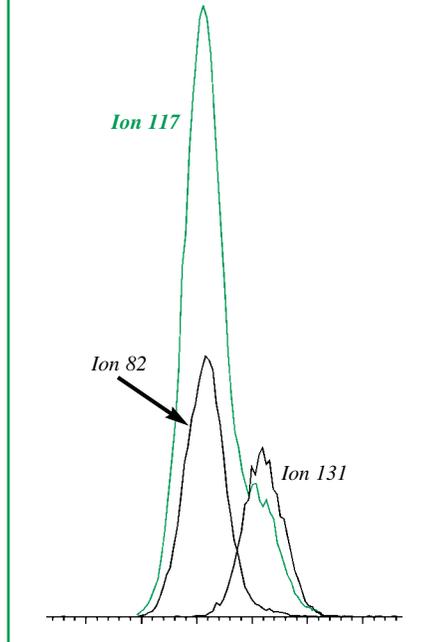
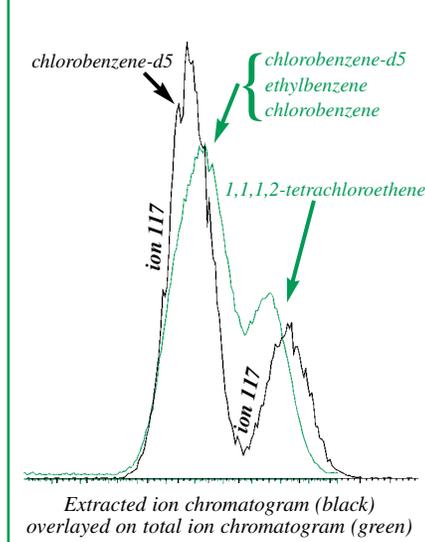


Figure 35.

The quantification ion for chlorobenzene-d5 can be changed from ion 117 to ion 82, to eliminate the need for chromatographic resolution from 1,1,1,2-tetrachloroethane.

**Figure 36.**

A slower oven temperature program eliminates the need to change the internal standard or the quantification ion.



Identifying Target Analytes

Qualitative identification of a target compound is based on retention time (± 0.06 minutes) and on comparison of the sample mass spectrum to a reference mass spectrum. Compounds are identified from three ions of the greatest intensity. The quantification ion, usually the highest m/z fragment, is used for determining the concentration of a particular analyte. When using any column for GC/MS, attention must be given to coeluting compounds to determine if acceptable quantification ions can be found. It is important that there be no coelution between compounds sharing ions used for quantification. As long as unique ions can be selected for quantifying compounds that share retention time, chromatographic coelution is acceptable. Reducing analysis time without carefully checking for coelutions can lead to problems. For example, internal standard chlorobenzene-d5 and analyte 1,1,1,2-tetrachloroethane, which share quantification ion 117, can coelute from a "VMS," "624," or "VRX" stationary phase. Many laboratories eliminate the need for chromatographic resolution by changing the quantification ion for chlorobenzene-d5 from 117 to 82 (Figure 35). An alternative solution is to replace chlorobenzene-d5 with another internal standard that elutes in the same region of the chromatogram, such as 4-bromofluorobenzene. In performance-based measurement systems (PBMS), surrogates and internal standards may be changed, as long as the analyst can show that the performance will be equivalent or better. Auditors for state or local regulatory agencies might not allow these changes, however. An additional option, the suggested surrogates can be used and the analysis performed using a slower GC oven temperature program that resolves the coelution (Figure 36). This option eliminates the need to change either the internal standard or the quantification ion, but prolongs analysis time. An analysis time of less than 10 minutes, with chromatographic resolution of these compounds, is possible with a 20m x 0.18mm x 1.0 μ m df Rtx[®]-VMS column (see Application section, page 51).

Identifying Non-Target Analytes

For samples containing analytes that do not match retention time and/or mass spectra for the target compounds, a library search can be used in an attempt to match the unknown spectra with known spectra. Unknown compounds in the sample, referred to as *tentatively identified compounds* (TICs), should be reported only as estimates.

Mass Spectral Clues for Identifying TICs: The MS provides three valuable clues to identifying TICs: parent ion, isotopic composition, and common fragmentation ions. The parent ion (also known as the molecular weight ion) is the ionized form of the neutral compound, but not all compounds are stable enough to produce a molecular weight ion. Most environmental contaminants, except compounds that contain nitrogen, will have an even number mass for a parent ion. The parent ion reveals information about the elemental composition and the distribution of isotopes. The term "isotope" is used to describe atoms of an element with differing numbers of neutrons. Most elements have isotopes in a particular distribution to each other. For example, carbon occurs primarily in two forms, ¹²C and ¹³C. ¹³C is at an abundance of 1.1% relative to ¹²C.¹² This information can contribute to determining the number of carbons present in the fragmented ion. Carbon is considered an A+1 element because its isotopes vary by 1amu. Compounds such as oxygen, sulfur, silicon, chlorine, and bromine are A+2 elements because their isotopes vary by 2amu or more (Figure 37, page 35). Fragmentation ions also can offer clues to compound composition (Table V).

Leak-Free Column/MS Installation Using an Injection Port Connection

The most common problem associated with volatiles analyses by GC/MS is the presence of leaks. The following procedure will help ensure optimum performance. Do not use this procedure with columns with IDs larger than 0.25mm, because the amount of oxygen that would be introduced into the MS source during the last step will oxidize the metal parts and reduce sensitivity.

12. F.W. McLafferty and F. Turecek. *Interpretation of Mass Spectra*, University Science Books, Mill Valley, 4th edition, 1993, pp. 283-291.

Connect the capillary column, 0.25mm ID or smaller, to the injection port, but not to the MS source, and condition the column. When the column has been conditioned, remove 50cm from the detector end of the column to ensure complete removal of siloxanes and other potential contaminants. Then, with the MS still turned off, insert the column end into the MS source. Cut the column several centimeters from the connection to the injection port. Use septa to cap the short length of column that is left in the injection port and the new, unconnected inlet end of the column. Also cap the split vent and septum purge vent lines on the GC (Figure 38). Perform a pressure decay test on the injection port by setting the pressure to 30psi, then shutting off the gas supply. The pressure should remain constant for at least 1 minute. If the pressure drops in less than 1 minute, turn on the gas supply and begin leak checking, using an electronic leak detector, such as the Restek Leak Detective™ II (cat. #20413, page 36).

Once you have confirmed the GC system is leak-tight, and while the injection port end of the column is capped and there is no flow in the column, evacuate the MS and record the source pressure in your maintenance logbook. After several hours equilibration, perform the instrument leak-check using PFTBA (see MS Calibration and Tuning, page 32). If a leak is present, draw 500µL of methanol into a syringe and apply drops of this solvent on areas where leaks might be suspected, while scanning for mass 31. Alternatively, bathe these areas with argon gas and scan for m/z 40. The brass source nut is the primary place for leaks in an MS, this nut should be replaced every time the column is changed (cat. #20643, page 35). Other areas to examine include the rubber seals and the PFTBA vial.

After you confirm the MS is leak-free, quickly install the inlet end of the column into the injection port. The MS will draw air into the source during this connection time; after another 2-hour equilibration the MS is ready for tuning/leak checking using PFTBA.

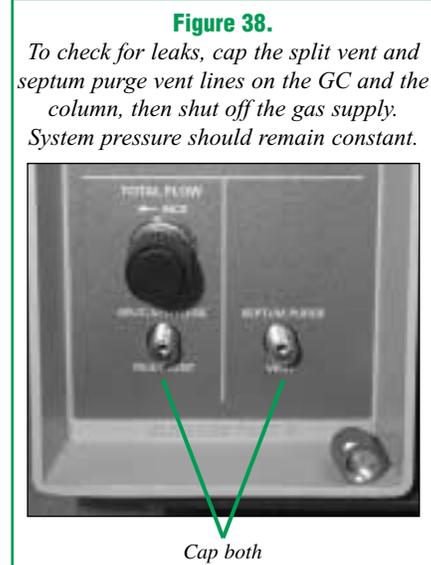


Figure 37.
Cl and Br are examples of A+2 elements (isotopes vary by 2amu or more).

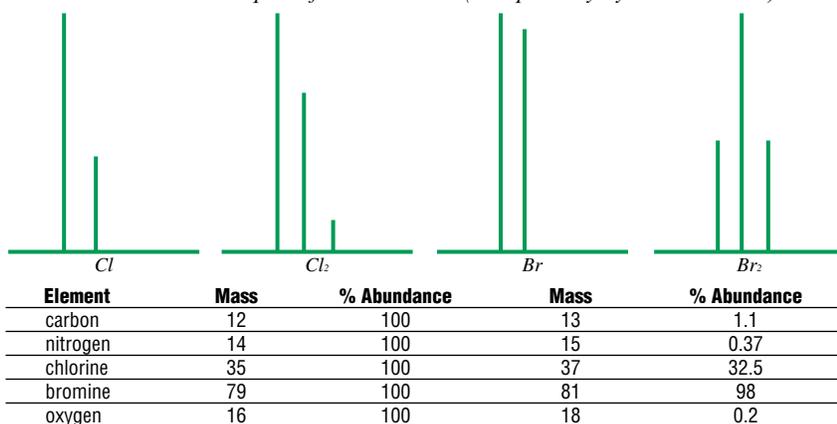


Table V.
Fragment ions can offer clues to compound composition.

Compound Class	Fragment Ions
Aldehydes, amides, amines	44, 58, 72, 59, 30
Aliphatic hydrocarbons	43, 57, 71, 85, 99
Alkylbenzenes	104, 91
Aromatic hydrocarbons	39, 50, 51, 52, 63, 65, 76, 77, 91
Fluorine-containing	50, 69
Methacrylates	41, 69
Methyl ketones	43, 58
Oxygen-containing	31, 45, 59, 73
Sulfur-containing	47, 61
Unsaturated hydrocarbons	41, 55, 69

MSD Source Nut

(Similar to Agilent part # 05988-20066.)

Description	qty.	cat.#
(Detector) MSD Source Nut	2-pk.	20643

Leak Detective™ II Leak Detector

- Affordable thermal conductivity leak detector—every analyst should have one*
- Compact, ergonomic design is easy to hold and operate with one hand.
- Helium, hydrogen, and nitrogen can be detected at 1×10^{-4} cc/sec. or at an absolute concentration as low as 100ppm**
- Fast results—responds in less than 2 seconds to trace leaks of gases with thermal conductivities different than air.
- Micro-chip design improves sensitivity and response time over previous models.
- Auto zeroing with the touch of a button.
- Battery-operated for increased portability (one 9-volt).



Description	qty.	cat.#
Leak Detective™ II Leak Detector	ea.	20413

* *Never use liquid leak detectors on a capillary system. Liquids can be drawn into the system.*

** **Caution:** *NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possibly hazardous conditions.*

MS Contamination

A universal detector, the MS responds to all organic compounds and, consequently, any contamination potentially can interfere with target analyte identification. The common sources of contamination are column bleed and septum bleed. High column bleed can be an indication of oxygen entering the system and degrading the stationary phase in the column. If high column bleed is suspected, conduct a leak check of the system, using the procedure described on page 34 and 35. Septum bleed introduces silicon fragments, characterized by ion 73, into the system. Silicon compounds also are components of GC and MS seals. Table VI lists common contaminants and the ions by which they can be identified.

Water / Methanol: The introduction of water and/or methanol vapor from the purge and trap system can cause problems in an MS system. Excess water vapor entering the MS can decrease the ionization of target analytes eluting at the same time. To overcome problems associated with water vapor, use a trap containing hydrophobic adsorbents, such as the Vocab® 3000 trap. If you are using an ion trap system, increase the split ratio in the injection port. This will prevent overloading the ion trap and will increase overall linearity for the gaseous analytes.

Table VI.

Common contaminants and their identifying ions.

Contaminant	Characteristic Fragmentation Ions
Silicon	73, 147, 207, 221, 281, 355, 429, 503
Rough vacuum pump oil	55-57, 61-67, 81-85, 95-99
Diffusion pump oil	77, 115, 141, 168, 223, 260, 446
Plasticizers	149, 223, 278

Practical Introduction to GC/MS Analysis with Quadrupoles

The text gives answers to questions such as: how does the mass spectrometer work, what problems can occur and how do I detect them, how must separation and detection be adapted to each other, and what pitfalls can be avoided when elucidating structures and quantifying compounds.

M. Oehme, Wiley-VCH
1999, 195pp., ISBN 3-527-29748-0
cat.# 21098



Interpretation of Mass Spectra, 4th Edition

This updated version builds on the strengths of the previous editions and presents the information required to clearly and concisely interpret mass spectra. Chapters include information on elemental composition, molecular ions, mechanisms of ion fragmentations, unimolecular ion decompositions, and mass spectra of common compound classes. It is valuable and necessary resource for every person practicing mass spectrometry.

F.W. McLafferty and F. Turecek, University Science
1993, 371pp., ISBN 0-935702-25-3
cat.# 20498



Applications Using GC Detection Systems

Purge and Trap Applications Using Tandem PID-ELCD: US EPA Methods 502.2, 601, 602, 8010, 8020, 8021B

EPA methods for GC analyses of volatile compounds require purge and trap units for concentrating the contaminants in water, soil, or wastewater. While purge and trap concentration significantly increases sensitivity, relative to other sample introduction techniques, it does have a downside: early-eluting volatile compounds typically exhibit broad peaks, due to inefficient sample transfer from the trap to the GC. This distorted peak shape decreases resolution between closely eluting compounds, placing demands on the analytical system and requiring optimized GC operating conditions. Although cryofocusing improves separations of early eluting compounds, most environmental laboratories do not use this approach because it increases costs.

EPA methods for monitoring volatiles by GC often recommend using a PID and an ELCD, connected in tandem or series. Coelutions of target compounds are allowed, as long as they are resolved by the detectors.¹³ For example, in Figure 39 bromoform and styrene elute with the same retention time, but bromoform elicits a response only from the ELCD and styrene elicits a response only from the PID. Thus, the selective detectors resolve these two compounds. Because it characteristically produces tailing peaks, the ELCD is the more problematic of the two detectors; sensitivity can be increased, but not without a sacrifice in peak shape. Optimization of an ELCD minimizes tailing and maximizes sensitivity.

Analysis Time: Several factors contribute to the total analysis time for volatiles separations, including purge and trap cycle time, sample analysis time, and GC oven cool-down time (time required for the oven to cool from the final temperature to the initial temperature for the next analysis). Long purge and trap cycles are a product of long purge times, dry purges, long desorb times, and long trap bake times. Long oven cycle times result from low initial oven temperatures (i.e., subambient to 35°C) and slow temperature program rates. A column that unnecessarily exceeds the length needed to resolve the analytes can increase analysis time and cost without significantly adding to the data obtained.

An Rtx[®]-VGC primary column paired with an Rtx[®]-VRX confirmation column make a good combination for analyzing the compounds listed in Figures 39A & B. The target list includes unregulated but commonly analyzed compounds such as methyl-*tert*-butyl ether (MTBE) and Freon[®] 113 (1,1,2-trichloro-1,2,2-trifluoroethane). A 35°C starting temperature is necessary to resolve Freon[®]113 from 1,1-dichloroethane. Figure 39A shows there are no early-analyte coelution problems on the primary column when using PID/ELCD detectors in tandem – the gases and the trihalomethanes are separated.

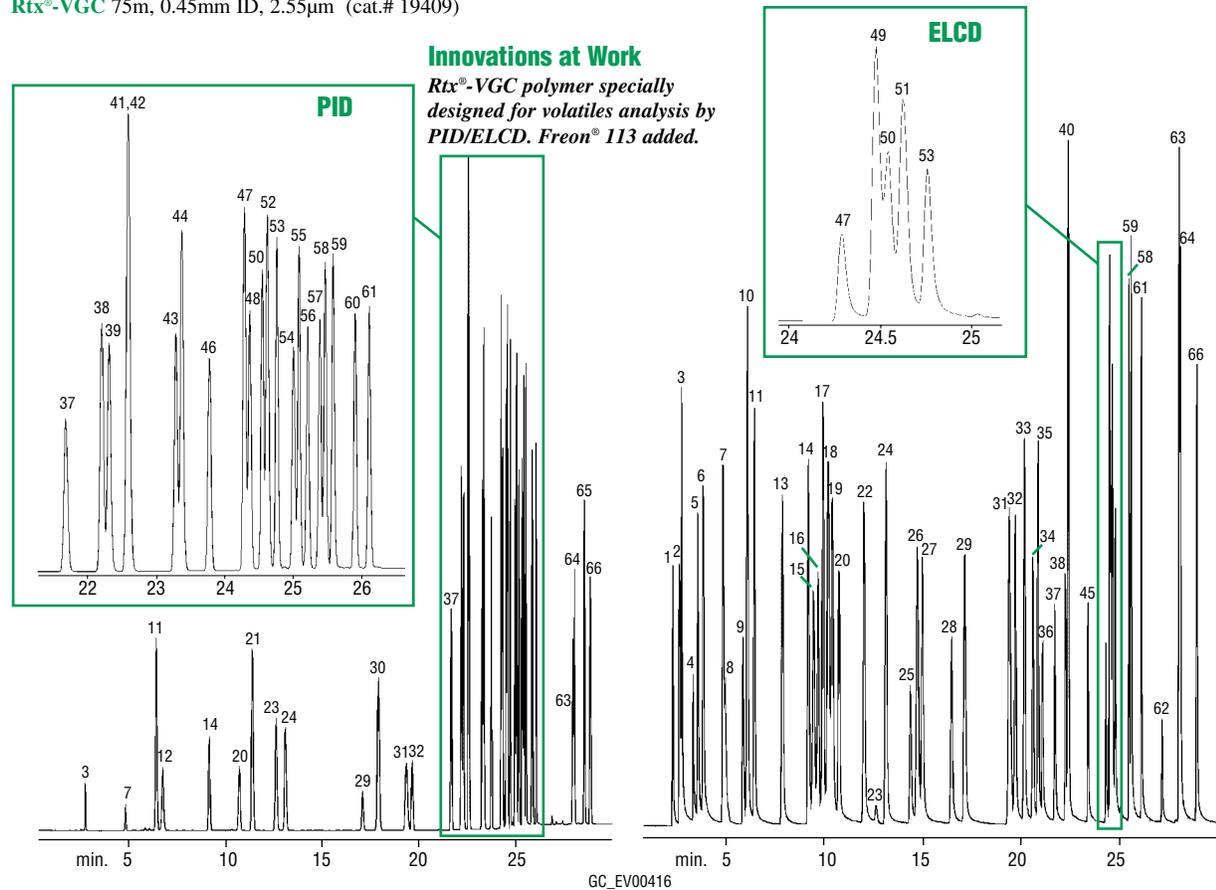
Figures 40A & B show the analysis of Method 8021A/502.2 compounds, without Freon[®] 113, using an Rtx[®]-VGC column and an Rtx[®]-502.2 column. A 50°C initial oven temperature can be used, which greatly reduces the time needed for the GC to complete the oven cycle and return to the starting temperature (cycle time) and, therefore, increases throughput. An Agilent 5890 GC oven will cool from 205°C to 35°C in 9 minutes; this time, added to the 28-minute analysis time in Figure 39, produces the fastest cycle time for this analysis: 37 minutes. In the analysis in Figure 40, the starting temperature is 50°C, the final temperature is 200°C, and the oven takes 4 minutes to cool. The total cycle time, less than 30 minutes, is significantly faster than for other pairs of columns. For example, an Rtx[®]-VRX column requires a starting temperature of 40°C; this, combined with a 28 minute analysis time, means the total cycle time cannot be faster than 35 minutes.

13. EPA Method 8000B, *Determinative Chromatographic Separations*; US EPA. U.S. Government Printing Office: Washington, DC, 1996, Rev. 2.

Figure 39A.

An Rtx®-VGC primary column and an Rtx®-VRX confirmation column separate volatile organic gases from the trihalomethanes – there are no coelutions on the primary column using the PID/ELCD detectors in tandem.

Rtx®-VGC 75m, 0.45mm ID, 2.55µm (cat.# 19409)



20ppb in 5mL of RO water.

Primary column: 75m, 0.45mm ID, 2.55 m Rtx®-VGC (cat.# 19409)

Confirmation column: 75m, 0.45mm ID, 2.55 m Rtx®-VRX (cat.# 19309)

Concentrator: Tekmar LSC-3000 Purge and Trap

Trap: Vocarb 3000

Purge: 11 min. @ 40mL/min.

Dry purge: 1 min. @ 40mL/min. (MCS by-passed with Silcosteel® tubing [cat.# 21035])

Desorb preheat: 245 C

Desorb: 250 C for 2 min.

Bake: 260 C for 8 min.

Interface: direct

Transfer line: 0.32mm ID Sittek tubing (Cat. #10027)

GC: Finnigan 9001

Oven temp.: 35 C (hold 4 min.) to 75 C @ 3 C/min. (hold 2 min.) to 175 C @ 21 C/min. to 205 C @ 35 C/min. (hold 5 min.)

Carrier gas: helium 11mL/min., constant pressure

Adjust dichlorodifluoromethane to a retention time of 2.28 min. @ 35 C on the Rtx®-VGC column.

Detectors: Gold Tandem PID/HALL 2000 ELCD

PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV, base temp 200 C.

Hall 2000 ELCD: RxnGas 25mL/min., RxnTemp. 940 C, propanol flow 470 L/min.

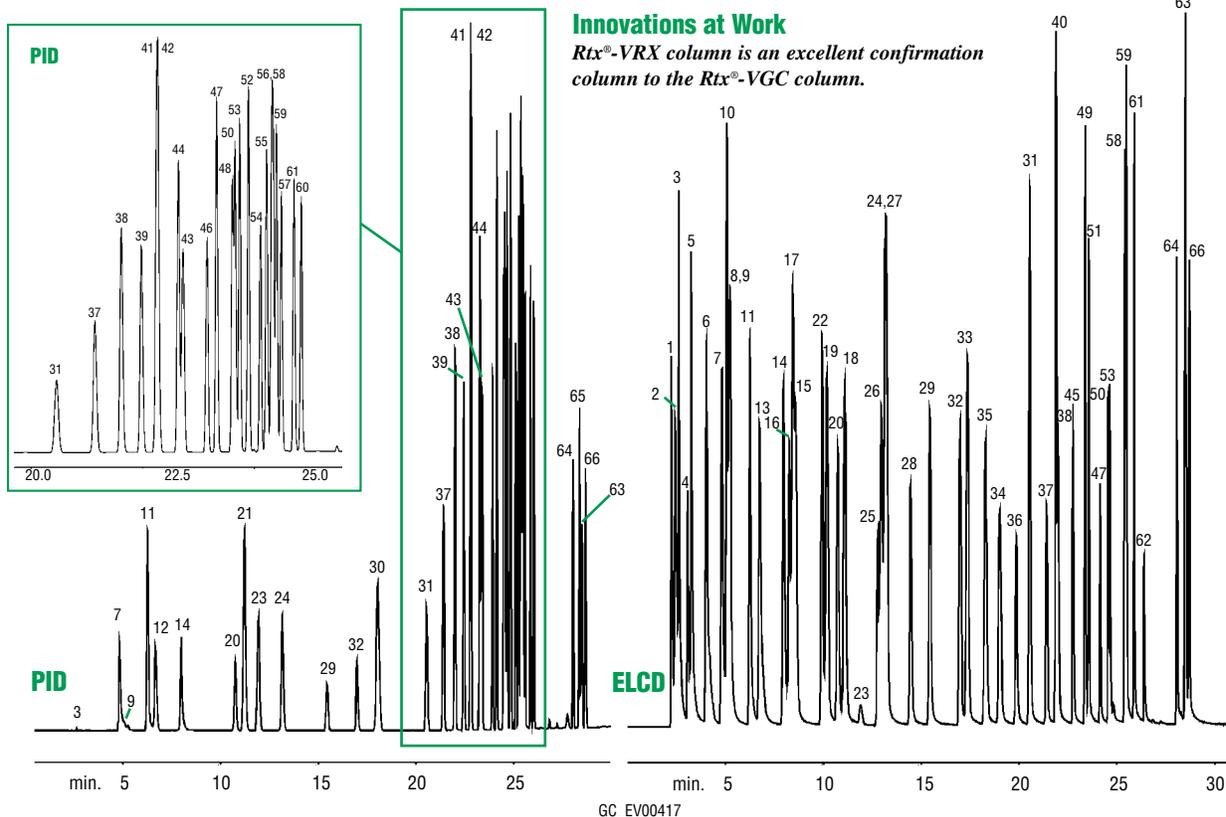
- | | | | |
|--------------------------------------|---------------------------------------|-----------------------------------|---------------------------------|
| 1. dichlorodifluoromethane | 18. carbon tetrachloride | 35. 1,3-dichloropropane | 52. 1,3,5-trimethylbenzene |
| 2. chloromethane | 19. 1,1,1-trichloroethane | 36. 1,2-dibromoethane | 53. 4-chlorotoluene |
| 3. vinyl chloride | 20. 1,1-dichloropropene | 37. 1-chloro-3-fluorobenzene (SS) | 54. <i>tert</i> -butylbenzene |
| 4. bromomethane | 21. benzene | 38. chlorobenzene | 55. 1,2,4-trimethylbenzene |
| 5. chloroethane | 22. 1,2-dichloroethane | 39. ethylbenzene | 56. <i>sec</i> -butylbenzene |
| 6. trichlorofluoromethane | 23. fluorobenzene (SS) | 40. 1,1,1,2-tetrachloroethane | 57. <i>p</i> -isopropyltoluene |
| 7. 1,1-dichloroethene | 24. trichloroethene | 41. <i>m</i> -xylene | 58. 1,3-dichlorobenzene |
| 8. Freon® 113 | 25. dibromomethane | 42. <i>p</i> -xylene | 59. 1,4-dichlorobenzene |
| 9. allyl chloride | 26. 1,2-dichloropropane | 43. <i>o</i> -xylene | 60. <i>n</i> -butylbenzene |
| 10. methylene chloride | 27. bromodichloromethane | 44. styrene | 61. 1,2-dichlorobenzene |
| 11. <i>trans</i> -1,2-dichloroethene | 28. 1-bromo-2-chloroethane (SS) | 45. bromoform | 62. 1,2-dibromo-3-chloropropane |
| 12. methyl <i>tert</i> -butyl ether | 29. <i>cis</i> -1,3-dichloropropene | 46. isopropylbenzene | 63. hexachlorobutadiene |
| 13. 1,1-dichloroethane | 30. toluene | 47. bromobenzene | 64. 1,2,4-trichlorobenzene |
| 14. <i>cis</i> -1,2-dichloroethene | 31. tetrachloroethene | 48. <i>n</i> -propylbenzene | 65. naphthalene |
| 15. 2,2-dichloropropane | 32. <i>trans</i> -1,3-dichloropropene | 49. 1,1,2,2-tetrachloroethane | 66. 1,2,3-trichlorobenzene |
| 16. bromochloromethane | 33. 1,1,2-trichloroethane | 50. 2-chlorotoluene | |
| 17. chloroform | 34. dibromochloromethane | 51. 1,2,3-trichloropropane | |

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL® 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

Figure 39B.

An Rtx[®]-VGC primary column and an Rtx[®]-VRX confirmation column separate volatile organic gases from the trihalomethanes – there are no coelutions on the primary column using the PID/ELCD detectors in tandem.

Rtx[®]-VRX 75m, 0.45mm ID, 2.55µm (cat.# 19309)



20ppb in 5mL of RO water.

Primary column: 75m, 0.45mm ID, 2.55µm Rtx[®]-VGC (cat.# 19409)
 Confirmation column: 75m, 0.45mm ID, 2.55µm Rtx[®]-VRX (cat.# 19309)
 Concentrator: Tekmar LSC-3000 Purge and Trap
 Trap: Vocarb[™] 3000
 Purge: 11 min. @ 40mL/min.
 Dry purge: 1 min. @ 40mL/min. (MCS by-passed with Silcosteel[®] tubing [cat.# 21035])
 Desorb preheat: 245°C
 Desorb: 250°C for 2 min.
 Bake: 260°C for 8 min.
 Interface: direct
 Transfer line: 0.32mm ID Siltek[™] tubing (Cat. #10027)

GC: Finnigan 9001
 Oven temp.: 35°C (hold 4 min.) to 75°C @ 3°C/min. (hold 2 min.) to 175°C @ 21°C/min. to 205°C @ 35°C/min. (hold 5 min.)
 Carrier: helium 1 mL/min., constant pressure
 Adjust dichlorodifluoromethane to a retention time of 2.28 min. @ 35°C on the Rtx[®]-VGC column.
 Detectors: µGold Tandem PID/HALL 2000 ELCD
 PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV, base temp 200°C.
 Hall 2000 ELCD: RxnGas 25mL/min., RxnTemp. 940°C, propanol flow 470µL/min.

1. dichlorodifluoromethane	18. carbon tetrachloride	35. 1,3-dichloropropane	52. 1,3,5-trimethylbenzene
2. chloromethane	19. 1,1,1-trichloroethane	36. 1,2-dibromoethane	53. 4-chlorotoluene
3. vinyl chloride	20. 1,1-dichloropropene	37. 1-chloro-3-fluorobenzene (SS)	54. <i>tert</i> -butylbenzene
4. bromomethane	21. benzene	38. chlorobenzene	55. 1,2,4-trimethylbenzene
5. chloroethane	22. 1,2-dichloroethane	39. ethylbenzene	56. <i>sec</i> -butylbenzene
6. trichlorofluoromethane	23. fluorobenzene (SS)	40. 1,1,1,2-tetrachloroethane	57. <i>p</i> -isopropyltoluene
7. 1,1-dichloroethene	24. trichloroethene	41. <i>m</i> -xylene	58. 1,3-dichlorobenzene
8. Freon [®] 113	25. dibromomethane	42. <i>p</i> -xylene	59. 1,4-dichlorobenzene
9. allyl chloride	26. 1,2-dichloropropane	43. <i>o</i> -xylene	60. <i>n</i> -butylbenzene
10. methylene chloride	27. bromodichloromethane	44. styrene	61. 1,2-dichlorobenzene
11. <i>trans</i> -1,2-dichloroethene	28. 1-bromo-2-chloroethane (SS)	45. bromoform	62. 1,2-dibromo-3-chloropropane
12. methyl <i>tert</i> -butyl ether	29. <i>cis</i> -1,3-dichloropropene	46. isopropylbenzene	63. hexachlorobutadiene
13. 1,1-dichloroethane	30. toluene	47. bromobenzene	64. 1,2,4-trichlorobenzene
14. <i>cis</i> -1,2-dichloroethene	31. tetrachloroethene	48. <i>n</i> -propylbenzene	65. naphthalene
15. 2,2-dichloropropane	32. <i>trans</i> -1,3-dichloropropene	49. 1,1,2,2-tetrachloroethane	66. 1,2,3-trichlorobenzene
16. bromochloromethane	33. 1,1,2-trichloroethane	50. 2-chlorotoluene	
17. chloroform	34. dibromochloromethane	51. 1,2,3-trichloropropane	

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL[®] 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

Figure 40A.

An Rtx®-VGC / Rtx®-502.2 column pair and a 50°C initial temperature reduce total cycle time to less than 30 minutes for EPA Method 8021A/502.2.

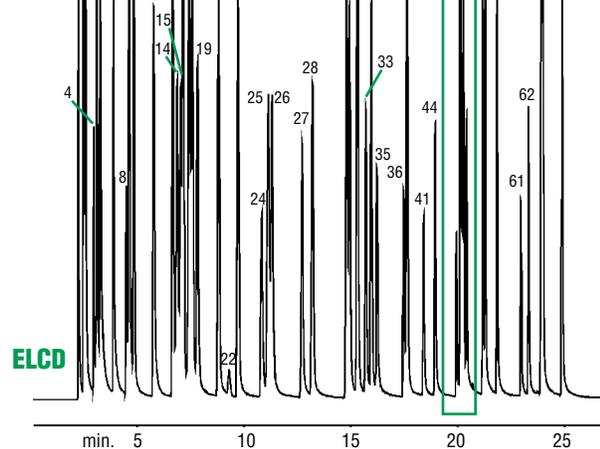
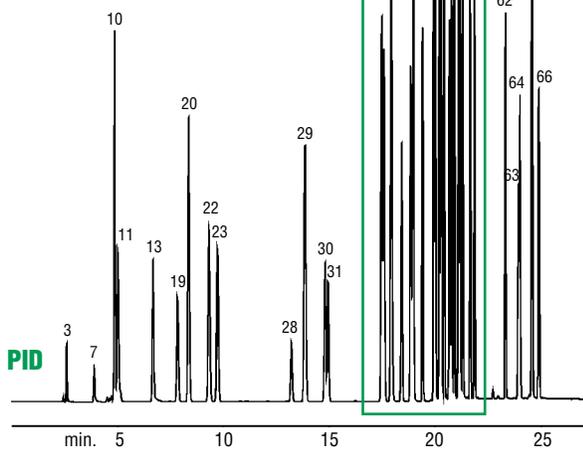
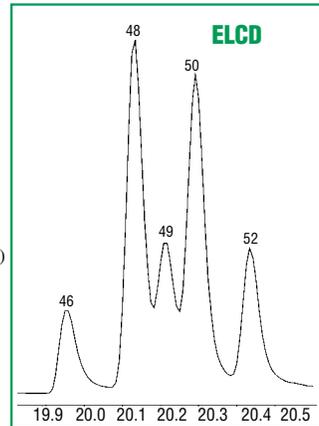
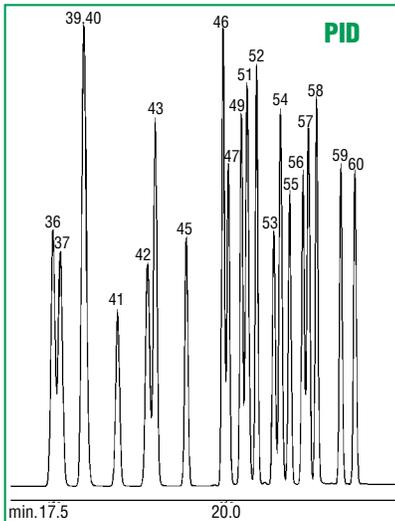
Innovations at Work

Fastest cycle time for 8021/502.2!
50°C starting temperature brings total cycle time to less than 30 min.

Rtx®-VGC 75m, 0.45mm ID, 2.55µm (cat.# 19409)

Primary column: 75m, 0.45mm ID, 2.55 m Rtx®-VGC (cat.# 19409)
 Confirmation column: 75m, 0.45mm ID, 2.55 m Rtx®-502.2 (cat.# 10986)
 Conc.: 10ppb in 5mL of RO water
 Concentrator: Tekmar LSC3100 Purge and Trap
 Trap: Vocarb 3000
 Purge: 11 min. @ 40mL/min.
 Dry purge: 1 min. @ 40mL/min.
 Desorb preheat: 245 C
 Desorb: 250 C for 2 min.
 Bake: 260 C for 8 min.
 Interface: direct connection from concentrator to column
 Transfer line: Siltek 0.32mm fused silica transfer line direct to columns w/ Press-Tight Y connector (cat. # 20403)

Gas chromatograph: Finnigan 9001
 Carrier gas: helium @ ~10mL/min. constant pressure
 Adjust dichlorodifluoromethane to a retention time of 2.28 min. @ 50 C on the Rtx®-VGC column.
 50 C (hold 2 min.) to 70 C @ 2 C/min. to 130 C @ to 200 C @ 40 C/min. (final hold 5 min.)
 Gold Tandem PID/Hall 2000 ELCD
 PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV base temp 200 C.
 Hall 2000 ELCD: RxnGas 25mL/min., Temp.940 C, propanol flow 470 L/min.



- | | | | | |
|--------------------------------------|-------------------------------------|---------------------------------------|--------------------------------|----------------------------------|
| 1. dichlorodifluoromethane | 15. bromochloromethane | 29. toluene | 43. styrene | 57. 1,3-dichlorobenzene |
| 2. chloromethane | 16. chloroform | 30. tetrachloroethene | 44. bromoform | 58. 1,4-dichlorobenzene |
| 3. vinyl chloride | 17. carbon tetrachloride | 31. <i>trans</i> -1,3-dichloropropene | 45. isopropylbenzene | 59. <i>n</i> -butylbenzene |
| 4. bromomethane | 18. 1,1,1-trichloroethane | 32. 1,1,2-trichloroethane | 46. bromobenzene | 60. 1,2-dichlorobenzene |
| 5. chloroethane | 19. 1,1-dichloropropene | 33. dibromochloromethane | 47. <i>n</i> -propylbenzene | 61. 1,2-dibromo-3-chloropropane |
| 6. trichlorofluoromethane | 20. benzene | 34. 1,3-dichloropropane | 48. 1,1,2,2-tetrachloroethane | 62. 2-bromo-1-chlorobenzene (SS) |
| 7. 1,1-dichloroethene | 21. 1,2-dichloroethane | 35. 1,2-dibromoethane | 49. 2-chlorotoluene | 63. hexachlorobutadiene |
| 8. allyl chloride | 22. fluorobenzene (SS) | 36. chlorobenzene | 50. 1,2,3-trichloropropane | 64. 1,2,4-trichlorobenzene |
| 9. methylene chloride | 23. trichloroethene | 37. ethylbenzene | 51. 1,3,5-trimethylbenzene | 65. naphthalene |
| 10. <i>trans</i> -1,2-dichloroethene | 24. dibromomethane | 38. 1,1,1,2-tetrachloroethane | 52. 4-chlorotoluene | 66. 1,2,3-trichlorobenzene |
| 11. methyl <i>tert</i> -butyl ether | 25. 1,2-dichloropropane | 39. <i>m</i> -xylene | 53. <i>tert</i> -butylbenzene | |
| 12. 1,1-dichloroethane | 26. bromodichloromethane | 40. <i>p</i> -xylene | 54. 1,2,4-trimethylbenzene | |
| 13. <i>cis</i> -1,2-dichloroethene | 27. 1-bromo-2-chloroethane (SS) | 41. 1-chloro-2-fluorobenzene (SS) | 55. <i>sec</i> -butylbenzene | |
| 14. 2,2-dichloropropane | 28. <i>cis</i> -1,3-dichloropropene | 42. <i>o</i> -xylene | 56. <i>p</i> -isopropyltoluene | |

GC_EV00418

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL® 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

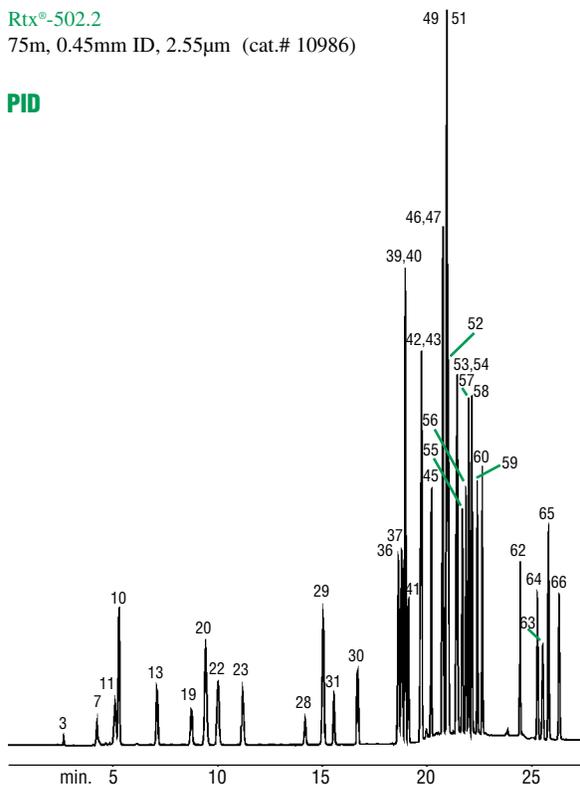
Figure 40B.

An Rtx®-VGC / Rtx®-502.2 column pair and a 50°C initial temperature reduce total cycle time to less than 30 minutes for EPA Method 8021A/502.2.

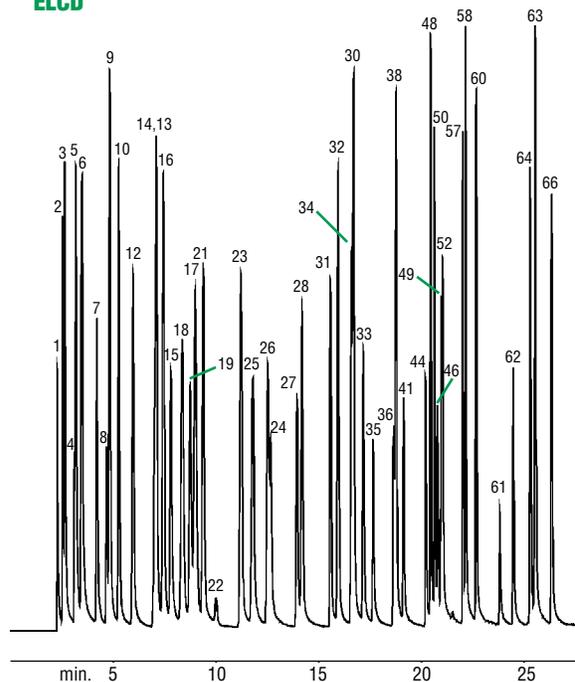
Rtx®-502.2

75m, 0.45mm ID, 2.55µm (cat.# 10986)

PID



ELCD



GC_EV00419

- | | |
|---------------------------------------|-----------------------------------|
| 1. dichlorodifluoromethane | 34. 1,3-dichloropropane |
| 2. chloromethane | 35. 1,2-dibromoethane |
| 3. vinyl chloride | 36. chlorobenzene |
| 4. bromomethane | 37. ethylbenzene |
| 5. chloroethane | 38. 1,1,1,2-tetrachloroethane |
| 6. trichlorofluoromethane | 39. <i>m</i> -xylene |
| 7. 1,1-dichloroethene | 40. <i>p</i> -xylene |
| 8. allyl chloride | 41. 1-chloro-2-fluorobenzene (SS) |
| 9. methylene chloride | 42. <i>o</i> -xylene |
| 10. <i>trans</i> -1,2-dichloroethene | 43. styrene |
| 11. methyl <i>tert</i> -butyl ether | 44. bromoform |
| 12. 1,1-dichloroethane | 45. isopropylbenzene |
| 13. <i>cis</i> -1,2-dichloroethene | 46. bromobenzene |
| 14. 2,2-dichloropropane | 47. <i>n</i> -propylbenzene |
| 15. bromochloromethane | 48. 1,1,2,2-tetrachloroethane |
| 16. chloroform | 49. 2-chlorotoluene |
| 17. carbon tetrachloride | 50. 1,2,3-trichloropropane |
| 18. 1,1,1-trichloroethane | 51. 1,3,5-trimethylbenzene |
| 19. 1,1-dichloropropene | 52. 4-chlorotoluene |
| 20. benzene | 53. <i>tert</i> -butylbenzene |
| 21. 1,2-dichloroethane | 54. 1,2,4-trimethylbenzene |
| 22. fluorobenzene (SS) | 55. <i>sec</i> -butylbenzene |
| 23. trichloroethene | 56. <i>p</i> -isopropyltoluene |
| 24. dibromomethane | 57. 1,3-dichlorobenzene |
| 25. 1,2-dichloropropane | 58. 1,4-dichlorobenzene |
| 26. bromodichloromethane | 59. <i>n</i> -butylbenzene |
| 27. 1-bromo-2-chloroethane (SS) | 60. 1,2-dichlorobenzene |
| 28. <i>cis</i> -1,3-dichloropropene | 61. 1,2-dibromo-3-chloropropane |
| 29. toluene | 62. 2-bromo-1-chlorobenzene (SS) |
| 30. tetrachloroethene | 63. hexachlorobutadiene |
| 31. <i>trans</i> -1,3-dichloropropene | 64. 1,2,4-trichlorobenzene |
| 32. 1,1,2-trichloroethane | 65. naphthalene |
| 33. dibromochloromethane | 66. 1,2,3-trichlorobenzene |

Primary column: 75m, 0.45mm ID, 2.55µm Rtx®-VGC (cat.# 19409)
 Confirmation column: 75m, 0.45mm ID, 2.55µm Rtx®-502.2 (cat.# 10986)
 Conc.: 10ppb in 5mL of RO water
 Concentrator: Tekmar LSC3100 Purge and Trap
 Trap: Vocarb 3000
 Purge: 11 min. @ 40mL/min.
 Dry purge: 1 min. @ 40mL/min.
 Desorb preheat: 245°C
 Desorb: 250°C for 2 min.
 Bake: 260°C for 8 min.
 Interface: direct connection from concentrator to column
 Transfer line: Siltek 0.32mm fused silica transfer line direct to columns w/ Press-Tight "Y" connector (cat. #20403)

Gas chromatograph: Finnigan 9001
 Carrier gas: helium @ ~10 mL/min. constant pressure
 Adjust dichlorodifluoromethane to a retention time of 2.28 min. @ 50°C on the Rtx®-VGC column.

Oven temp.: 50°C (hold 2 min.) to 70°C @ 2°C/min. to 130°C @ 9°C/min. to 200°C @ 40°C/min. (final hold 5 min.)

Detectors: µGold Tandem PID/Hall 2000 ELCD
 PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV base temp 200°C.
 Hall 2000 ELCD: RxnGas 25mL/min., RxnTemp. 940°C, propanol flow 470µL/min.

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL® 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

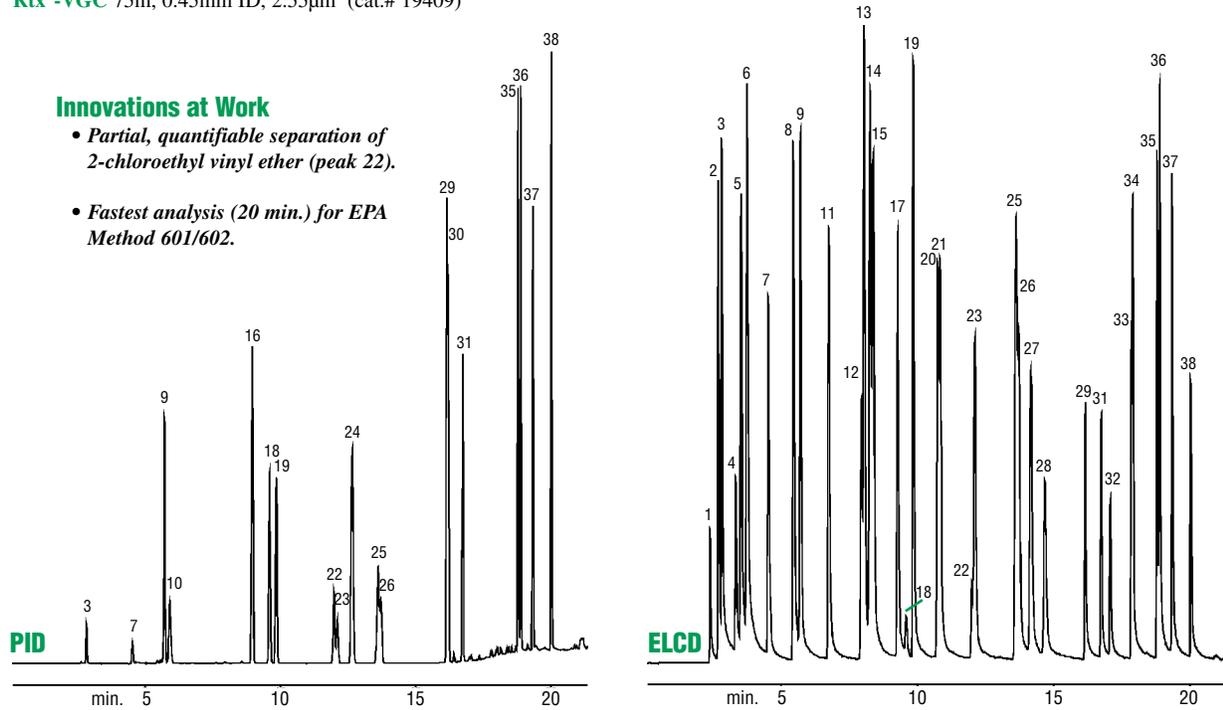
Figure 41A.

Analytes listed in EPA Method 601/602 can be separated in 20 minutes by an Rtx®-VGC / Rtx®-502.2 column pair.

Rtx®-VGC 75m, 0.45mm ID, 2.55µm (cat.# 19409)

Innovations at Work

- Partial, quantifiable separation of 2-chloroethyl vinyl ether (peak 22).
- Fastest analysis (20 min.) for EPA Method 601/602.



GC_EV00420

- | | |
|-------------------------------------|---------------------------------------|
| 1. dichlorodifluoromethane | 20. 1,2-dichloropropane |
| 2. chloromethane | 21. bromodichloromethane |
| 3. vinyl chloride | 22. 2-chloroethyl vinyl ether |
| 4. bromomethane | 23. <i>cis</i> -1,3-dichloropropene |
| 5. chloroethane | 24. toluene |
| 6. trichlorofluoromethane | 25. tetrachloroethene |
| 7. 1,1-dichloroethene | 26. <i>trans</i> -1,3-dichloropropene |
| 8. methylene chloride | 27. 1,1,2-trichloroethane |
| 9. <i>trans</i> -1,2-dichloroethene | 28. dibromochloromethane |
| 10. methyl <i>tert</i> -butyl ether | 29. chlorobenzene |
| 11. 1,1-dichloroethane | 30. ethylbenzene |
| 12. bromochloromethane (SS) | 31. 1-chloro-2-fluorobenzene (SS) |
| 13. chloroform | 32. bromoform |
| 14. carbon tetrachloride | 33. 1,4-dichlorobutane (SS) |
| 15. 1,1,1-trichloroethane | 34. 1,1,2,2-tetrachloroethane |
| 16. benzene | 35. 1,3-dichlorobenzene |
| 17. 1,2-dichloroethane | 36. 1,4-dichlorobenzene |
| 18. fluorobenzene (SS) | 37. 1,2-dichlorobenzene |
| 19. trichloroethene | 38. 4-bromo-1-chlorobenzene (SS) |

suggested surrogates: peaks 12, 18, 31, & 38

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL® 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

Primary column: 75m, 0.45mm ID, 2.55µm Rtx®-VGC (cat.# 19409)
 Confirmation column: 75m, 0.45mm ID, 2.55µm Rtx®-502.2 (cat.# 10986)
 Conc.: 10ppb in 5mL of RO water
 Concentrator: Tekmar LSC3100 Purge and Trap
 Trap: Vocabr 3000
 Purge: 11 min. @ 40mL/min.
 Dry purge: 1 min. @ 40mL/min.
 Desorb preheat: 245°C
 Desorb: 250°C for 2 min.
 Bake: 260°C for 8 min.
 Interface: direct connection from concentrator to column
 Transfer line: 0.53mm ID Silcosteel® tubing (cat. #70045)
 Gas chromatograph: Finnigan 9001
 Carrier gas: helium @ ~10mL/min. constant pressure
 Adjust dichlorodifluoromethane to a retention time of 2.47 min. @ 40°C.
 Oven temp.: 40°C (hold 2 min.) to 58°C @ 4°C/min. to 90°C @ 10°C/min. (hold 5 min.) to 220°C @ 40°C/min. (hold 5 min.)
 Detectors: µGold Tandem PID/Hall 2000 ELCD
 PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV base temp: 200 C.
 Hall 2000 ELCD: RxnGas 25mL/min., Rxn Temp. 940 C, propanol flow 470 L/min.

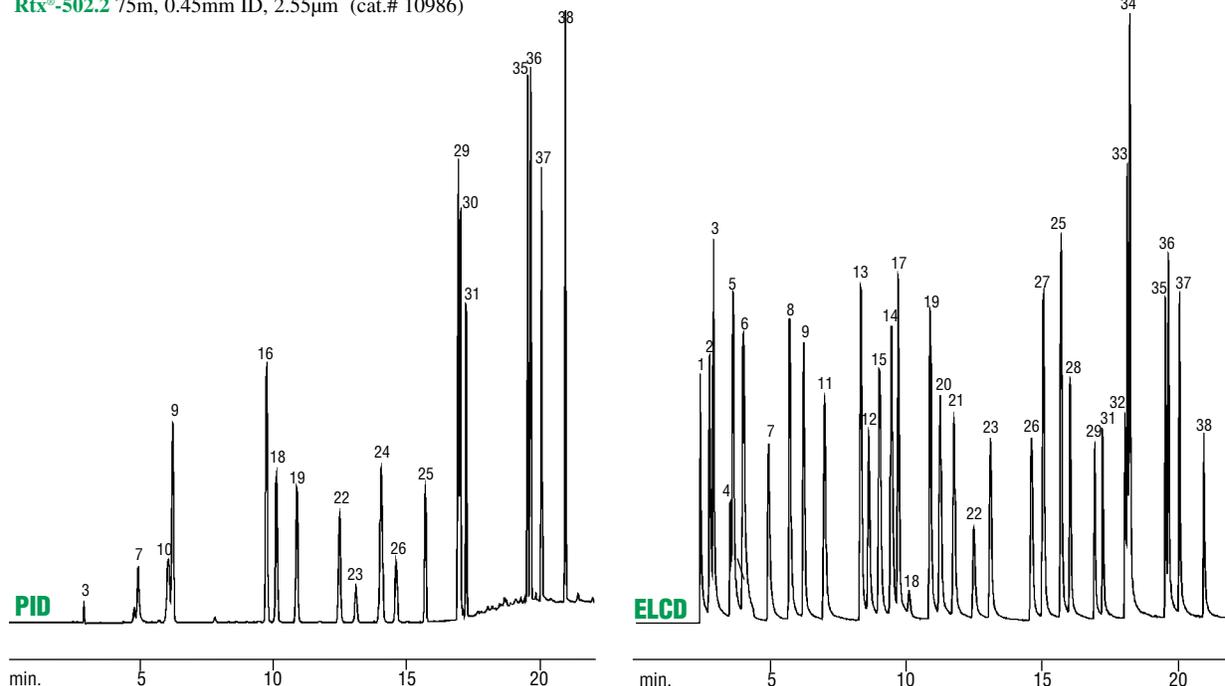
Standard	cat.#	Standard	cat.#
502.2 mix#1	30042	fluorobenzene	30030
624 cal mix #2	30021	1-chloro-2-fluorobenzene	30040
624 cal mix #3	30022	4-bromo-1-chlorobenzene	30230
MTBE	30402	bromochloromethane	30225
1,4-dichlorobutane	30227		

Client compound lists might match the compound list in Method 601/602, but the calibration criteria and low detection limits of Method 8021 are enforced. Figures 41A & B illustrate common compounds analyzed by GC/PID-ELCD, along with 2-chloroethyl vinyl ether. The starting temperature is 40°C, and the analysis time is 20 minutes. An Rtx®-502.2 column is a good choice for this analysis because it exhibits good resolution for 2-chloroethyl vinyl ether.

Figure 41B.

Analytes listed in EPA Method 601/602 can be separated in 20 minutes by an Rtx®-VGC / Rtx®-502.2 column pair.

Rtx®-502.2 75m, 0.45mm ID, 2.55µm (cat.# 10986)



GC_EV00540

- | | |
|-------------------------------------|---------------------------------------|
| 1. dichlorodifluoromethane | 21. bromodichloromethane |
| 2. chloromethane | 22. 2-chloroethyl vinyl ether |
| 3. vinyl chloride | 23. <i>cis</i> -1,3-dichloropropene |
| 4. bromomethane | 24. toluene |
| 5. chloroethane | 25. tetrachloroethene |
| 6. trichlorofluoromethane | 26. <i>trans</i> -1,3-dichloropropene |
| 7. 1,1-dichloroethene | 27. 1,1,2-trichloroethane |
| 8. methylene chloride | 28. dibromochloromethane |
| 9. <i>trans</i> -1,2-dichloroethene | 29. chlorobenzene |
| 10. methyl <i>tert</i> -butyl ether | 30. ethylbenzene |
| 11. 1,1-dichloroethane | 31. 1-chloro-2-fluorobenzene (SS) |
| 12. bromochloromethane (SS) | 32. bromoform |
| 13. chloroform | 33. 1,4-dichlorobutane |
| 14. carbon tetrachloride | 34. 1,1,2,2-tetrachloroethane |
| 15. 1,1,1-trichloroethane | 35. 1,3-dichlorobenzene |
| 16. benzene | 36. 1,4-dichlorobenzene |
| 17. 1,2-dichloroethane | 37. 1,2-dichlorobenzene |
| 18. fluorobenzene (SS) | 38. 4-bromo-1-chlorobenzene (SS) |
| 19. trichloroethene | |
| 20. 1,2-dichloropropane | |

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL[®] 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

Primary column: 75m, 0.45mm ID, 2.55 m Rtx[®]-VGC (cat.# 19409)
 Confirmation column: 75m, 0.45mm ID, 2.55 m Rtx[®]-502.2 (cat.# 10986)
 Conc.: 10ppb in 5mL of RO water
 Concentrator: Tekmar LSC3100 Purge and Trap
 Trap: Vocarb 3000
 Purge: 11 min. @ 40mL/min.
 Dry purge: 1 min. @ 40mL/min.
 Desorb preheat: 245 C
 Desorb: 250 C for 2 min.
 Bake: 260 C for 8 min.
 Interface: direct connection from concentrator to column
 Transfer line: 0.53mm ID Silcosteel[®] tubing (cat. #70045)
 Gas chromatograph: Finnigan 9001
 Carrier gas: helium @ ~10mL/min. constant pressure
 Adjust dichlorodifluoromethane to a retention time of 2.47 min. @ 40 C.
 2.04 min.
 Dead time:
 Oven temp.: 40 C (hold 2 min.) to 58 C @ 4 C/min. to 90 C @ 10 C/min. (hold 5 min.) to 220 C @ 40 C/min. (hold 5 min.)
 Detectors: Gold Tandem PID/Hall 2000 ELCD
 PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV base temp: 200 C.
 Hall 2000 ELCD: RxnGas 25mL/min., Rxn Temp. 940 C, propanol flow 470 L/min.

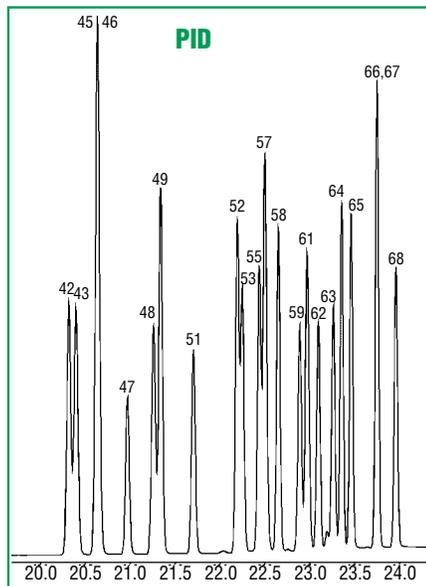
Standard	cat.#	Standard	cat.#
502.2 mix#1	30042	fluorobenzene	30030
624 cal mix #2	30021	1-chloro-2-fluorobenzene	30040
624 cal mix #3	30022	4-bromo-1-chlorobenzene	30230
MTBE	30402	bromochloromethane	30225
1,4-dichlorobutane	30227		

Figure 42A.

An Rtx®-VGC / Rtx®-502.2 column pair separates the expanded list of compounds in EPA Method 8021B in less than 30 minutes.

Rtx®-VGC

75m, 0.45mm ID, 2.55µm (cat.# 19409)



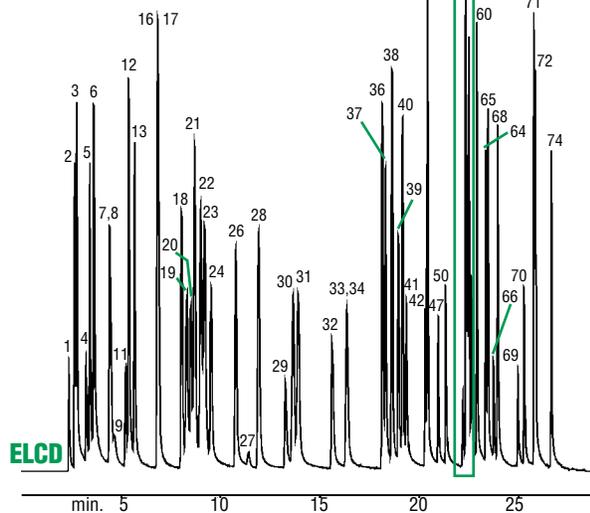
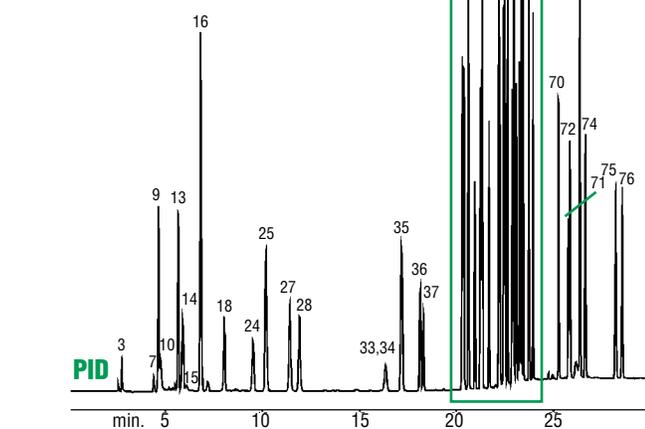
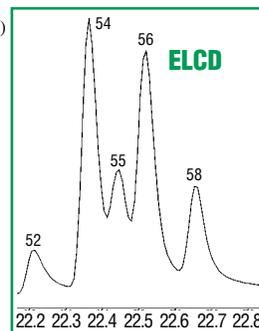
Primary column: 75m, 0.45mm ID, 2.55 µm Rtx®-VGC (cat.# 19409)
 Confirmation column: 75m, 0.45mm ID, 2.55 µm Rtx®-502.2 (cat.# 10986)
 Conc.: 10ppb in 5mL of RO Water
 Concentrator: Tekmar LSC-3000 Purge and Trap
 Trap: Vocarb 3000
 Purge: 11 min. @ 40 mL/min.
 Dry purge: 1 min. @ 40mL/min. (MCS bypassed using Silcosteel® tubing)
 Desorb preheat: 245 C, Flow 10mL/min.
 Desorb: 250 C for 2 min.
 Bake: 260 C for 8 min.
 Interface: direct using 0.32mm ID Siltek transfer line (cat. #10027)
 GC: Finnigan 9001
 Carrier gas: helium @ ~10mL/min. constant pressure

Oven temp.:

Detectors:

Adjust dichlorodifluoromethane to a retention time of 2.40 min. @ 45 C on the Rtx®-VGC column.
 45 C (hold 4 min.) to 70 C @ 2 C/min. to 210 C @ 20 C/min. (hold 10 min.)
 Gold Tandem PID/HALL 2000 ELCD
 PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV base temp. 200 C.
 Hall 2000 ELCD: RxnGas 25mL/min., RxnTemp.940 C, propanol flow 470 L/min.

Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL® 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728



- | | | | | |
|--|------------------------------------|---------------------------------------|--------------------------------|----------------------------------|
| 1. dichlorodifluoromethane | 17. 1,1-dichloroethane | 33. 2-chloroethyl vinyl ether | 49. styrene | 65. 1,4-dichlorobenzene |
| 2. chloromethane | 18. <i>cis</i> -1,2-dichloroethene | 34. <i>cis</i> -1,3-dichloropropene | 50. bromoform | 66. benzyl chloride |
| 3. vinyl chloride | 19. 2,2-dichloropropane | 35. toluene | 51. isopropylbenzene | 67. <i>n</i> -butylbenzene |
| 4. bromomethane | 20. bromochloromethane | 36. tetrachloroethene | 52. bromobenzene | 68. 1,2-dichlorobenzene |
| 5. chloroethane | 21. chloroform | 37. <i>trans</i> -1,3-dichloropropene | 53. <i>n</i> -propylbenzene | 69. 1,2-dibromo-3-chloropropane |
| 6. trichlorofluoromethane | 22. carbon tetrachloride | 38. 1,1,2-trichloroethane | 54. 1,1,2,2-tetrachloroethane | 70. 2-bromo-1-chlorobenzene (SS) |
| 7. 1,1-dichloroethene | 23. 1,1,1-trichloroethane | 39. dibromochloromethane | 55. 2-chlorotoluene | 71. hexachlorobutadiene |
| 8. Freon® 113 | 24. 1,1-dichloropropene | 40. 1,3-dichloropropane | 56. 1,2,3-trichloropropane | 72. 1,2,4-trichlorobenzene |
| 9. chloromethyl methyl ether | 25. benzene | 41. 1,2-dibromoethane | 57. 1,3,5-trimethylbenzene | 73. naphthalene |
| 10. iodomethane (40ppb) | 26. 1,2-dichloroethane | 42. chlorobenzene | 58. 4-chlorotoluene | 74. 1,2,3-trichlorobenzene |
| 11. allyl chloride | 27. fluorobenzene (SS) | 43. ethylbenzene | 59. <i>tert</i> -butylbenzene | 75. 2-methylnaphthalene (40ppb) |
| 12. methylene chloride | 28. tetrachloroethene | 44. 1,1,1,2-tetrachloroethane | 60. pentachloroethane | 76. 1-methylnaphthalene (40ppb) |
| 13. <i>trans</i> -1,2-dichloroethene | 29. dibromomethane | 45. <i>m</i> -xylene | 61. 1,2,4-trimethylbenzene | |
| 14. methyl <i>tert</i> -butyl ether | 30. 1,2-dichloropropane | 46. <i>p</i> -xylene | 62. <i>sec</i> -butylbenzene | |
| 15. <i>tert</i> -butyl alcohol (40ppb) | 31. bromodichloromethane | 47. 1-chloro-2-fluorobenzene (SS) | 63. <i>p</i> -isopropyltoluene | |
| 16. chloroprene | 32. 1-bromo-2-chloroethane (SS) | 48. <i>o</i> -xylene | 64. 1,3-dichlorobenzene | |

GC_EV00421

The chromatograms in Figures 42A & B incorporate a broader range of analytes, many of which are listed in EPA Method 8021B, along with other requested compounds, such as 1-methylnaphthalene and 2-methylnaphthalene. Of these 72 target compounds, those that coelute on the Rtx®-VGC column are resolved by the Rtx®-502.2 column. Even with the addition of the semivolatile methylnaphthalenes, the analysis time is less than 30 minutes.

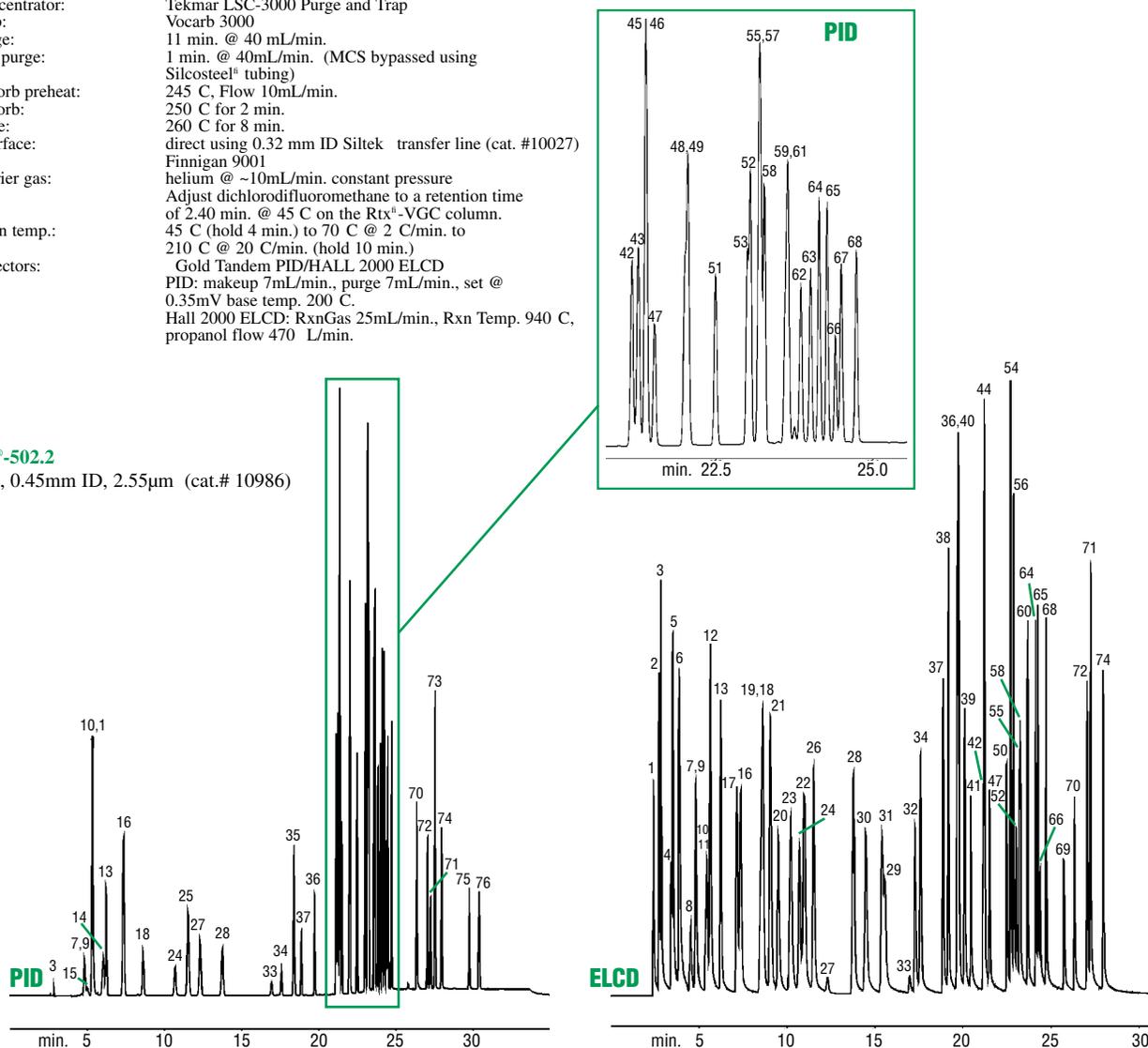
Figure 42B.

An Rtx[®]-VGC / Rtx[®]-502.2 column pair separates the expanded list of compounds in EPA Method 8021B in less than 30 minutes.

Primary column: 75m, 0.45mm ID, 2.55 m Rtx[®]-VGC (cat.# 19409)
 Confirmation column: 75m, 0.45mm ID, 2.55 m Rtx[®]-502.2 (cat.# 10986)
 Conc.: 10 ppb in 5 mL of RO Water
 Concentrator: Tekmar LSC-3000 Purge and Trap
 Trap: Vocarb 3000
 Purge: 11 min. @ 40 mL/min.
 Dry purge: 1 min. @ 40mL/min. (MCS bypassed using Silcosteel[®] tubing)
 Desorb preheat: 245 C, Flow 10mL/min.
 Desorb: 250 C for 2 min.
 Bake: 260 C for 8 min.
 Interface: direct using 0.32 mm ID Siltek transfer line (cat. #10027)
 GC: Finnigan 9001
 Carrier gas: helium @ ~10mL/min. constant pressure
 Adjust dichlorodifluoromethane to a retention time of 2.40 min. @ 45 C on the Rtx[®]-VGC column.
 Oven temp.: 45 C (hold 4 min.) to 70 C @ 2 C/min. to 210 C @ 20 C/min. (hold 10 min.)
 Detectors: Gold Tandem PID/HALL 2000 ELCD
 PID: makeup 7mL/min., purge 7mL/min., set @ 0.35mV base temp. 200 C.
 Hall 2000 ELCD: RxnGas 25mL/min., Rxn Temp. 940 C, propanol flow 470 L/min.

Rtx[®]-502.2

75m, 0.45mm ID, 2.55µm (cat.# 10986)



- | | | | | |
|--|------------------------------------|---------------------------------------|--------------------------------|----------------------------------|
| 1. dichlorodifluoromethane | 17. 1,1-dichloroethane | 33. 2-chloroethyl vinyl ether | 49. styrene | 65. 1,4-dichlorobenzene |
| 2. chloromethane | 18. <i>cis</i> -1,2-dichloroethene | 34. <i>cis</i> -1,3-dichloropropene | 50. bromoform | 66. benzyl chloride |
| 3. vinyl chloride | 19. 2,2-dichloropropane | 35. toluene | 51. isopropylbenzene | 67. <i>n</i> -butylbenzene |
| 4. bromomethane | 20. bromochloromethane | 36. tetrachloroethene | 52. bromobenzene | 68. 1,2-dichlorobenzene |
| 5. chloroethane | 21. chloroform | 37. <i>trans</i> -1,3-dichloropropene | 53. <i>n</i> -propylbenzene | 69. 1,2-dibromo-3-chloropropane |
| 6. trichlorofluoromethane | 22. carbon tetrachloride | 38. 1,1,2-trichloroethane | 54. 1,1,2,2-tetrachloroethane | 70. 2-bromo-1-chlorobenzene (SS) |
| 7. 1,1-dichloroethene | 23. 1,1,1-trichloroethane | 39. dibromochloromethane | 55. 2-chlorotoluene | 71. hexachlorobutadiene |
| 8. Freon [®] 113 | 24. 1,1-dichloropropene | 40. 1,3-dichloropropane | 56. 1,2,3-trichloropropane | 72. 1,2,4-trichlorobenzene |
| 9. chloromethyl methyl ether | 25. benzene | 41. 1,2-dibromoethane | 57. 1,3,5-trimethylbenzene | 73. naphthalene |
| 10. iodomethane (40ppb) | 26. 1,2-dichloroethane | 42. chlorobenzene | 58. 4-chlorotoluene | 74. 1,2,3-trichlorobenzene |
| 11. allyl chloride | 27. fluoroethane (SS) | 43. ethylbenzene | 59. <i>tert</i> -butylbenzene | 75. 2-methylnaphthalene (40ppb) |
| 12. methylene chloride | 28. trichloroethene | 44. 1,1,1,2-tetrachloroethane | 60. pentachloroethane | 76. 1-methylnaphthalene (40ppb) |
| 13. <i>trans</i> -1,2-dichloroethene | 29. dibromomethane | 45. <i>m</i> -xylene | 61. 1,2,4-trimethylbenzene | |
| 14. methyl <i>tert</i> -butyl ether | 30. 1,2-dichloropropane | 46. <i>p</i> -xylene | 62. <i>sec</i> -butylbenzene | |
| 15. <i>tert</i> -butyl alcohol (40ppb) | 31. bromodichloromethane | 47. 1-chloro-2-fluorobenzene (SS) | 63. <i>p</i> -isopropyltoluene | |
| 16. chloroprene | 32. 1-bromo-2-chloroethane (SS) | 48. <i>o</i> -xylene | 64. 1,3-dichlorobenzene | |

GC_EV00422

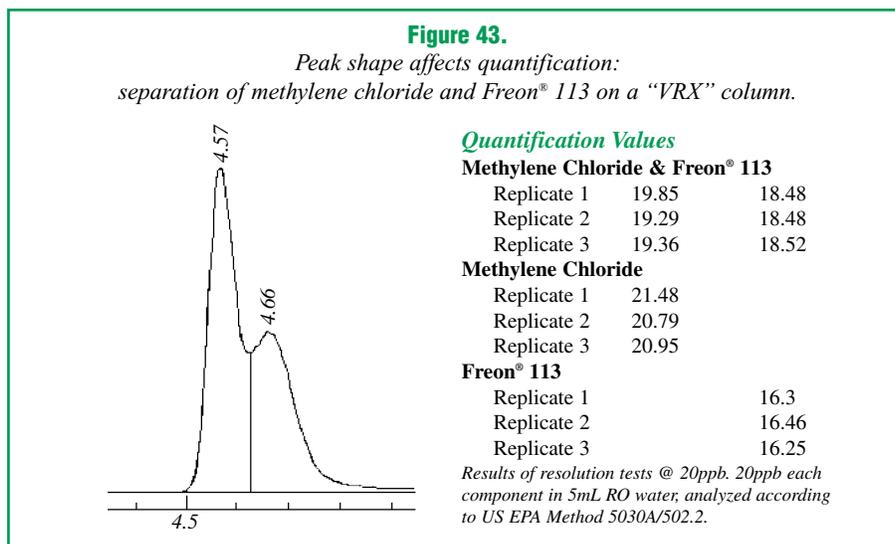
Acknowledgement: Finnigan 9001 GC, µGold Tandem Photoionization/HALL[®] 2000 Electrolytic Conductivity Detector provided courtesy of Thermo Finnigan GC & GC/MS Division, 2215 Grand Avenue Pkwy, Austin, Texas 78728

Importance of Resolution in GC Analysis

Figure 43 shows the effects of resolution on quantification for methylene chloride and Freon® 113 (1,1,2-trichloro-1,2,2-trifluoroethane) when separated on an Rtx®-VRX column. The first three replicates are check standards analyzed on a six-point curve. These values are taken from integrating the areas for the two closely eluting peaks. Based on peak height (the methylene chloride peak is about twice the height of the Freon®113 peak), the contribution of the first peak, methylene chloride, to the Freon®113 peak is more than twice the contribution of Freon® 113 to the methylene chloride peak. Both compounds are quantified from the ELCD, creating another quantification issue with tailing peaks: the tail of the methylene chloride peak contributes to the area of the Freon® 113 peak, thereby increasing the quantification error.

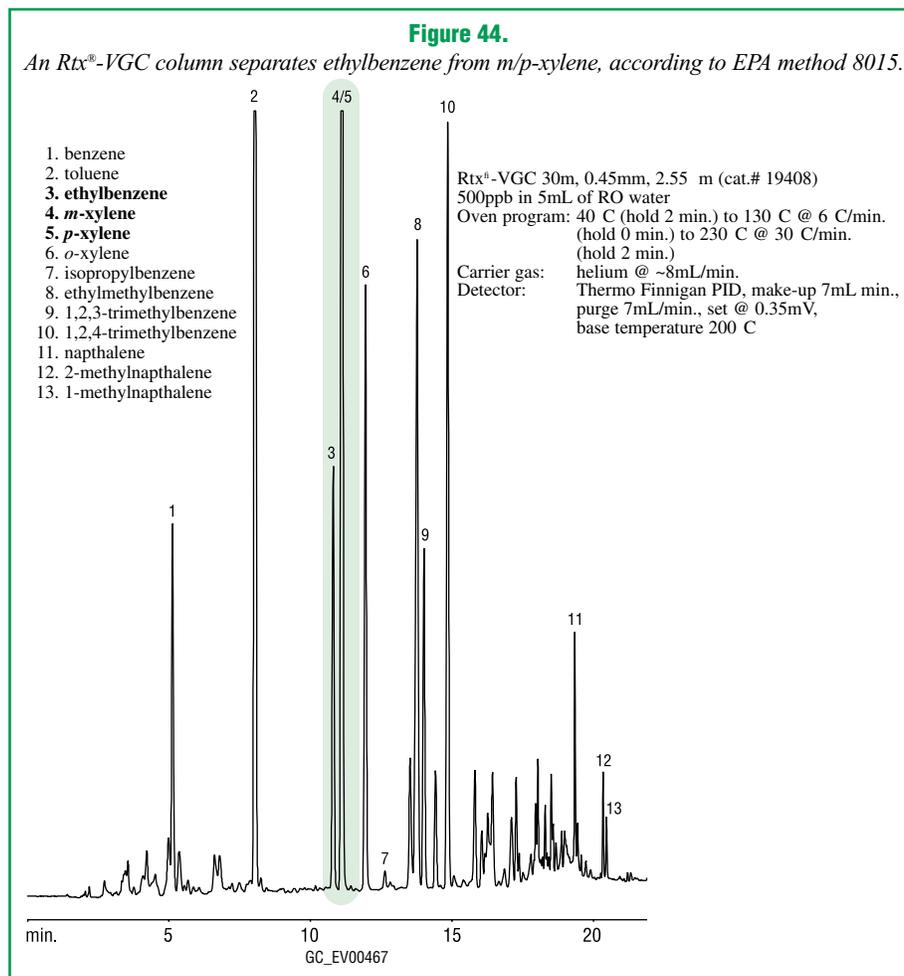
Next, we analyzed methylene chloride and Freon® 113 separately, to determine if there would be quantification differences off the six-point curve. With the addition of the peak tail, the peak area for methylene chloride increased slightly, and the peak area for Freon® 113 decreased. Without the contribution from methylene chloride, Freon® 113 exhibited a noticeably lower quantification value of 16ppb.

This experiment illustrates the importance of resolution for accurate quantification of analytes in environmental samples taken from the field. Obviously, no column can provide baseline separation of all Method 502.2/8021 compounds. The key to accurate quantification is to recognize which compounds are most commonly present in your samples and choose a column that best resolves these analytes. As shown in the example described above, both the Rtx®-VRX column and the Rtx®-VGC column have difficulty resolving methylene chloride from Freon® 113.

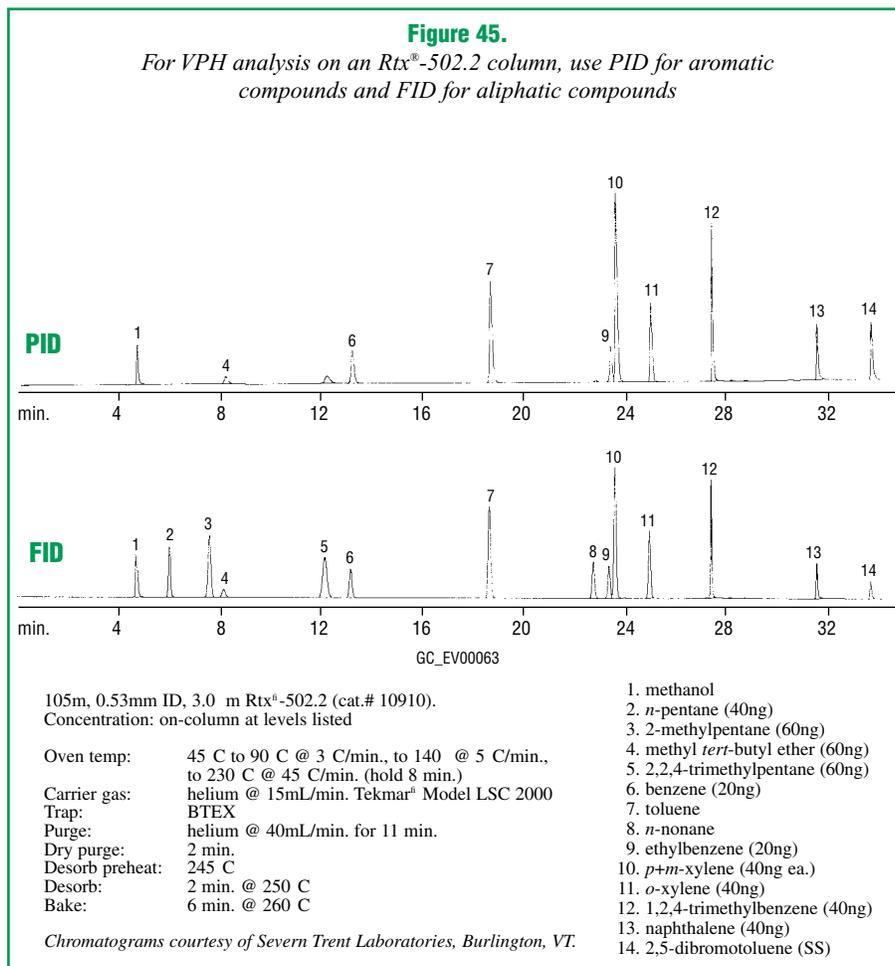


**Purge and Trap Applications Using PID/FID in Tandem:
EPA Method 602 and Others; State Gasoline Methods**

EPA Methods 602, 8015, 8020, and 8260 describe analyses of gasoline and oxygenates. Most environmental laboratories have relied on one of these methods to report gasoline and oxygenate concentrations in difficult sample matrixes. Success with these methods is based on the ability of the capillary column to resolve close-eluting pairs. An analysis according to Method 8015, for example, involves separating ethylbenzene from *m/p*-xylene (Figure 44).



Many state methods, such as the Massachusetts Volatile Petroleum Hydrocarbon (VPH) method, require resolution of the oxygenates and the early eluting alkanes, such as 2-methylpentane and 3-methylpentane (Figure 45). The most common column used for GRO analysis is a 30m, 0.53mm ID column with a 1.5 μ m df film of 5% diphenylpolysiloxane phase, such as Rtx[®]-5. These columns resolve the difficult compounds chlorobenzene and ethylbenzene from the xylenes. An Rtx[®]-502.2 column performs this separation equally well, but a longer column is needed.



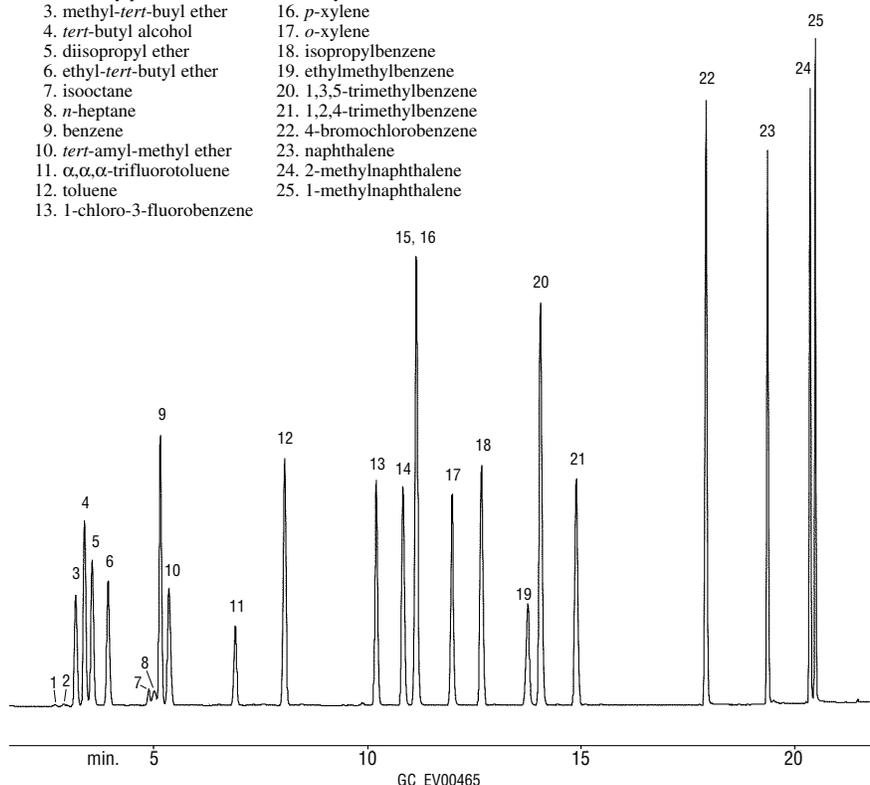
Gasoline Analysis with Oxygenates and Alcohols

Success of gasoline range organics (GRO) analyses are based on the ability of the analytical column to resolve oxygenates from the alkanes, alkenes, and, to a lesser extent, alkynes. To minimize false positive results for methyl-*tert*-butyl ether (MTBE) it is important to separate this analyte from 2-methylpentane and 3-methylpentane. Another potential interference is *tert*-butyl alcohol (TBA). Both MTBE and TBA elicit response on a PID, and they share ions used in MS detection, so they must be resolved regardless of which detector is used. Figure 46 is an example of GRO target compounds analyzed using PID detection, with the oxygen-containing gasoline additives resolved from the other analytes.

Figure 46.

An Rtx®-VGC column resolves oxygen-containing gasoline derivatives from other GRO.

- | | |
|--|----------------------------|
| 1. 2-methylpentane | 14. ethylbenzene |
| 2. 3-methylpentane | 15. <i>m</i> -xylene |
| 3. methyl- <i>tert</i> -butyl ether | 16. <i>p</i> -xylene |
| 4. <i>tert</i> -butyl alcohol | 17. <i>o</i> -xylene |
| 5. diisopropyl ether | 18. isopropylbenzene |
| 6. ethyl- <i>tert</i> -butyl ether | 19. ethylmethylbenzene |
| 7. isooctane | 20. 1,3,5-trimethylbenzene |
| 8. <i>n</i> -heptane | 21. 1,2,4-trimethylbenzene |
| 9. benzene | 22. 4-bromochlorobenzene |
| 10. <i>tert</i> -amyl-methyl ether | 23. naphthalene |
| 11. α,α,α -trifluorotoluene | 24. 2-methylnaphthalene |
| 12. toluene | 25. 1-methylnaphthalene |
| 13. 1-chloro-3-fluorobenzene | |



30m, 0.45mm, 2.55 m Rtx®-VGC (cat.# 19408)

each component 100ppb in 5mL of RO water, except *tert*-butyl alcohol 5000ppb; 2/1-methynaphthalene 150ppb; ethylmethylbenzene 50ppb.

Concentrator: Tekmar LSC-3100 Purge and Trap

Trap: Vocarb 3000

Purge: 11 min. @ 40mL/min. @ 35 C

Dry purge: 1 min. (MCS bypassed)

GC: Finnigan 9001

Oven temp.: 40 C (hold 2 min.) to 130 C @ 6 C/min. (hold 0 min.) to 230 C @ 30 C/min. (hold 2 min.).

Carrier gas: helium @ ~8mL/min.

Detector: Thermo Finnigan PID, make-up 7mL min., purge 7mL/min., set @ 0.35mV, base temperature 200 C.

Advances in Sample Throughput

The demand for increased productivity in volatiles analysis by GC/MS has resulted in the creation of automated water and soil autosamplers that reduce the amount of manual sample preparation required. Autosamplers enable environmental laboratories to run purge and trap systems around the clock. Even though prices for analyses of samples by Methods 8260 and 524.2 have stabilized, laboratories still push for faster turn-around-time, to get a better return on capital equipment investments. This has resulted in a need for columns that can drastically reduce separation time and for instruments that can accommodate short cycle times. Currently, the limiting factor in VOA is the purge and trap cycle time, because it includes an 11-minute purge time followed by a 6-12 minute bake-out time. A modern GC, on the other hand, can acquire a sample in 10 minutes or less. To overcome the time limitations of the purge and trap, connect two purge and traps, each with its own autosampler unit, to one GC/MS operating system. Use the dual-concentrator configuration to synchronize the steps so while the first system is desorbing the sample and starting the GC/MS analysis, the second system is completing the bake cycle and starting to purge the next sample to be desorbed onto the column. The Duet® system, designed and sold by Tekmar-Dohrmann, allows communication between the two concentrators for configuration to one GC/MS. The Duet® interface gates the signals between the concentrators to prevent a faster system from catching up to a slightly slower one and allowing a double injection. Calibration curves and quality control samples (QC, MS, MSD) must be run for each concentrator.

A tracer compound must be added to one of the concentrators, to eliminate any potential question as to which purge and trap system purged/desorbed the sample. With this system it is possible to run 80 samples in 24 hours, thereby increasing output from a single GC/MS instrument. Figure 47 (page 51) shows an analysis on an Rtx®-VMS column according to US EPA Method 8260B, using the correct internal standards and surrogates. For more information see the literature cited.¹⁴

Applications Using GC/MS Detection Systems

EPA Methods 8260, 524.2, 624, 8240 and OLM 04.2

Method 8260: Client target lists may remain the same as the Method 8240 compound list, but the calibration criteria and low detection limits set by Method 8260 are enforced (Figure 48, page 52).¹⁵ Chromatograms for the 8240 compound list can be produced from different GC oven conditions, different compound concentrations, and altered MS scan windows. Alcohols analyses require scanning below 35amu because many of the fragments used to identify the spectra for these compounds are between 25 and 35amu. A good example is 2-chloroethanol – this target analyte purges poorly and does not respond well by MS detection. The best way to increase sensitivity by MS detection is by changing the scan rate to include ion 31, the base peak. This also improves the ability of the software and the analyst to identify alcohols because it gives more spectral data. The disadvantage of this approach is an increase in noise, producing an overall decrease in sensitivity for all compounds. In Figure 48, the second chromatogram shows an increase in baseline noise as a result of the lower scan window. A comparison of peak 38 (2-chloroethanol) on the two chromatograms clearly shows a significantly higher response on the second chromatogram, despite a lower concentration.

Method 8260 contains many mid-range volatile compounds that are the most common non-petroleum contaminants in the environment. Unfortunately, these compounds tend to exhibit broad peak shapes due to poor sample transfer from the purge and trap, making them difficult to resolve. Rtx®-VMS columns were designed using computer-assisted stationary phase design (CASPD) software to improve solubility of these analytes in the stationary phase, and thus provide greater separation for these compounds.¹⁶ This tuned selectivity ensures separation of tetrahydrofuran/2-butanone, carbon tetrachloride/1,1,1-trichloroethane, and methyl acrylate/propionitrile. Although these compounds share common ions and have very similar spectra, they are resolved by retention time difference on an Rtx®-VMS column (Figure 49, page 53). Analytes that share ions and coelute on an Rtx®-624 column, but are resolved by an Rtx®-VMS column, include: ether/ethanol, vinyl acetate/ethyl-tert-butyl ether, and tert-butyl alcohol/methyl-tert-butyl ether. Several of these compounds require a lower initial oven temperature (35°C), which is not shown in these applications.

Higher-boiling volatile compounds, typically branched or substituted aromatic compounds, provide analytical challenges of their own. Isomers of the branched aromatic compounds share the same parent ions and cannot be identified accurately by MS alone. The Rtx®-VMS phase also was modeled for maximum separation of the substituted aromatic isomers, such as 2- and 4-chlorotoluene. The comparison in Table VII shows isomer resolution on four other stationary phases, modeled under the same conditions, compared to resolution on the Rtx®-VMS phase. The tuned selectivity of the Rtx®-VMS phase allows a rapid final GC oven ramp rate of 40°C/min., or faster, thereby promoting fast analysis times. Also, initial temperatures of up to 60°C are possible (Figure 49, page 53). This higher initial temperature provides the required separation and allows faster oven cycle times, although some laboratories prefer to start at 50°C, to better enhance the resolution of chloromethane from vinyl chloride (peaks 2 and 3).

Figure 47 (page 51) shows an analysis of the Method 8260B compound list, using an Rtx®-VMS column (20m, 0.18mm ID, 1.0µm film) without cryogenic cooling. Resolution is greatly enhanced, due to the higher efficiency of the 0.18mm ID column. The desorb flow rate is set at 40mL/min. for 1 minute. Many laboratories desorb under these conditions for 2 minutes, but the Rtx®-VMS column makes this unnecessary, because the higher flow rate will desorb the volatiles from the trap in less than a minute.

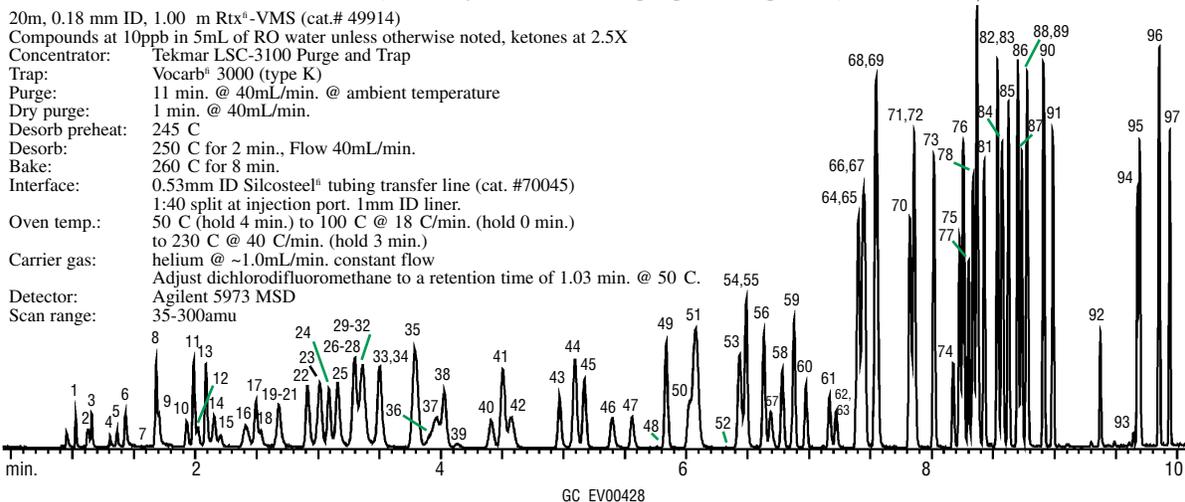
Table VII.

An Rtx®-VMS column best separates 2- and 4-chlorotoluene.

Retention Time (min.)	Rtx®-VMS	Rtx®-624	Rtx®-502.2	Rtx®-VRX	Rtx®-1
2-Chlorotoluene	8.35	8.63	8.80	8.49	8.38
4-Chlorotoluene	8.44	8.69	8.84	8.53	8.41
RT diff.	0.09	0.06	0.04	0.04	0.03

Figure 47.

Volatile organics by US EPA Method 8260B on an Rtx®-VMS column; conditions optimized for fast Method 8260 analysis.
(Suitable for use with dual purge and trap units.)



1. dichlorodifluoromethane	26. ethyl acetate	51. toluene	76. <i>n</i> -propylbenzene
2. chloromethane	27. carbon tetrachloride	52. pyridine (250ppb)	77. 1,1,2,2-tetrachloroethane
3. vinyl chloride	28. methyl acrylate	53. tetrachloroethene	78. 2-chlorotoluene
4. bromomethane	29. propargyl alcohol (500ppb)	54. 4-methyl-2-pentanone	79. 1,3,5-trimethylbenzene
5. chloroethane	30. dibromofluoromethane (SMC)	55. <i>trans</i> -1,3-dichloropropene	80. 1,2,3-trichloropropane
6. trichlorofluoromethane	31. tetrahydrofuran	56. 1,1,2-trichloroethane	81. 4-chlorotoluene
7. ethanol (2500ppb)	32. 1,1,1-trichloroethane	57. ethyl methacrylate	82. <i>tert</i> -butylbenzene
8. 1,1-dichloroethene	33. 2-butanone	58. dibromochloromethane	83. pentachloroethane
9. carbon disulfide (40ppb)	34. 1,1-dichloropropene	59. 1,3-dichloropropane	84. 1,2,4-trimethylbenzene
10. allyl chloride	35. benzene	60. 1,2-dibromoethane	85. <i>sec</i> -butylbenzene
11. methylene chloride	36. pentafluorobenzene (IS)	61. <i>n</i> -butyl acetate	86. <i>p</i> -isopropyltoluene
12. acetone	37. <i>tert</i> -amyl-methyl ether	62. 2-hexanone	87. 1,3-dichlorobenzene
13. <i>trans</i> -1,2-dichloroethene	38. 1,2-dichloroethane	63. 2-picoline (250ppb)	88. 1,4-dichlorobenzene-d4 (IS)
14. methyl <i>tert</i> -butyl ether	39. isobutyl alcohol (500ppb)	64. chlorobenzene-d5 (IS)	89. 1,4-dichlorobenzene
15. <i>tert</i> -butyl alcohol (100ppb)	40. isopropyl acetate	65. chlorobenzene	90. <i>n</i> -butylbenzene
16. diisopropyl ether	41. trichloroethene	66. ethylbenzene	91. 1,2-dichlorobenzene
17. 1,1-dichloroethane	42. 1,4-difluorobenzene (SMC)	67. 1,1,1,2-tetrachloroethane	92. 1,2-dibromo-3-chloropropane
18. acrylonitrile	43. dibromomethane	68. <i>m</i> -xylene	93. nitrobenzene (250ppb)
19. vinyl acetate	44. 1,2-dichloropropane	69. <i>p</i> -xylene	94. hexachlorobutadiene
20. allyl alcohol (250ppb)	45. bromodichloromethane	70. <i>o</i> -xylene	95. 1,2,4-trichlorobenzene
21. ethyl- <i>tert</i> -butyl ether	46. methyl methacrylate	71. styrene	96. naphthalene
22. <i>cis</i> -1,2-dichloroethene	47. <i>n</i> -propyl acetate	72. bromoform	97. 1,2,3-trichlorobenzene
23. 2,2-dichloropropane	48. 2-chloroethanol (2500ppb)	73. isopropylbenzene	
24. bromochloromethane	49. <i>cis</i> -1,3-dichloropropene	74. 4-bromo-1-fluorobenzene (SMC)	
25. chloroform	50. toluene-d8(SMC)	75. bromobenzene	

EPA-recommended SMC/IS used.

One of the most important factors in optimizing conditions for using the narrow-bore column is adjusting the flow. Most MS systems are designed for optimum sensitivity at 1mL/min.; flow rates higher or lower will greatly compromise the method detection limit (MDL). For Figure 47 (page 51) the retention time for the first gas, dichlorodifluoromethane, is 1.03 minutes at 50°C, which dictates a column flow of 1mL/min. Figure 49 (page 53) also lists specific information for setting the correct flow rate.

Electronic pressure control (EPC) makes it possible to maintain a constant flow over the course of the oven temperature program, which can cut several minutes from the analysis time, compared to a system set up for constant pressure. When setting up a system for constant pressure, always adjust the flow at the initial oven temperature to be approximately 1mL/min. It is true that, under constant pressure, higher flows at the beginning of the analysis will equate to normal flows (closer to 1mL/min.) as the temperature, and carrier gas viscosity, increases, but maximum sensitivity is needed for the more volatile analytes because they exhibit broader peaks. Also, higher flows at the start of the analysis, while methanol and water are entering the MS, could cause excessive source pressure and automatically shut the filament off. Figure 49 (page 53) shows an analysis on a 60m, 0.25mm ID, 1.4µm film Rtx®-VMS column, using an initial temperature of 60°C. The injection port is set for a 1:20 split and constant flow is adjusted to 1.3mL/min. Again, the best way to set the flow for these columns is to use the retention time for dichlorodifluoromethane or for an unretained compound. Carbon dioxide is a good choice for an unretained compound.

14. A.L. Hilling and G. Smith, *Environmental Testing & Analysis*, 10(3),15-19, 2001.

15. *Method 8260B Volatile Organic Compounds in Water by Gas Chromatography/Mass Spectrometry*, Revision 2.0, 1996, SW-846, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268.

16. F.L. Dorman, P.D. Schettler, C.M. English and D.V. Patwardhan. "Predicting Gas Chromatographic Separation and Stationary-Phase Selectivity Using Computer Modeling." *Anal. Chem.* 2002, 74, 2133-2138.

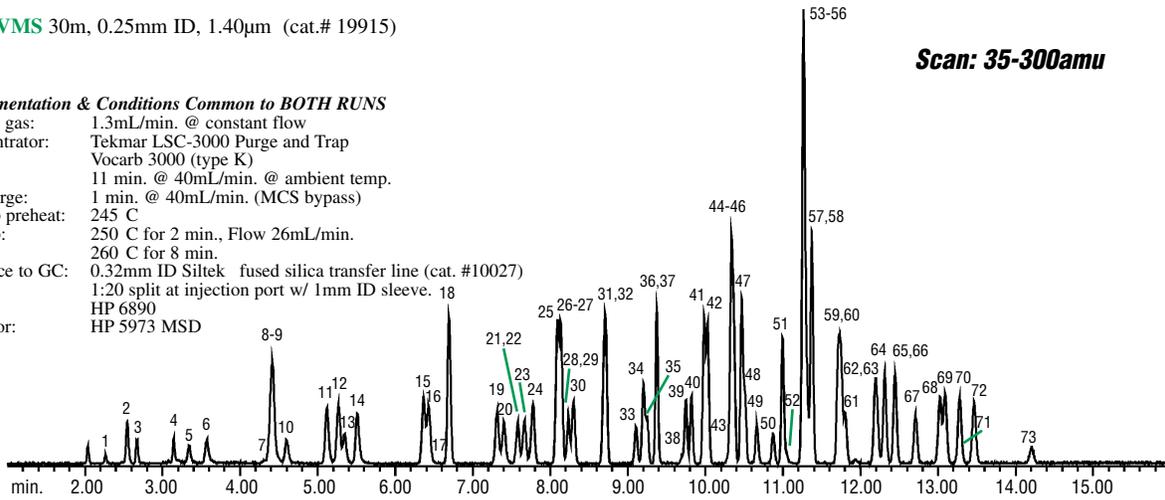
Figure 48.

EPA Method 8240 analysis using an Rtx[®]-VMS column. Alcohols require scans below 35amu.Rtx[®]-VMS 30m, 0.25mm ID, 1.40µm (cat.# 19915)

Scan: 35-300amu

Instrumentation & Conditions Common to BOTH RUNS

Carrier gas: 1.3mL/min. @ constant flow
 Concentrator: Tekmar LSC-3000 Purge and Trap
 Trap: Vocab 3000 (type K)
 Purge: 11 min. @ 40mL/min. @ ambient temp.
 Dry purge: 1 min. @ 40mL/min. (MCS bypass)
 Desorb preheat: 245 C
 Desorb: 250 C for 2 min., Flow 26mL/min.
 Bake: 260 C for 8 min.
 Interface to GC: 0.32mm ID Siltek fused silica transfer line (cat. #10027)
 1:20 split at injection port w/ 1mm ID sleeve. 18
 GC: HP 6890
 Detector: HP 5973 MSD

**Top chromatogram:**

Oven temp.: 40 C (hold 4 min.) to 90 C @ 16 C/min. (no hold)
 to 210 C @ 32 C/min. (hold 5 min.)
 Adjust dichlorodifluoromethane to a retention time of
 2.27 min. @ 40 C.

MS Scan Range: 35-300amu

Compound Concentrations, by mix: (in 5mL of RO water)

Compounds at 100ppb (cat.# 30213, 30004, 30006, 30011, 30042)

Alcohols at 1ppm (cat.# 30214); 2-chloroethanol at 10ppm.

vinyl acetate at 500ppb (cat.#30216)

8240 Nitrile Mix at 200ppb (cat.# 30215)

8240 Mix 1A at 300ppb (cat.# 30217)

8240 Mix 2A at 500ppb (cat.# 30218)

Bottom chromatogram:

Oven temp.: 45 C (hold 4 min.) to 110 C @ 19 C/min. (hold 5 min.) to
 220 C @ 32 C/min. (hold 5 min.)
 Adjust dichlorodifluoromethane to 2.23 min. @ 45 C.

MS Scan Range: 29-260amu, for 2-chloroethanol response

Compound Concentrations, by mix: (in 5mL of RO water)

Compounds at 100ppb (cat.# 30213, 30004, 30006, 30011, 30042)

Alcohols at 1ppm (cat.# 30214) (see MS scan)

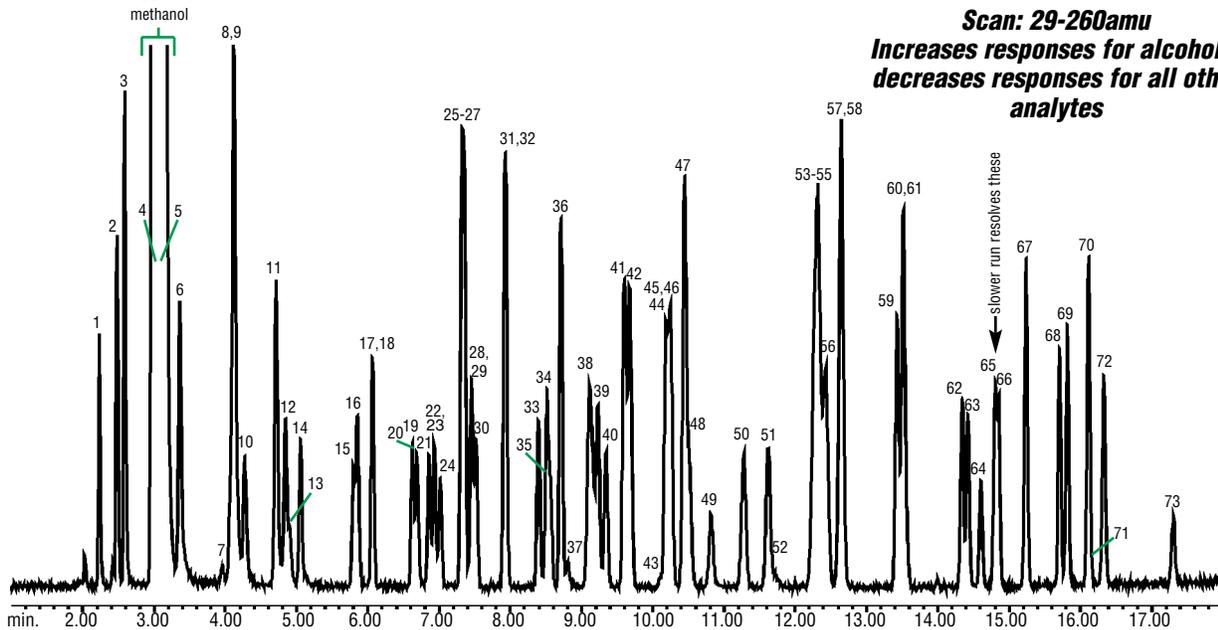
vinyl acetate at 100ppb (cat.# 30216)

8240 Nitrile Mix at 400ppb (cat.# 30215)

8240 Mix 1A at 300ppb (cat.# 30217)

8240 Mix 2A at 500ppb (cat.# 30218)

Scan: 29-260amu
Increases responses for alcohols,
decreases responses for all other
analytes



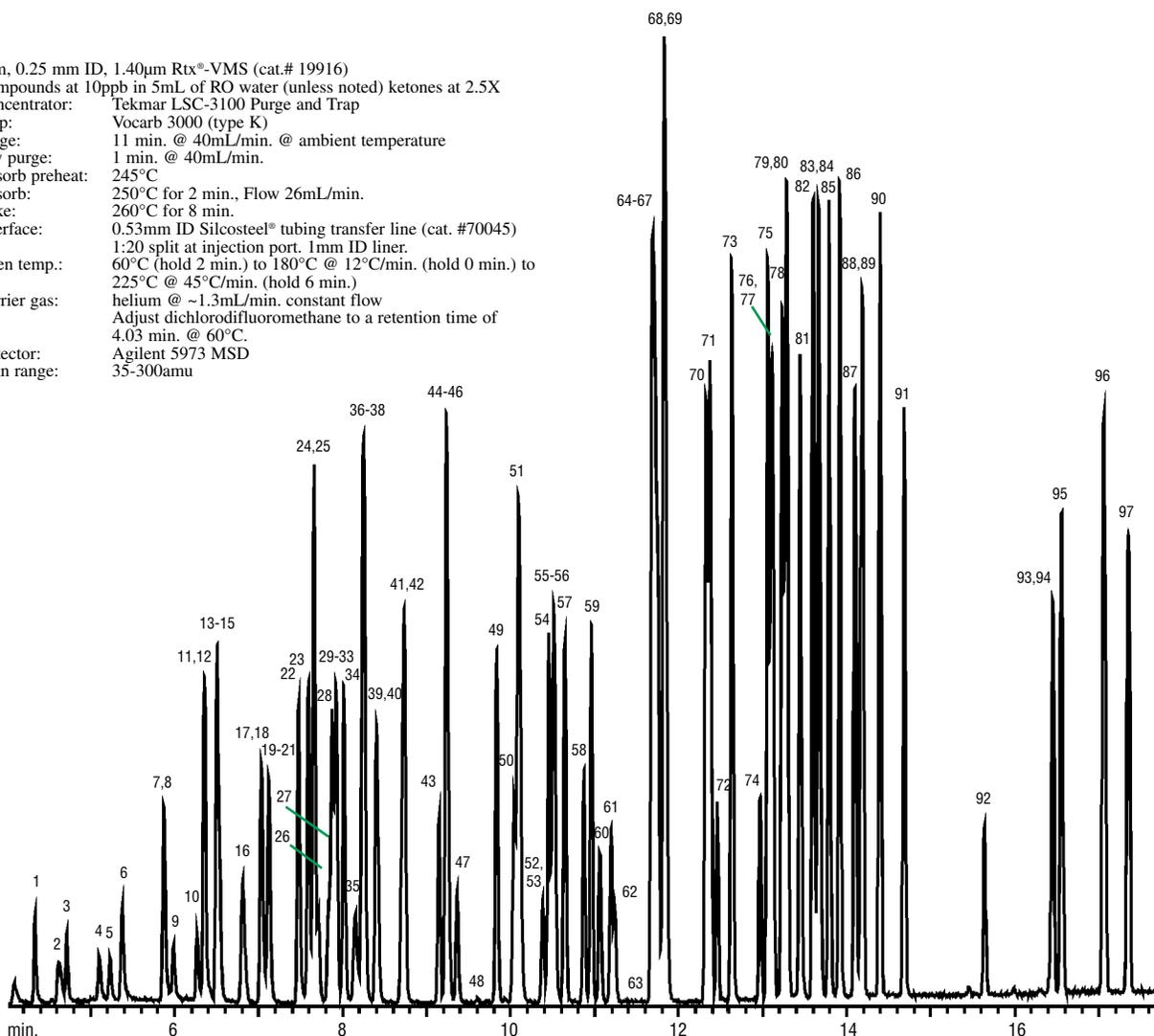
1. dichlorodifluoromethane	14. <i>trans</i> -1,2-dichloroethene	27. methacrylonitrile	40. <i>cis</i> -1,3-dichloropropene	53. chlorobenzene-D5	66. <i>trans</i> -1,4-dichloro-2-butene
2. chloromethane	15. 1,1-dichloroethane	28. 1,2-dichloroethane-d4	41. toluene-d8	54. ethylbenzene	67. pentachloroethane
3. vinyl chloride	16. acrylonitrile	29. isobutyl alcohol	42. toluene	55. chlorobenzene	68. 1,3-dichlorobenzene
4. bromomethane	17. allyl alcohol	30. 1,2-dichloroethane	43. pyridine	56. 1,1,1,2-tetrachloroethane	69. 1,4-dichlorobenzene
5. chloroethane	18. vinyl acetate	31. trichloroethene	44. 4-methyl-2-pentanone	57. <i>m</i> -xylene	70. benzyl chloride
6. trichlorofluoromethane	19. bromochloromethane	32. 1,4-difluorobenzene	45. tetrachloroethene	58. <i>p</i> -xylene	71. malononitrile
7. ethanol	20. chloroform	33. dibromomethane	46. <i>trans</i> -1,3-dichloropropene	59. <i>o</i> -xylene	72. 1,2-dichlorobenzene
8. 1,1-dichloroethene	21. carbon tetrachloride	34. 1,2-dichloropropane	47. ethyl methacrylate	60. styrene	73. 1,2-dibromo-3-chloropropane
9. carbon disulfide	22. propargyl alcohol	35. bromodichloromethane	48. 1,1,2-trichloroethane	61. bromoform	
10. iodomethane	23. 1,1,1-trichloroethane	36. methyl methacrylate	49. dibromochloromethane	62. 4-bromo-1-fluorobenzene	
11. allyl chloride	24. 2-butanone	37. 1,4-dioxane	50. 1,2-dibromoethane	63. <i>cis</i> -1,4-dichloro-2-butene	
12. methylene chloride	25. benzene	38. 2-chloroethanol	51. 2-hexanone	64. 1,1,2,2-tetrachloroethane	
13. acetone	26. propionitrile	39. 2-chloroethyl vinyl ether	52. 2-picoline	65. 1,2,3-trichloropropane	

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Figure 49.

An Rtx®-VMS column allows an initial temperature of 60°C, for fast oven cycle times by EPA Method 8260.

60m, 0.25 mm ID, 1.40µm Rtx®-VMS (cat.# 19916)
 Compounds at 10ppb in 5mL of RO water (unless noted) ketones at 2.5X
 Concentrator: Tekmar LSC-3100 Purge and Trap
 Trap: Vocarb 3000 (type K)
 Purge: 11 min. @ 40mL/min. @ ambient temperature
 Dry purge: 1 min. @ 40mL/min.
 Desorb preheat: 245°C
 Desorb: 250°C for 2 min., Flow 26mL/min.
 Bake: 260°C for 8 min.
 Interface: 0.53mm ID Silcosteel® tubing transfer line (cat. #70045)
 1:20 split at injection port. 1mm ID liner.
 Oven temp.: 60°C (hold 2 min.) to 180°C @ 12°C/min. (hold 0 min.) to
 225°C @ 45°C/min. (hold 6 min.)
 Carrier gas: helium @ ~1.3mL/min. constant flow
 Adjust dichlorodifluoromethane to a retention time of
 4.03 min. @ 60°C.
 Detector: Agilent 5973 MSD
 Scan range: 35-300amu



1. dichlorodifluoromethane	21. ethyl- <i>tert</i> -butyl ether*	41. 1,4-difluorobenzene (SMC)	61. 1,2-dibromoethane	81. 4-chlorotoluene
2. chloromethane	22. <i>cis</i> -1,2-dichloroethene	42. trichloroethene	62. 2-hexanone	82. <i>tert</i> -butylbenzene
3. vinyl chloride	23. 2,2-dichloropropane	43. dibromomethane	63. 2-picoline (250ppb)	83. 1,2,4-trimethylbenzene
4. bromomethane	24. bromochloromethane	44. bromodichloromethane	64. ethylbenzene	84. pentachloroethane
5. chloroethane	25. chloroform	45. 1,2-dichloropropane	65. chlorobenzene-D5 (IS)	85. <i>sec</i> -butylbenzene
6. trichlorofluoromethane	26. ethyl acetate	46. methyl methacrylate	66. chlorobenzene	86. <i>p</i> -isopropyltoluene
7. ethanol (2500ppb)	27. methyl acrylate	47. <i>n</i> -propyl acetate	67. 1,1,1,2-tetrachloroethane	87. 1,3-dichlorobenzene
8. 1,1-dichloroethene	28. propargyl alcohol (500ppb)	48. 2-chloroethanol (2500ppb)	68. <i>m</i> -xylene	88. 1,4-dichlorobenzene-d4 (IS)
9. carbon disulfide (40ppb)	29. dibromofluoromethane (SMC)	49. <i>cis</i> -1,3-dichloropropene	69. <i>p</i> -xylene	89. 1,4-dichlorobenzene
10. allyl chloride	30. tetrahydrofuran	50. toluene-d8 (SMC)	70. <i>o</i> -xylene	90. <i>n</i> -butylbenzene
11. methylene chloride	31. carbon tetrachloride	51. toluene	71. styrene	91. 1,2-dichlorobenzene
12. acetone	32. 2-butanone	52. 4-methyl-2-pentanone	72. bromoform	92. 1,2-dibromo-3-chloropropane
13. <i>trans</i> -1,2-dichloroethene	33. 1,1,1-trichloroethane	53. pyridine (250ppb)	73. isopropylbenzene	93. nitrobenzene (250ppb)
14. <i>tert</i> -butyl alcohol (100ppb)	34. 1,1-dichloropropene	54. <i>trans</i> -1,3-dichloropropene	74. 4-bromo-1-fluorobenzene (SMC)	94. hexachlorobutadiene
15. methyl <i>tert</i> -butyl ether	35. pentafluorobenzene (IS)	55. ethyl methacrylate	75. <i>n</i> -propylbenzene	95. 1,2,4-trichlorobenzene
16. diisopropyl ether	36. <i>tert</i> -amyl methyl ether	56. tetrachloroethene	76. 1,1,2,2-tetrachloroethane	96. naphthalene
17. 1,1-dichloroethane	37. benzene	57. 1,1,2-trichloroethane	77. bromobenzene	97. 1,2,3-trichlorobenzene
18. acrylonitrile	38. isobutyl alcohol (500ppb)	58. dibromochloromethane	78. 1,3,5-trimethylbenzene	
19. vinyl acetate*	39. 1,2-dichloroethane	59. 1,3-dichloropropane	79. 2-chlorotoluene	
20. allyl alcohol (250ppb)	40. isopropyl acetate	60. <i>n</i> -butyl acetate	80. 1,2,3-trichloropropane	

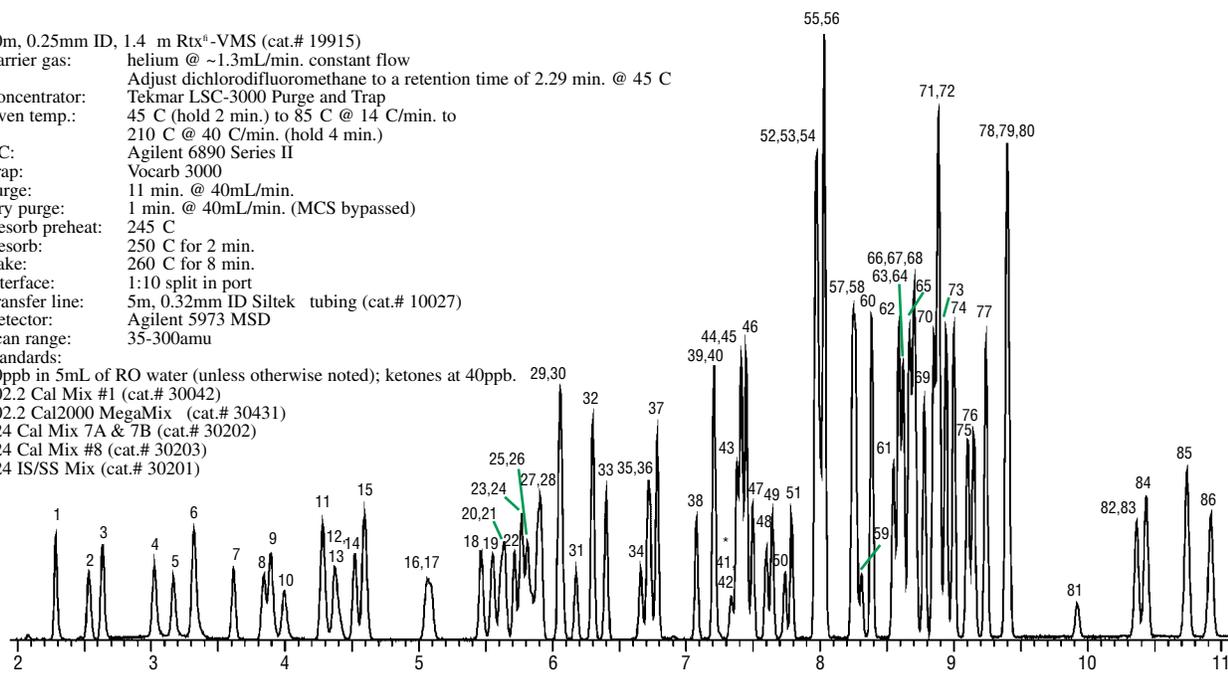
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*These compounds share ions, and coelute on "624"-type columns.

Figure 50.

Volatile Organics by US EPA Method 524.2, rev. IV. A 30m x 0.25mm ID column and 11 minute analysis provide excellent resolution of gaseous VOAs.

30m, 0.25mm ID, 1.4 m Rtx[®]-VMS (cat.# 19915)
 Carrier gas: helium @ ~1.3mL/min. constant flow
 Adjust dichlorodifluoromethane to a retention time of 2.29 min. @ 45 C
 Concentrator: Tekmar LSC-3000 Purge and Trap
 Oven temp.: 45 C (hold 2 min.) to 85 C @ 14 C/min. to 210 C @ 40 C/min. (hold 4 min.)
 GC: Agilent 6890 Series II
 Trap: Vocarb 3000
 Purge: 11 min. @ 40mL/min.
 Dry purge: 1 min. @ 40mL/min. (MCS bypassed)
 Desorb preheat: 245 C
 Desorb: 250 C for 2 min.
 Bake: 260 C for 8 min.
 Interface: 1:10 split in port
 Transfer line: 5m, 0.32mm ID Siltek tubing (cat.# 10027)
 Detector: Agilent 5973 MSD
 Scan range: 35-300amu
 Standards:
 20ppb in 5mL of RO water (unless otherwise noted); ketones at 40ppb.
 502.2 Cal Mix #1 (cat.# 30042)
 502.2 Cal2000 MegaMix (cat.# 30431)
 524 Cal Mix 7A & 7B (cat.# 30202)
 524 Cal Mix #8 (cat.# 30203)
 524 IS/SS Mix (cat.# 30201)



1. dichlorodifluoromethane	19. 2,2-dichloropropane	37. methyl methacrylate	55. <i>m</i> -xylene	73. <i>sec</i> -butylbenzene
2. chloromethane	20. bromochloromethane	38. <i>cis</i> -1,3-dichloropropene	56. <i>p</i> -xylene	74. <i>p</i> -isopropyltoluene
3. vinyl chloride	21. chloroform	39. toluene	57. <i>o</i> -xylene	75. 1,3-dichlorobenzene
4. bromomethane	22. methyl acrylate	40. chloroacetonitrile	58. styrene	76. 1,4-dichlorobenzene
5. chloroethane	23. carbon tetrachloride	41. 2-nitropropane*	59. bromoform	77. <i>n</i> -butylbenzene
6. trichlorofluoromethane	24. tetrahydrofuran (40ppb)	42. 1,1-dichloropropanone*	60. isopropylbenzene	78. hexachloroethane
7. diethyl ether	25. 1,1,1-trichloroethane	43. 4-methyl-2-pentanone	61. 4-bromofluorobenzene	79. 1,2-dichlorobenzene-d4
8. 1,1-dichloroethene	26. 2-butanone	44. tetrachloroethene	62. <i>n</i> -propylbenzene	80. 1,2-dichlorobenzene
9. carbon disulfide (40ppb)	27. 1,1-dichloropropene	45. <i>trans</i> -1,3-dichloropropene	63. bromobenzene	81. 1,2-dibromo-3-chloropropane
10. iodomethane (40ppb)	28. 1-chlorobutane	46. ethyl methacrylate	64. 1,1,2,2-tetrachloroethane	82. nitrobenzene
11. allyl chloride	29. benzene	47. 1,1,2-trichloroethane	65. 1,3,5-trimethylbenzene	83. hexachlorobutadiene
12. methylene chloride	30. propionitrile	48. dibromochloromethane	66. 2-chlorotoluene	84. 1,2,4-trichlorobenzene
13. acetone	31. 1,2-dichloroethane	49. 1,3-dichloropropane	67. 1,2,3-trichloropropane	85. naphthalene
14. <i>trans</i> -1,2-dichloroethene	32. fluorobenzene	50. 1,2-dibromoethane	68. <i>trans</i> -1,4-dichloro-2-butene	86. 1,2,3-trichlorobenzene
15. methyl <i>tert</i> -butyl ether	33. trichloroethene	51. 2-hexanone	69. 4-chlorotoluene	
16. 1,1-dichloroethane	34. dibromomethane	52. ethylbenzene	70. <i>tert</i> -butylbenzene	
17. acrylonitrile	35. 1,2-dichloropropane	53. chlorobenzene	71. 1,2,4-trimethylbenzene	
18. <i>cis</i> -1,2-dichloroethene	36. bromodichloromethane	54. 1,1,1,2-tetrachloroethane	72. pentachloroethane	

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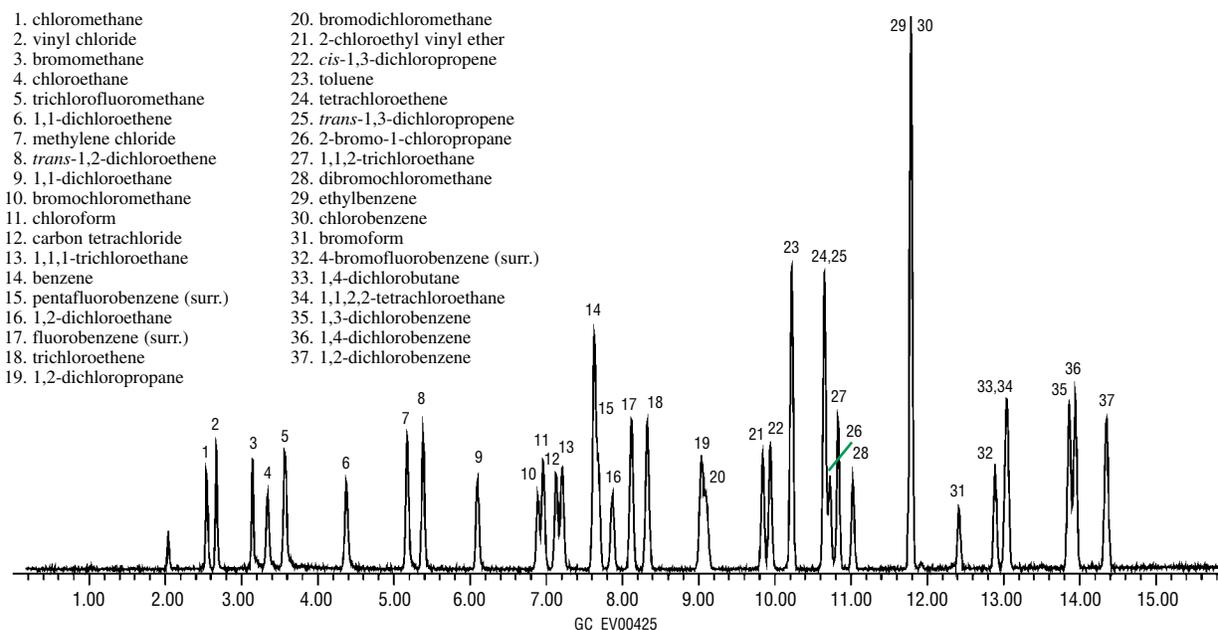
*These compounds share a quantitation ion (43)

Method 524.2: In EPA method updates, such as Method 524.2, rev. IV, minor ions from newly listed target compounds interfere with the quantification ions from other target compounds.¹⁷ An example of this problem occurs when a 75m, 0.53mm ID “624/1301” column is used to resolve methyl acrylate and propionitrile. The quantification ion for methyl acrylate is mass 55, and propionitrile has a minor ion of mass 55, which thus can interfere with determining concentrations of methyl acrylate in “real world” samples. 1,1-dichloro-2-propanone and 4-methyl-2-pentanone are another difficult pair to resolve on a “624/1301” column, because they share ion 43. These compounds can be resolved (in more than 30 minutes), however, by using a 60m, 0.32mm ID column. Because Rtx[®]-VMS columns were designed to resolve compounds by primary quantification ion, using extracted ion chromatography (Figure 50), the only compounds from Method 524.2, rev. IV, that are difficult for an Rtx[®]-VMS column to resolve are 2-nitropropane and 1,1-dichloro-2-propanone, (peaks 41 and 42) which share ion 43.

17. *Methods for the Determination of Organic Compounds in Drinking Water, Supplement II Method 524.2: U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring Systems Laboratory, Cincinnati, OH. 1992.*

Figure 51.

Gaseous analytes resolved to baseline on an Rtx®-VMS column.



30m, 0.25mm ID, 1.40 m Rtx®-VMS (cat#19915)

Conc.: 20 ppb in 5mL of RO water

Concentrator: Tekmar LSC-3000 Purge and Trap

Trap: Vocab 3000 (type K)

Purge: 11 min. @ 40mL/min. @ ambient temperature.

Dry purge: 1 min. @ 40mL/min. (MCS bypassed using Silcosteel® tubing)

Desorb preheat: 245 C

Desorb: 250 C for 2 min., Flow 10mL/min.

Bake: 260 C for 8 min.

GC Interface: 1:10 split at injection port. 1mm ID sleeve.

GC: HP 6890

Oven temp.: 40 C (hold 4 min.) to 95 C @ 24 C/min. (hold 3 min.), to

210 C @ 40 C/min. (hold 6 min.)

Carrier gas: helium @ ~1mL/min. constant flow

Adjust dichlorodifluoromethane to a retention time of

2.54 min. @ 40 C

Detector: HP 5973 MSD

Scan range: 25-300amu

Method 624: Previously, analysts used packed columns to perform Method 624, but now they generally use capillary chromatographic techniques. The compound list in Method 624 includes commonly analyzed aromatic and halogenated compounds in wastewater. This analysis can be achieved on many capillary columns; Figure 51 shows a 30m, 0.25mm ID Rtx®-VMS column resolves the gaseous analytes to the baseline.

Table VIII

Volatile organic compounds: retention time indexes

Data collected using 105m, 0.53mm ID, 3.0µm Rtx®-502.2, Rtx®-1, and Rtx®-624 columns.

Oven temp.: Rtx®-502.2: 35°C (hold 10 min.) to 220°C @ 4°C/min. (hold 2 min.), Rtx®-1: 35°C (hold 10 min.) to 220°C @ 4°C/min. (hold 2 min.), Rtx®-624: 35°C (hold 12 min.) to 220°C @ 7°C/min.; Carrier gas: helium; Regulation: constant flow; Flow rate: 10.0mL/min.

Component Name	Retention Time			Component Name	Retention Time			Component Name	Retention Time		
	Rtx-502.2	Rtx-1	Rtx-624		Rtx-502.2	Rtx-1	Rtx-624		Rtx-502.2	Rtx-1	Rtx-624
dichlorodifluoromethane	4.173	4.24	4.255	1,1-dichloropropene	20.163	17.808	19.448	<i>m</i> -xylene	33.351	30.898	28.37
C3	4.181	4.241	4.211	carbon tetrachloride	20.502	18.493	19.333	<i>p</i> -xylene	33.354	30.961	28.358
chloromethane	4.838	4.513	4.857	1,2-dichloroethane-d4	20.726	16.133	20.04	1-chloro-2-fluorobenzene	33.68	30.232	28.605
C4	5.136	5.319	6.321	benzene-d6	20.91	17.998	19.888	malononitrile	34.623	26.078	31.796
vinyl chloride	5.159	4.872	5.243	C7	20.935	21.532	20.582	<i>o</i> -xylene	34.877	32.149	29.238
ethylene oxide	6.289	5.467	6.389	1,2-dichloroethane	21.047	16.411	20.263	styrene	34.975	31.843	29.294
bromomethane	6.527	5.604	6.412	benzene	21.097	18.124	19.999	1,3-dichloro-2-propanol	36.032	31.018	31.41
chloroethane	6.812	5.938	6.798	fluorobenzene	21.897	18.917	20.802	bromoform	36.123	30.983	29.746
ethanol	7.109	6.261	9.056	2-chloroethanol	21.951	17.171	23.115	isopropylbenzene	36.139	33.718	30.014
C5	7.692	8.133	8.069	trichloroacetonitrile	22.125	33.558	30.181	<i>cis</i> -1,4-dichloro-2-butene	36.312	31.594	30.302
trichlorofluoromethane	7.715	7.426	7.672	1,4-difluorobenzene	22.209	19.061	21.179	1,4-dichlorobutane	36.471	31.864	30.377
diethyl ether	8.857	8.232	8.977	trichloroethene	23.326	20.674	21.713	1,1,2,2-tetrachloroethane	36.735	32.053	30.779
1,1,2-trichloro-1,2,2-trifluoroethane	9.142	9.745	10.044	trifluorotoluene	23.607	21.311	22.527	4-bromofluorobenzene	37.004	33.318	30.426
acrolein	9.285	6.831	9.817	1,2-dichloropropane	23.924	20.036	22.378	1,2,3-trichloropropane	37.233	32.402	30.902
acetone	9.558	7.042	10.6	methyl methacrylate	24.233	21.258	22.632	C10	37.305	37.539	31.095
1,1-dichloroethene	10.117	8.74	9.953	chloroacetonitrile	24.239	17.035	23.993	<i>trans</i> -1,4-dichloro-2-butene	37.516	32.626	30.929
<i>tert</i> -butyl alcohol	10.516	8.864	13.546	bromodichloromethane	24.743	20.545	23.045	<i>n</i> -propylbenzene	37.566	35.151	30.895
acetonitrile	10.661	6.571	12.909	1,4-dioxane	24.808	20.645	22.644	bromobenzene	37.652	33.875	30.772
iodomethane	11.311	8.567	10.634	dibromomethane	24.881	19.975	22.668	dibromoacetonitrile	37.812	30.956	31.86
methyl iodide	11.311	8.567	10.634	2-nitropropane	24.999	19.356	23.762	1,3,5-trimethylbenzene	38.133	35.817	31.268
allyl chloride	11.717	9.323	11.9	2-chloroethyl vinyl ether	25.888	22.108	23.796	2-chlorotoluene	38.166	34.986	31.145
<i>n</i> -propylamine	11.815	9.36	11.69	dichloroacetonitrile	25.894	19.998	25.478	4-chlorotoluene	38.33	35.214	31.4
carbon disulfide	12.069	9.658	10.747	4-methyl-2-pentanone (MIBK)	26.001	22.887	24.458	<i>tert</i> -butylbenzene	39.393	37.008	31.959
dichloromethane	12.169	9.001	13.034	epichlorohydrin	26.109	20.907	23.971	1,2,4-trimethylbenzene	39.521	37.033	32.089
methylene chloride	12.169	9.001	13.034	1,1-dichloropropanone	26.444	22.124	25.469	pentachloroethane	39.576	35.98	32.088
allyl alcohol	12.659	10.215	16.115	<i>cis</i> -1,3-dichloropropene	26.674	22.744	24.104	thiophenol	39.606	35.186	31.919
methyl <i>tert</i> -butyl ether	12.785	12.074	13.734	C8	27.186	27.642	24.868	<i>sec</i> -butylbenzene	40.151	37.886	32.428
acrylonitrile	13.429	7.947	14.158	toluene-d8	27.502	24.835	24.612	<i>p</i> -isopropyltoluene	40.657	38.413	32.731
<i>trans</i> -1,2-dichloroethene	13.438	11.346	13.725	pyridine	27.525	22.8	24.598	1,3-dichlorobenzene	40.938	37.404	32.765
C6	13.828	14.557	14.668	toluene	27.78	25.081	24.768	1,4-dichlorobenzene-d4	41.228	37.544	32.92
propargyl alcohol	14.458	11.32	18.532	<i>trans</i> -1,3-dichloropropene	28.428	24.014	25.417	1,4-dichlorobenzene	41.317	37.624	32.972
di-isopropyl ether	14.874	14.602	15.745	ethyl methacrylate	28.455	25.852	25.489	C11	41.585	41.76	33.638
1,1-dichloroethane	15.151	11.849	15.616	1,1,2-trichloroethane	28.925	24.384	25.829	benzyl chloride	41.739	37.378	33.279
vinyl acetate	15.256	12.283	15.883	2-bromo-1-chloropropane	28.965	25.018	25.569	<i>n</i> -butylbenzene	42.12	39.764	33.612
2-butanone	16.82	12.834	17.766	2-hexanone	29.03	25.778	26.326	bis(2-chloroisopropyl)ether	42.498	39.009	34.229
methyl ethyl ketone	16.82	12.834	17.766	1,3-dichloropropane	29.826	25.124	26.205	1,2-dichlorobenzene-d4	42.536	38.589	33.74
propionitrile	17.053	10.733	18.167	tetrachloroethene	30.036	27.8	25.928	1,2-dichlorobenzene	42.62	38.685	33.8
2,2-dichloropropane	17.337	14.659	17.506	dibromochloromethane	30.685	26.024	26.639	hexachloroethane	43.898	40.901	34.244
<i>cis</i> -1,2-dichloroethene	17.478	13.924	17.628	2-picoline	31.242	27.462	26.977	3-bromochlorobenzene	45.335	41.342	35.231
methacrylonitrile	17.837	12.732	18.505	1,2-dibromoethane (EDB)	31.375	26.613	26.924	1,2-dibromo-3-chloropropane	45.501	40.095	35.485
methylacrylate	17.947	14.54	18.351	1-chlorohexane	32.066	29.717	27.831	4-bromochlorobenzene	45.567	41.476	35.377
isobutyl alcohol	18.015	15.723	20.048	bromochloroacetonitrile	32.079	25.757	28.837	C12	45.586	45.659	35.96
chloroform	18.104	14.659	18.625	C9	32.546	32.893	28.243	nitrobenzene	45.742	40.282	35.992
pentafluorobenzene	18.628	16.371	19.273	1,1,1-trichloro-2-propanone	32.633	28.967	28.104	2-bromochlorobenzene	46.885	42.47	36.162
bromochloromethane	18.667	14.289	18.355	chlorobenzene-d5	32.749	29.372	27.887	bis(2-chloroethoxy)methane	47.514	42.935	36.98
tetrahydrofuran	18.777	15.554	18.341	ethylbenzene-d10	32.756	30.191	27.934	1,2,4-trichlorobenzene	48.643	44.704	37.165
dibromofluoromethane	18.893	14.966	19.086	chlorobenzene	32.864	29.473	27.949	hexachlorobutadiene	49.219	46.558	37.418
1,1,1-trichloroethane	19.575	17.042	18.972	ethylbenzene-d5	32.976	30.378	28.089	C13	49.276	49.295	38.078
cyclohexane	19.619	18.779	16.854	3-chloropropionitrile	33.001	24.824	29.176	naphthalene	49.544	45.073	37.729
1-chlorobutane	19.724	17.221	19.314	1,1,1,2-tetrachloroethane	33.03	29.429	28.133	1,2,3-trichlorobenzene	50.481	46.076	38.23
				ethylbenzene	33.087	30.476	28.118	C14	52.771	52.704	40.173

Table IX

Volatile organic compounds: retention time indexes

Data collected using a 60m, 0.25mm ID, 1.4µm Rtx®-VMS column; Oven: 40°C (hold 6 min.) to 230°C @ 14°C/min. (hold 11 min); Carrier gas: helium; Regulation: constant pressure; Flow rate: 1mL/min.; Linear velocity: 21cm/sec.; Dead time: 4.90 min.

Component Name	Rtx®-VMS Ret. Time	Component Name	Rtx®-VMS Ret. Time	Component Name	Rtx®-VMS Ret. Time	Component Name	Rtx®-VMS Ret. Time
dichlorodifluoromethane	5.52	methyl acrylate	13.87	4-methyl-2-pentanone	17.76	<i>trans</i> -1,4-dichloro-2-butene	21.40
chloromethane	6.26	carbon tetrachloride	13.94	2-bromo-1-chloropropane	17.81	2-chlorotoluene	21.40
vinyl chloride	6.54	tetrahydrofuran	14.03	2-nitropropane	17.83	4-chlorotoluene	21.61
water	6.70	1,1,1-trichloroethane	14.06	pyridine	17.86	cyclohexane	21.78
bromomethane	7.61	ethyl acetate	14.13	1,1-dichloropropanone	17.88	<i>tert</i> -butylbenzene	21.81
methanol	7.93	2-butanone	14.18	<i>trans</i> -1,3-dichloropropene	17.88	1-ethyl-2-methylbenzene	21.82
2-methylbutane	7.96	dibromofluoromethane	14.18	tetrachloroethene	17.89	1,2,4-trimethylbenzene	21.88
chloroethane	8.00	1,1-dichloropropene	14.20	ethyl methacrylate	17.92	pentachloroethane	21.92
trichlorofluoromethane	8.41	propargyl alcohol	14.35	1,1,2-trichloroethane	18.11	1,3-dichloro-2-propanol	22.05
<i>n</i> -pentane	8.61	1-chlorobutane	14.51	dibromochloromethane	18.40	<i>sec</i> -butylbenzene	22.06
diethylether	9.59	2,2,4-trimethylpentane	14.53	1,3-dichloropropane	18.49	isocaproic acid	22.09
1,1-dichloroethene	9.64	propionitrile	14.59	isobutyric acid	18.55	<i>p</i> -isopropyltoluene	22.22
carbon disulfide	9.65	benzene	14.60	1,2-dibromoethane	18.78	1,3-dichlorobenzene	22.53
Freon® 113	9.70	<i>n</i> -heptane (C7)	14.62	<i>n</i> -butyl acetate	18.80	caproic acid	22.55
ethanol	9.74	methacrylonitrile	14.64	2-hexanone	18.82	1,4-dichlorobenzene	22.64
iodomethane	9.99	benzene-d6	14.72	butyric acid	19.17	<i>n</i> -butylbenzene	22.88
3-chlorotrifluoropropane	10.45	pentafluorobenzene	14.75	1-chloro-3-fluorobenzene	19.17	malononitrile	22.89
chloro-methyl-methylether	10.54	1,2-dichloroethane-d4	14.79	ethylbenzene	19.36	benzyl chloride	23.23
acrolein	10.57	1,2-dichloroethane	14.90	chlorobenzene	19.39	1,2-dichlorobenzene-d4	23.36
2-methylpentane	10.59	<i>tert</i> -amyl-methyl ether	15.00	1-chloro-4-fluorobenzene	19.39	1,2-dichlorobenzene	23.38
allyl chloride	10.72	isobutyl alcohol	15.06	ethylbenzene-d10	19.40	hexachloroethane	23.63
methylene chloride	10.98	fluorobenzene	15.16	1-chlorohexane	19.41	1-octanol	23.70
3-methylpentane	11.09	isopropyl acetate	15.34	1,1,1,2-tetrachloroethane	19.44	<i>bis</i> (2-chloroisopropyl) ether	24.06
acetone	11.24	formic acid	15.37	<i>m</i> -xylene	19.53	4-bromo-1-chlorobenzene	24.09
<i>trans</i> -1,2-dichloroethene	11.24	trichloroethene	15.39	<i>p</i> -xylene	19.54	benzyl alcohol	24.23
methyl <i>tert</i> -butyl ether	11.42	1,4-difluorobenzene	15.58	chlorobenzene-d5	19.55	heptanoic acid	24.29
2-propanol	11.52	<i>n</i> -butanol	15.60	1-chloro-2-fluorobenzene	19.67	<i>n</i> -dodecane	24.54
<i>tert</i> -butyl alcohol	11.56	methyl cyclohexane	15.78	<i>o</i> -xylene	20.13	3-bromochlorobenzene	24.61
methyl acetate	11.63	acetic acid	15.93	styrene	20.17	1,2-dibromo-3-chloropropane	24.78
hexane	11.64	dibromomethane	16.05	isovaleric acid	20.18	2-bromochlorobenzene	25.54
acetonitrile	12.22	1,2-dichloropropane	16.17	bromoform	20.30	hexachlorobutadiene	25.99
chloroprene	12.30	bromodichloromethane	16.23	isopropylbenzene	20.51	nitrobenzene	26.02
1,1-dichloroethane	12.42	methyl methacrylate	16.28	1,2-butanediol	20.82	1,2,4-trichlorobenzene	26.19
acrylonitrile	12.60	α,α,α -trifluorotoluene	16.45	valeric acid	20.89	benzyl acetate	26.29
diisopropyl ether	12.62	1,4-dioxane	16.49	1,4-dichlorobutane	20.90	<i>n</i> -tridecane	26.51
2,4-dimethylpentane	12.68	<i>n</i> -propyl acetate	16.70	bromobenzene	20.91	naphthalene	27.01
vinyl acetate	13.02	2-chloroethyl vinyl ether	16.92	4-bromo-1-fluorobenzene	20.95	1,2,3-trichlorobenzene	27.46
ethyl- <i>tert</i> -butyl ether	13.08	2-chloroethanol	16.93	<i>cis</i> -1,4-dichloro-2-butene	20.97	<i>n</i> -tetradecane	28.83
1-propanol	13.18	<i>cis</i> -1,3-dichloropropene	17.04	<i>n</i> -decane	21.04	2-methylnaphthalene	30.36
<i>cis</i> -1,2-dichloroethene	13.32	1-bromo-2-chloroethane	17.05	<i>n</i> -propylbenzene	21.07	1-methyl-naphthalene	30.96
allyl alcohol	13.35	<i>n</i> -octane	17.17	1,1,2,2-tetrachloroethane	21.10	<i>n</i> -pentadecane	31.65
2,2-dichloropropane	13.48	toluene-d8	17.28	1,3,5-trimethylbenzene	21.30	2-chloronaphthalene	33.36
bromochloromethane	13.62	toluene	17.36	1-ethyl-4-methylbenzene	21.33		
chloroform	13.75	propionic acid	17.61	1-ethyl-3-methylbenzene	21.34		
cyclohexane	13.84	chloroacetonitrile	17.64	1,2,3-trichloropropane	21.39		

Choosing Columns for Subsets of Method 502.2/8021 Compound Lists

Table X shows the elution times for compounds listed in EPA Method 502.2 under the GC conditions given. Analysts monitoring subsets of Method 502.2 compounds may find one of these columns to be more suitable for the analysis than a commonly used volatile phase.

Table X.

Elution times for Method 502.2 compounds on various Rtx® GC columns.

Column: 60m x 0.53mm x 3.00 mdf

Flow: ~10mL/min., dead time 1.80 min.

Oven: 35 C (hold 9 min.) to 220 C @ 11 min. (hold 10 min.)

Elution Time in Minutes	Rtx-VGC	Rtx-502.2	Rtx-VRX	Rtx-624	Rtx-1	Rtx-1701	Rtx-200	Rtx-35	Rtx-50
dichlorodifluoromethane	2.31	2.18	2.54	2.59	2.16	1.99	1.95	2.04	1.96
chloromethane	2.64	2.51	2.75	2.94	2.30	2.36	2.18	2.39	2.39
vinyl chloride	2.78	2.67	2.99	3.16	2.51	2.51	2.23	2.51	2.47
bromomethane	3.35	3.35	3.46	3.86	2.89	3.16	2.62	3.21	3.27
chloroethane	3.58	3.44	3.66	4.07	3.04	3.32	2.85	3.21	3.27
trichlorofluoromethane	3.83	3.91	4.50	4.56	3.80	3.45	2.85	3.46	3.25
1,1-dichloroethene	4.83	4.93	5.32	5.75	4.74	4.57	3.34	4.58	4.46
Freon® 113	4.91	4.61	5.77	5.68	4.93	4.17	3.56	3.78	3.38
methylene chloride	6.10	6.02	5.68	7.39	4.62	6.64	3.97	6.07	6.40
trans-1,2-dichloroethene	6.49	6.78	7.07	8.07	5.85	6.68	4.20	6.40	6.59
methyl-tert-butyl-ether	6.80	6.67	7.68	8.21	6.28	6.34	4.88	5.76	5.52
tert-butyl alcohol	7.19	5.76	5.99	8.40	4.62	7.61	4.86	5.08	5.48
1,1-dichloroethane	8.10	8.08	7.74	9.74	6.16	8.68	5.54	8.00	8.30
cis-1,2-dichloroethene	9.80	10.14	9.47	11.51	7.59	10.75	6.12	10.27	10.50
2,2-dichloropropane	10.10	10.01	10.24	11.37	8.22	10.15	7.41	9.60	9.41
bromochloromethane	10.33	11.05	9.88	12.13	7.91	11.66	6.12	11.46	12.06
chloroform	10.61	10.65	10.11	12.38	8.21	11.79	5.69	10.66	11.02
carbon tetrachloride	10.87	12.31	12.60	12.87	11.39	11.26	6.44	11.80	11.72
1,1,1-trichloroethane	11.07	11.68	11.85	12.59	10.28	11.31	7.59	11.40	11.25
1,1-dichloropropene	11.39	12.09	12.31	12.98	10.89	11.67	7.79	11.74	11.62
benzene	11.97	12.72	12.70	13.43	11.13	12.44	8.66	12.86	12.95
1,2-dichloroethane	12.44	12.73	11.67	13.70	8.78	13.38	9.52	12.90	13.60
fluorobenzene	12.90	13.22	13.24	14.11	11.70	13.27	10.55	13.25	13.38
trichloroethene	13.24	14.08	14.09	14.83	12.89	13.79	9.52	13.93	13.98
dibromomethane	14.03	14.97	13.89	15.63	12.47	15.23	10.03	15.51	16.02
1,2-dichloropropane	14.25	14.44	14.00	15.39	12.51	14.83	12.04	14.54	14.77
bromodichloromethane	14.39	14.92	14.20	15.94	12.83	15.48	9.68	15.22	15.60
2-chloroethyl vinyl ether	15.48	15.57	14.92	16.52	13.80	16.08	13.32	15.81	16.01
cis-1,3-dichloropropene	15.54	15.96	15.46	16.76	14.18	16.21	13.19	16.20	16.36
toluene	15.95	16.53	16.74	17.23	15.48	16.30	13.55	16.58	16.57
tetrachloroethene	16.58	17.67	17.92	18.13	16.94	16.88	13.77	17.51	17.35
trans-1,3-dichloropropene	16.61	16.87	16.23	17.78	14.91	17.39	14.47	17.16	17.45
2-bromo-1-chloropropane	16.71	17.13	16.48	17.88	15.46	17.28	14.43	17.32	17.52
1,1,2-trichloroethane	16.91	17.13	16.45	18.10	15.13	17.76	14.23	17.49	17.74
dibromochloromethane	17.18	18.01	17.20	18.71	16.03	18.28	13.30	18.50	18.86
1,3-dichloropropane	17.36	17.57	16.86	18.38	15.33	17.99	15.64	17.91	18.10
1,2-dibromoethane	17.56	18.35	17.58	18.92	16.34	18.46	14.49	18.77	19.16
chlorobenzene	18.42	19.05	18.97	19.69	17.79	18.91	16.31	19.27	19.38
ethyl benzene	18.47	19.15	19.31	19.79	18.26	18.83	16.14	19.25	19.09
1,1,1,2-tetrachloroethane	18.53	19.13	18.87	19.83	17.78	19.14	16.02	19.29	19.39
m-xylene	18.69	19.28	19.62	19.98	18.48	19.02	16.42	19.27	19.17
p-xylene	18.71	19.28	19.62	19.99	18.48	19.02	16.45	19.27	19.17
1-chloro-2-fluorobenzene	19.08	19.44	19.14	20.20	18.16	19.50	17.28	19.61	19.67
o-xylene	19.37	20.01	20.22	20.66	19.07	19.74	17.26	20.07	20.02
styrene	19.45	20.04	20.10	20.69	18.93	19.92	17.44	20.25	20.33
bromoform	19.49	20.56	19.96	21.08	18.58	20.64	15.88	21.19	21.63
isopropyl benzene	19.86	20.60	20.77	21.23	19.80	20.20	17.54	20.54	20.35
bromobenzene	20.47	21.30	21.09	21.83	19.92	21.08	18.32	21.65	21.78
n-propyl benzene	20.51	21.22	21.43	21.89	20.46	20.87	18.20	21.19	20.99
1,4-dichlorobutane	20.56	20.71	19.95	21.54	18.94	21.14	19.02	21.01	21.16
1,1,2,2-tetrachloroethane	20.62	20.83	20.21	21.84	19.06	21.49	17.72	21.19	21.41
2-chlorotoluene	20.76	21.47	21.55	22.10	20.42	21.22	18.66	21.68	21.66
1,3,5-trimethylbenzene	20.81	21.52	21.88	22.17	20.75	21.15	18.50	21.42	21.31
1,2,3-trichloropropane	20.84	21.08	20.42	21.93	19.22	21.61	18.81	21.45	21.65
4-chlorotoluene	21.03	21.59	21.67	22.28	20.52	21.46	19.04	21.68	21.76
tert-butylbenzene	21.32	22.04	22.26	22.69	21.31	21.62	19.06	21.98	21.72
1,2,4-trimethylbenzene	21.42	22.11	22.44	22.78	21.32	21.79	19.15	22.09	22.02
sec-butylbenzene	21.60	22.38	22.59	23.04	21.69	21.95	19.16	22.29	22.04
p-isopropyl toluene	21.81	22.60	22.87	23.26	21.91	22.17	19.30	22.49	22.24
1,3-dichlorobenzene	21.95	22.77	22.66	23.33	21.54	22.49	19.87	22.95	23.03
1,4-dichlorobenzene	22.09	22.93	22.77	23.48	21.64	22.68	20.00	23.15	23.30
n-butylbenzene	22.47	23.25	23.46	23.92	22.53	22.85	20.12	23.18	22.89
1,2-dichlorobenzene	22.76	23.52	23.30	24.10	22.13	23.36	20.73	23.84	23.96
4-bromo-1-chlorobenzene	23.77	24.82	22.18	25.28	23.37	24.50	21.55	25.15	25.33
1,2-bromo-3-chloropropane	24.02	24.75	23.96	25.38	22.78	24.98	21.70	25.26	25.54
hexachlorobutadiene	25.03	26.37	26.42	26.82	25.55	25.53	22.42	26.41	26.15
1,2,4-trichlorobenzene	25.13	26.16	25.95	26.66	24.79	25.77	22.96	26.37	26.55
naphthalene	25.64	26.61	26.30	27.13	24.98	26.40	23.70	27.18	27.45
1,2,3-trichlorobenzene	25.94	27.04	26.63	27.57	25.41	26.72	23.81	27.46	27.65

Table XI.
Choosing a Volatiles GC Column for PID/ELCD

Restek Rtx® Phase	Coelutions by Peak #'s (Coelutions by PID/ELCD are indicated in BOLD)	Close Pairs by PID/ELCD	Suggested Confirmation Column	Poor Choice for Confirmation Column	Thick Phase Stable Temp. (°C)	Recommended High Temp. for VOA Work (°C)	Advantages
Rtx-VGC	28/29 ¹ , 53/54	7/8,32/33	Rtx-502.2, Rtx-VRX, Rtx-1	Rtx-VMS, Rtx-DX1	260	230	fast runtime
Rtx-502.2	14/15, 33/34, 39/40	4/5, 44/45, 56/57, 52/55, 64/65	Rtx-VGC, Rtx-VRX, Rtx-1	Rtx-Volatiles, Vocol, Rtx-35, Rtx-20	270	240	low bleed
Rtx-Volatiles	14/15, 21/22, 38/40, 44/45, 53/55	56/57, 68/69	Rtx-VGC, Rtx-1, Rtx-624	Rtx-502.2, Vocol, Rtx-20, Rtx-35	270	240	low bleed
Rtx-624	7/8, 10/11, 52/53, 31/34, 53/55, 59/60	32/33, 44/45, 51/54	Rtx-VGC, Rtx-502.2	Rtx-1701	280	240	
Rtx-VRX	11/13, 39/43, 46/50, 40/44	8/9,15/17, 24/27, 58/60	Rtx-VGC, Rtx-502.2	Rtx-1	260	230	
Rtx-1	9/12, 15/17, 25/26, 24/27, 33/36, 38/40, 40/44, 45/50, 56/57	7/12, 49/55	Rtx-502	Rtx-VRX	320	260	
Rtx-1701	9/10, 18/19, 16/20, 50/53, 51/55, 54/56	5/6,29/30,32/33	Rtx-502.2	Rtx-624	270	240	
Rtx-200	2/3, 5/6, 11/12, 14/16, 22/24, 28/35, 32/33/37, 43/44, 50/55/56, 57/58	13/17,36/37	Rtx-VGC		320	240	m/p xylene separation
Rtx-35	4/5, 16/19, 18/20, 21/22, 34/31, 39/38/41/42/40, 46/51/49, 53/54, 48/52/55, 61/62, 66/67	2/3	Rtx-VGC, Rtx-624	Rtx-502.2, Rtx-Volatiles, Rtx-20	270	240	
Rtx-50	4/5/6, 8/7/12, 25/28, 32/33, 37/41/42, 38/40, 45/47, 46/54/52, 56/55/48, 57/58	2/3, 20/18, 31/32, 39/41/42	Rtx-VGC, Rtx-624	Rtx-35	280	240	
Rtx-DX1 (custom)	4/5, 9/10, 25/27, 38/39, 47/50, 49/46/48, 52/54, 53/55	27/28, 32/36/31, 65/67	Rtx-502.2	Rtx-VGC	220	200	

¹ 2-chloroethyl vinyl ether can be resolved under different conditions. See the applications section of our current catalog.

Volatile Analytes:

- | | | | |
|--------------------------------------|---------------------------------------|-------------------------------|---------------------------------|
| 1. dichlorodifluoromethane | 19. 1,1,1-trichloroethane | 37. 1,2-dibromoethane | 55. 4-chlorotoluene |
| 2. chloromethane | 20. 1,1-dichloropropene | 38. chlorobenzene | 56. <i>tert</i> -butylbenzene |
| 3. vinyl chloride | 21. benzene | 39. ethyl benzene | 57. 1,2,4-trimethylbenzene |
| 4. bromomethane | 22. 1,2-dichloroethane | 40. 1,1,1,2-tetrachloroethane | 58. <i>sec</i> -butylbenzene |
| 5. chloroethane | 23. fluorobenzene | 41. <i>m</i> -xylene | 59. <i>p</i> -isopropyl toluene |
| 6. trichlorofluoromethane | 24. trichloroethene | 42. <i>p</i> -xylene | 60. 1,3-dichlorobenzene |
| 7. 1,1-dichloroethene | 25. dibromomethane | 43. 1-chloro-2-fluorobenzene | 61. 1,4-dichlorobenzene |
| 8. Freon®113 | 26. 1,2-dichloropropane | 44. <i>o</i> -xylene | 62. <i>n</i> -butylbenzene |
| 9. methylene chloride | 27. bromodichloromethane | 45. styrene | 63. 1,2-dichlorobenzene |
| 10. <i>trans</i> -1,2-dichloroethene | 28. 2-chloroethyl vinyl ether | 46. bromoform | 64. 4-bromo-1-chlorobenzene |
| 11. methyl <i>tert</i> -butyl ether | 29. <i>cis</i> -1,3-dichloropropene | 47. isopropyl benzene | 65. 1,2-bromo-3-chloropropane |
| 12. <i>tert</i> -butyl alcohol | 30. toluene | 48. bromobenzene | 66. hexachlorobutadiene |
| 13. 1,1-dichloroethane | 31. tetrachloroethene | 49. <i>n</i> -propylbenzene | 67. 1,2,4-trichlorobenzene |
| 14. <i>cis</i> -1,2-dichloroethene | 32. <i>trans</i> -1,3-dichloropropene | 50. 1,4-dichlorobutane | 68. naphthalene |
| 15. 2,2-dichloropropane | 33. 2-bromo-1-chloropropane | 51. 1,1,2,2-tetrachloroethane | 69. 1,2,3-trichlorobenzene |
| 16. bromochloromethane | 34. 1,1,2-trichloroethane | 52. 2-chlorotoluene | |
| 17. chloroform | 35. dibromochloromethane | 53. 1,3,5-trimethylbenzene | |
| 18. carbon tetrachloride | 36. 1,3-dichloropropane | 54. 1,2,3-trichloropropane | |

Compounds listed in US EPA Methods 502.2, 8021, 8010, 8020, 601 & 602, plus commonly added compounds.

m/p xylene coelute on all phases except Rtx®-200 in 60m, 0.25mm ID, 1.0µm under optimized conditions. See the applications section of our current catalog.

Conditions for Rtx®-VGC, Rtx®-502.2, Rtx®-Volatiles, Rtx®-VRX and Rtx®-1: optimum conditions on 75m, 0.45mm ID, 2.55µm columns. For more details please see chromatograms in the applications section of our current catalog or call technical service (1-800-356-1688, ext. 4) or your Restek representative.

Conditions for all other columns:

Column: 60m, 0.53mm ID, 3.0µm

Column Flow: 10mL/min.

GC Program: 35°C (hold 9 min.) to 220°C @ 11 min. (hold 10 min.)

Analytes identified using Thermo Finnigan PID/ELCD or HP5971a mass selective detector with splitless injection.

Conclusion

Analyses of volatile organic compounds – VOCs – generally require concentration of the sample using dynamic headspace (purge and trap), which reduces matrix effects and increases sensitivity, relative to other extraction techniques. The concentrated sample is transferred to the capillary GC column. Because GC detectors operate well with higher flows (10mL/min.), wide-bore columns, 0.45mm ID to 0.53mm ID, are appropriate. GC/MS instruments have the greatest sensitivity, but flow into the MS source cannot exceed 1mL/min. This makes narrow-bore columns, usually 0.18mm ID to 0.25mm ID, but up to 0.32mm ID, the preferred choice for GC/MS analysis. The column is plumbed through the injection port to allow high desorb flows (>10mL/min.) and sample splitting at the injection port (>10:1). Determining the type of stationary phase in the capillary column is a matter of preference and requires an examination of conditions specific to the analysis to be performed.

Information in this guide explains many of the factors to be considered in analyses of VOCs, but cannot anticipate every situation. If you have any questions regarding this guide, or your particular application, please contact our Technical Service Team via email at support@restekcorp.com or via phone at 800-356-1688 or 814-353-1300, ext. 4.

Recommended Products

Columns

Rtx [®] -502.2 Columns.....	61
Rtx [®] -Volatiles Columns.....	61
Rtx [®] -624 Columns.....	61
Rtx [®] -VMS Columns.....	61
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624 Mixes & Kits.....	67, 68
8260 Mixes & Kits.....	66, 67
CLP VOC Mixes & Kits.....	68-70
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Note: Many additional reference materials, including custom mixes, are available from Restek. Please call us at 800-356-1688, or contact your Restek distributor; we'll help you find what you're looking for.

Rtx®-VMS

- Special polymer formulation designed specifically for volatiles analysis by GC/MS.
- Complete separation of all US EPA Method 8260 compounds in less than 18 minutes.
- Excellent thermal stability resulting in low bleed.
- Wide variety of column dimensions.

Ordering Information | Rtx®-VMS (Fused Silica)

ID	df (µm)	temp. limits	30-Meter	60-Meter	75-Meter
0.25mm	1.40	-40 to 240/260°C	19915	19916	
0.45mm	2.55	-40 to 240/260°C	19908	19909	
0.53mm	3.00	-40 to 240/260°C	19985	19988	19974
ID	df (µm)	temp. limits	20-Meter	40-Meter	
0.18mm	1.00	-40 to 240/260°C	49914	49915	

Rtx®-VGC

- Special polymer formulation designed for volatiles analysis using PID/ELCD.
- Performs US EPA Method 8021A analysis in less than 28 minutes.
- Excellent separation of the trihalomethanes.
- Excellent inertness and thermally stable to 260°C.

Ordering Information | Rtx®-VGC (Fused Silica)

ID	df (µm)	temp. limits	30-Meter**	60-Meter**	75-Meter	105-Meter
0.25mm	1.40	-40 to 240/260°C	19415	19416		
0.45mm	2.55	-40 to 240/260°C	19408		19409	
0.53mm	3.00	-40 to 240/260°C	19485	19488	19474	19489
ID	df (µm)	temp. limits	20-Meter	40-Meter		
0.18mm	1.00	-40 to 240/260°C	49414	49415		

Rtx®-VRX

- Excellent selectivity for volatile compound analysis.
- Equivalent performance to DB-VRX column.
- Excellent for US EPA Method 8021 analyses.

Ordering Information | Rtx®-VRX (Fused Silica)

ID	df (µm)	temp. limits	30-Meter**	60-Meter**	75-Meter	105-Meter
0.25mm	1.40	-40 to 240/260°C	19315	19316		
0.45mm	2.55	-40 to 240/260°C	19308		19309	
0.53mm	3.00	-40 to 240/260°C	19385	19388	19374	19389
ID	df (µm)	temp. limits	20-Meter	40-Meter		
0.18mm	1.00	-40 to 240/260°C	49314	49315		

Rtx®-624

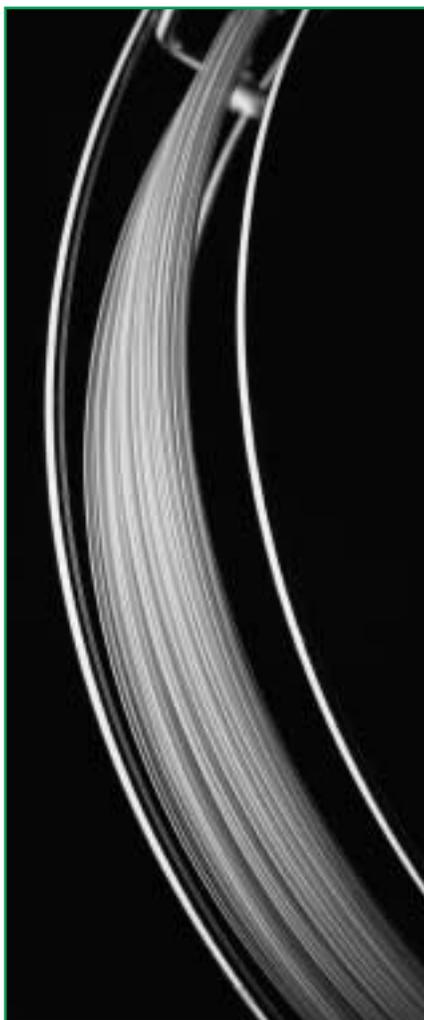
- Recommended for analyses of volatile organic compounds (VOCs) in EPA Methods.
- Crossbond® technology.
- 280°C thermal stability.
- Similar to DB-624 and HP-624 columns.

Ordering Information | Rtx®-624 (Fused Silica)

(Crossbond® 6% cyanopropylphenyl/94% dimethyl polysiloxane)

ID	df (µm)	temp. limits	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	1.40	-20 to 240°C	10968	10969		
0.45mm	2.55	-20 to 240°C			10982	
0.53mm	3.00	-20 to 240°C	10971	10973	10974	10975
ID	df (µm)	temp. limits	10-Meter	20-Meter	40-Meter	
0.18mm	1.00	-20 to 240°C		40924	40925	





Rtx®-502.2

- Recommended for the analysis of volatile organic compounds (VOCs) in EPA Methods.
- Specified in many GRO methods for monitoring leaking underground storage tanks.
- Crossbond® technology. **Reduced bleed, Increased column lifetime, Solvent rinsability.**
- Similar to DB-502.2 columns.

Ordering Information | Rtx®-502.2 (Fused Silica)

(EPA Volatiles in Methods 502.2, 524.2)

ID	df (µm)	temp. limits	30-Meter	60-Meter	75-Meter	105-Meter
0.25mm	1.40	-20 to 250/270°C	10915	10916		
0.45mm	2.55	-20 to 250/270°C			10986	
0.53mm	3.00	-20 to 250/270°C	10908	10909		10910
ID	df (µm)	temp. limits	20-Meter	40-Meter		
0.18mm	1.00	-20 to 250/270°C	40914	40915		

Rtx®-Volatiles

- Analyze volatile organic compounds (VOCs) in EPA methods.
- Crossbond® technology.
- 280°C thermal stability.
- Similar to VOCOL® columns.

Ordering Information | Rtx®-Volatiles (Fused Silica)

(EPA Volatile Organic Methods)

ID	df (µm)	temp. limits*	30-Meter	60-Meter	105-Meter
0.25mm	1.00	-20 to 270/280°C	10900	10903	
0.53mm	2.00	-20 to 270/280°C	10902	10905	10906

*The maximum temperatures listed are for 15- and 30-meter lengths.

Longer lengths may have a slightly reduced maximum temperature.

Rtx®-1

- Ideal for analysis of solvents and petrochemicals.
- Available in unbreakable Silcosteel® (MXT®) tubing.
- Thermally stable to 350°C (MXT® stable to 400°C).
- Similar to DB-1, SPB-1, HP-1, and Ultra-1 phases.

Ordering Information | Rtx®-1 (Fused Silica)

(Crossbond® 100% dimethyl polysiloxane)

ID	df (µm)	temp. limits	75-Meter	105-Meter
0.45mm	2.55	-60 to 270/290°C	10992	
0.53mm	3.00	-60 to 270/290°C		10189

The maximum temperatures listed are for 15- and 30-meter lengths.

Longer lengths may have a slightly reduced maximum temperature.

Intermediate-Polarity Deactivated Guard Columns & Transfer Lines

- Useful for a wide range of applications.
- Use with most common solvents.
- Maximum temperature: 325°C
- Reduce effects of dirty samples on column performance.
- Reduce downtime and maintenance.

Ordering Information | Fused Silica

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.
0.32mm	0.45 ± 0.04mm	10044	10044-600
0.53mm	0.69 ± 0.05mm	10045	10045-600

Ordering Information | MXT® (Silcosteel®)

Nominal ID	Nominal OD	5-Meter	5-Meter/6-pk.	10-Meter
0.53mm	0.74 ± 0.025mm	70045	70045-600	70047

* Not tested with the Grob test mix because of a large pressure drop.

** 30- and 60-meter lengths are banded in 5-meter sections.

Siltek™-Deactivated Guard Columns/Transfer Lines

- Revolutionary deactivation process lowers endrin breakdown to less than 1%.
- Minimize bleed.
- Ideal for chlorinated pesticide analysis.
- Analyze tough samples quickly and accurately.
- Maximum temperature of 380°C.
- Reduces effects of dirty samples on column performance.
- Reduces downtime and maintenance.

Ordering Information | Siltek™-Deactivated Guard Columns

Nominal ID	Nominal OD	5-Meter
0.32mm	0.45 ± 0.04mm	10027
0.53mm	0.69 ± 0.05mm	10028

Low-Volume Injector for Agilent 5890 Septum Packed Purge Port

- Allows syringe injections onto the column for purge & trap troubleshooting or calibration.
- Silcosteel® treatment eliminates adsorption of active compounds.
- Attaches to GC inlet without cutting injection port lines.

Everything you need for installation is provided, including a 1/16-inch nut, a 1/16-inch ferrule, a base nut and 1/4-inch Vespel®/graphite ferrule, a 1/16-inch capillary nut, a 5-pack of low-bleed plug septa, and a special low-mass septum nut. Order appropriate capillary ferrules separately—see our catalog.

Description	qty.	cat.#
Low-Volume Injector for Agilent 5890 Septum Packed Purge Port	kit	21698



Low-Volume Injector for Agilent GCs

- Fits Agilent split/splitless injectors.
- Attaches to the GC inlet without cutting existing injection port lines.
- Allows injections from a syringe onto the column for purge & trap troubleshooting or calibration.
- Silcosteel® treatment eliminates adsorption of active compounds.

Our low-volume injector can be installed in a matter of minutes. Remove the septum nut or splitless weldment and insert the Restek low-volume injector through the split injector. Tighten the base nut and you're ready! Includes a 1/16-inch nut, a 1/16-inch ferrule, a base nut and 1/4-inch Vespel®/graphite ferrule, a 1/16-inch capillary nut, a 5-pack of low-bleed plug septa, and a low-mass septum nut. Order appropriate capillary ferrules separately—see our catalog.

Description	qty.	cat.#
Low-Volume Injector for Agilent Split/Splitless GC Inlets	kit	21692



Low-Volume Injector for Varian Split/Splitless Inlets

- Attaches to the GC inlet without cutting existing injection port lines.
- Allows injections from a syringe onto the column for purge & trap troubleshooting or calibration.
- Silcosteel® treatment eliminates adsorption of active compounds.
- Order capillary ferrules separately—see our catalog.

Description	qty.	cat.#
Low-Volume Injector for Varian Split/Splitless GC Inlets	kit	21693



MXT®-Union Connector Kits—For Fused Silica Columns

- Low-dead-volume, leak-free connection.
- Reusable.
- Silcosteel® treatment ensures maximum inertness.
- Ideal for connecting a guard column or transfer line to an analytical column.
- Use to oven temperatures of 350°C.
- Available in union and “Y” configurations.



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

Description	qty.	cat.#
For 0.25mm ID Fused Silica Columns	kit	21386
For 0.32mm ID Fused Silica Columns	kit	21385
For 0.53mm ID Fused Silica Columns	kit	21384

Valco® Connectors—One-Piece Fused Silica Adaptor Ferrule

For connecting fused silica or 1/16- or 1/8-inch metal tubing.

- Use with Mxt®-Union Connectors
- Use to oven temperature of 350°C
- Made from graphite-reinforced polyimide



1/32-Inch Adaptor Ferrule

Tubing OD	Tubing ID	Valco® #	Valcon Polyimide	
			qty.	cat.#
0.25–0.4mm	0.25mm	FS.4-5	5-pk.	20137
0.4–0.5mm	0.32mm	FS.5-5	5-pk.	20140
0.5–0.8mm	0.53mm	ZF.5V-5	5-pk.	20141
1/32" Replacement Nut			5-pk.	20389

Heavy Duty Purge & Trap Syringe (Dynatech Precision Sampling)

- Heavy-duty glass barrel with metal front and rear flanges.
- Teflon® Luer-Lock® tip.
- Can fill and empty sparge tubes.
- Accepts standard Luer-Lock® needles.



Syringe	2mL cat.#	5mL cat.#	10mL cat.#
without Sample-Lok	21205	21206	21209
with Sample-Lok	—	21208	21207

Teflon® Tip, Gas-Tight Syringe Replacement Needles for Luer-Lock Syringes

Hub Material	Needle Gauge	Needle Length	Point Style	SGE cat.#	Restek	
					qty.	cat.#
metal	23	50mm	2	039802	5-pk.	24763
metal	22	2"/51mm	3	039895**	2-pk.	24765
metal	18	50mm	2	039842	5-pk.	24764

**For Rheodyne®/Valco® valves.

Leak Detective™ II Leak Detector

- Affordable thermal conductivity leak detector—every analyst should have one.
- Compact, ergonomic design is easy to hold and operate.
- Detects helium, hydrogen, and nitrogen at 1×10^{-4} cc/sec. or at an absolute concentration as low as 100ppm.*
- Fast results—responds in less than 2 seconds to trace leaks of gases with thermal conductivities different than air.
- Microchip design improves sensitivity and response time over previous models.
- Auto zeroing with the touch of a button.
- Battery-operated for increased portability (one 9-volt).

The compact, affordable tool that every analyst should have!

Description	qty.	cat.#
Leak Detective™ II Leak Detector	ea.	20413

*Never use liquid leak detectors on a capillary system because liquids can be drawn into the column.

Caution: NOT designed for determining leaks of combustible gases. A combustible gas detector should be used for determining combustible gas leaks in possible hazardous conditions.



Pre-Cleaned Volatile Organic Analyte Sampling Vials

- Container, liner and closure cleaned and assembled.
- Clear or amber.
- Open top caps.
- Teflon® faced 0.125" silicone septa.
- Each case lot numbered.

Description	qty.	cat.#
20mL Clear Pre-Cleaned VOA Vials	72-pk.	21798
20mL Amber Pre-Cleaned VOA Vials	72-pk.	21799
40mL Clear Pre-Cleaned VOA Vials	72-pk.	21796
40mL Amber Pre-Cleaned VOA Vials	72-pk.	21797



1mm ID Liners

for Agilent/Finnigan GCs	ID**/OD & Length (mm)	cat.# ea.	cat.# 5-pk.
	1.0 ID 6.3 OD x 78.5	20972	20973

for Varian GCs	ID**/OD & Length (mm)	cat.# ea.	cat.# 5-pk.
	1.0 ID 6.3 OD x 72	20970	20971

for Shimadzu GCs	ID**/OD & Length (mm)	cat.# ea.	cat.# 5-pk.	cat.# 25-pk.
	1.0 ID 5.0 OD x 95	20976	20977	20978

PSS Liners for PerkinElmer GCs	ID*/OD & Length (mm)	Similar to PE part #	cat.# ea.	cat.# 5-pk.	cat.# 25-pk.
	1.0 ID 4.0 OD x 86.2	N612-1006	20738	20741	—

for Thermo Finnigan 8000 & TRACE Series GCs	ID**/OD & Length (mm)	Similar to TF Part #	cat.# ea.	cat.# 5-pk.
	1.0 ID 8.0 OD x 105	453 20075	20916	20917

all liners are
100%
deactivated

We deactivate Restek liners using our unique polymeric deactivation process to ensure accurate chromatographic data. We evaluate them with an endrin breakdown test for complete inertness, and each liner is dimensionally checked for a perfect fit. Siltek™ and base deactivation are available for specialized analyses

Method 8260B

8260A/B Internal Standard Mix

chlorobenzene-d5 fluorobenzene
1,4-dichlorobenzene-d4
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30241	30241-510	—
w/data pack		
30241-500	30241-520	30341

8260A/B Surrogate Mix

4-bromofluorobenzene 1,2-dichloroethane-d4
dibromofluoromethane toluene-d8
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30240	30240-510	—
w/data pack		
30240-500	30240-520	30340

4-Bromofluorobenzene

4-bromofluorobenzene
2,500µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30067	30067-510	—
w/data pack		
30067-500	30067-520	30167

10,000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30082	30082-510	—
w/data pack		
30082-500	30082-520	30182

8260B Matrix Spike Mix

benzene toluene
chlorobenzene trichloroethylene
1,1-dichloroethene
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30479	30479-510	—
w/data pack		
30479-500	30479-520	30579

PFTBA (MS Tuning Compound)

perfluorotributylamine (PFTBA)
1mL/ampul, neat

Each
30482

VOA Calibration Mix #1 (ketones)

acetone 2-hexanone
2-butanone 4-methyl-2-pentanone
5,000µg/mL each in P&T methanol:water (90:10),
1mL/ampul

Each	5-pk.	10-pk.
30006	30006-510	—
w/data pack		
30006-500	30006-520	30106

8260B Calibration Mix #1

(76 + 1 components)

Note: This product is provided as a two ampul set:
acetonitrile *trans*-1,3-dichloropropene
acrylonitrile diethyl ether
allyl chloride 1,4-dioxane
benzene ethylbenzene
bromobenzene ethyl methacrylate
bromochloromethane hexachlorobutadiene
bromodichloromethane iodomethane
bromoform isobutyl alcohol
n-butylbenzene isopropylbenzene
sec-butylbenzene *p*-isopropyltoluene
tert-butylbenzene methacrylonitrile
carbon disulfide methyl acrylate
carbon tetrachloride methyl methacrylate
chlorobenzene methylene chloride
2-chloroethanol naphthalene
chloroform nitrobenzene
chloroprene 2-nitropropane
2-chlorotoluene pentachloroethane
4-chlorotoluene propionitrile
dibromochloromethane *n*-propylbenzene
1,2-dibromo-3-chloro- styrene
propane 1,1,1,2-tetrachloroethane
1,2-dibromoethane 1,1,2,2-tetrachloroethane
dibromomethane tetrachloroethene
1,2-dichlorobenzene tetrahydrofuran
1,3-dichlorobenzene toluene
1,4-dichlorobenzene 1,2,3-trichlorobenzene
cis-1,4-dichloro-2-butene 1,2,4-trichlorobenzene
trans-1,4-dichloro-2-butene 1,1,1-trichloroethane
1,1-dichloroethane 1,1,2-trichloroethane
1,2-dichloroethane trichloroethene
1,1-dichloroethene 1,2,3-trichloropropane
cis-1,2-dichloroethene 1,1,2-trichlorotrifluoroethane
trans-1,2-dichloroethene (Freon® 113)
1,2-dichloropropane 1,2,4-trimethylbenzene
1,3-dichloropropane 1,3,5-trimethylbenzene
2,2-dichloropropane *m*-xylene
1,1-dichloropropene *o*-xylene
cis-1,3-dichloropropene *p*-xylene

2,000µg/mL each in P&T methanol, 1mL/ampul

2-chloroethyl vinyl ether

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30475	30475-510	—
w/data pack		
30475-500	30475-520	30575

1,2-Dichlorotetrafluoroethane

1,2-dichlorotetrafluoroethane (Freon® 114)

2,000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30476	30476-510	—
w/data pack		
30476-500	30476-520	30576

California Oxygenates Mix

diisopropyl ether 2,000µg/mL *tert*-butyl alcohol 10,000
ethyl-*tert*-butyl ether 2,000 methyl *tert*-butyl ether 2,000
tert-amyl methyl ether 2,000

In P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30465	30465-510	—
w/data pack		
30465-500	30465-520	30565

Ethanol Mix

ethanol
10,000µg/mL in DI water, 1mL/ampul

Each	5-pk.	10-pk.
30466	30466-510	—
w/data pack		
30466-500	30466-520	30566

Acrolein Mix

acrolein
10,000µg/mL in water, 1mL/ampul

Each	5-pk.	10-pk.
30478	30478-510	—
w/data pack		
30478-500	30478-520	30578

8260B Acetate Mix

vinyl acetate *n*-propyl acetate
ethyl acetate *n*-butyl acetate
isopropyl acetate

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30477	30477-510	—
w/data pack		
30477-500	30477-520	30577

8260B Acetate Mix (Revised)

n-amyl acetate methyl acetate
butyl acetate propyl acetate
ethyl acetate vinyl acetate
isopropyl acetate

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30489	30489-510	—
w/data pack		
30489-500	30489-520	30589

Method 8260A

8260A Volatile Organics Kit

30005: VOA Matrix Spike Mix 30067: 4-bromofluorobenzene
30042: 502.2 Calib. Mix #1 30240: 8260A Surrogate Mix
30043: 502.2 Calib. Mix #2 30241: 8260A Internal
30044: 502.2 Calib. Mix #3 Standard Mix
30045: 502.2 Calib. Mix #4 30075: 8240/8260 System
30046: 502.2 Calib. Mix #5 Performance Check Mix
30047: 502.2 Calib. Mix #6

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30242	30242-500

Method 8260

8260 Internal Standard Mix

chlorobenzene-d5 1,4-difluorobenzene
1,4-dichlorobenzene-d4 pentafluorobenzene
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30074	30074-510	—
w/data pack		
30074-500	30074-520	30174

8260 Surrogate Mix

4-bromofluorobenzene toluene-d8
dibromofluoromethane
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30073	30073-510	—
w/data pack		
30073-500	30073-520	30173

8240/8260 System Performance Check Mix

bromoform 1,1-dichloroethane
chlorobenzene 1,1,2,2-tetrachloroethane
chloromethane
2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30075	30075-510	—
w/data pack		
30075-500	30075-520	30175

8240/8260 Calibration Check Mix

chloroform ethylbenzene
1,1-dichloroethene toluene
1,2-dichloropropane vinyl chloride
2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30427	30427-510	—
w/data pack		
30427-500	30427-520	30527

8260 Volatile Organics Kit

30005: VOA Matrix Spike Mix 30067: 4-bromofluorobenzene
30042: 502.2 Calib. Mix #1 30073: 8260 Surrogate Mix
30043: 502.2 Calib. Mix #2 30074: 8260 Internal
30044: 502.2 Calib. Mix #3 Standard Mix
30045: 502.2 Calib. Mix #4 30075: 8240/8260 System
30046: 502.2 Calib. Mix #5 Performance Check Mix
30047: 502.2 Calib. Mix #6

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30076	30076-500

PVOC, GRO, & BTEX

PVOC Mix (California)

benzene *m*-xylene
ethylbenzene *o*-xylene
methyl *tert*-butyl ether *p*-xylene
toluene

1,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30231	30231-510	—
w/data pack		
30231-500	30231-520	30331

PVOC/GRO Mix (Wisconsin)

benzene 1,2,4-trimethylbenzene
ethylbenzene 1,3,5-trimethylbenzene
methyl *tert*-butyl ether *m*-xylene
naphthalene *o*-xylene
toluene *p*-xylene

1,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30095	30095-510	—
w/data pack		
30095-500	30095-520	30195

GRO Mix

benzene 2,2,4-trimethylpentane
ethylbenzene (isooctane)
3-methylpentane toluene
naphthalene *m*-xylene
1,2,4-trimethylbenzene *o*-xylene

1,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30069	30069-510	—
w/data pack		
30069-500	30069-520	30169

Gasoline Component Standard

Component	Conc., (µg/mL)	Component	Conc., (µg/mL)
benzene	500	1,2,4-trimethylbenzene	1000
ethylbenzene	500	2,2,4-trimethylpentane	1500
heptane	500	<i>m</i> -xylene	1000
2-methylpentane	1500	<i>o</i> -xylene	1000
toluene	1500	<i>p</i> -xylene	1000

10,000µg/mL total in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30486	30486-510	—
w/data pack		
30486-500	30486-520	30586

GRO Mix (EPA)

benzene	500µg/mL	2,2,4-trimethylpentane	1,500
ethylbenzene	500	toluene	1,500
heptane	500	<i>m</i> -xylene	1,000
2-methylpentane	1,500	<i>o</i> -xylene	1,000
1,2,4-trimethylbenzene	1,000		

In P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30065	30065-510	—
w/data pack		
30065-500	30065-520	30165

BTEX Standard

benzene *m*-xylene
ethylbenzene *o*-xylene
toluene *p*-xylene

200µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30051	30051-510	—
w/data pack		
30051-500	30051-520	30151

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30213	30213-510	—
w/data pack		
30213-500	30213-520	30313

2,000µg/mL each in P&T methanol, except *m*-xylene and *p*-xylene at 1,000µg/mL, 1mL/ampul

Each	5-pk.	10-pk.
30488	30488-510	—
w/data pack		
30488-500	30488-520	30588

Certified BTEX in Unleaded Gas Composite Standard

Certified for:

benzene	toluene
ethylbenzene	<i>m</i> -xylene
isopropyl benzene	<i>o</i> -xylene
methyl <i>tert</i> -butyl ether	<i>p</i> -xylene
naphthalene	

5,500ppm gasoline in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30237	30237-510	—
w/data pack		
30237-500	30237-520	30337

BTEX Gas Mix

benzene *m*-xylene
ethylbenzene *o*-xylene
toluene *p*-xylene

In nitrogen, 104 liters @ 1800psig

1ppm	100ppb
34414 (ea.)	34428 (ea.)

Method 624

624 Internal Standard Mix

bromochloromethane 1,4-dichlorobutane
2-bromo-1-chloropropane
1,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30023	30023-510	—
w/data pack		
30023-500	30023-520	30123

624 Surrogate Standard Mix

4-bromofluorobenzene pentafluorobenzene
fluorobenzene
2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30243	30243-510	—
w/data pack		
30243-500	30243-520	30343

Surrogate Standard

1,4-bromofluorobenzene α,α,α-trifluorotoluene
2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30484	30484-510	—
w/data pack		
30484-500	30484-520	30584

624 Calibration Mix #1

bromomethane trichlorofluoromethane
chloroethane vinyl chloride
chloromethane

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30020	30020-510	—
w/data pack		
30020-500	30020-520	30120

624 Calibration Mix #2

benzene 1,1-dichloroethene
carbon tetrachloride 1,2-dichloropropane
chlorobenzene methylene chloride
2-chloroethyl vinyl ether tetrachloroethene
dibromochloromethane 1,1,2-trichloroethane
1,1-dichloroethane trichloroethene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30021	30021-510	—
w/data pack		
30021-500	30021-520	30121

624 Calibration Mix #3

bromodichloromethane *trans*-1,2-dichloroethene
bromoform *cis*-1,3-dichloropropene
chloroform *trans*-1,3-dichloropropene
1,2-dichlorobenzene ethylbenzene
1,3-dichlorobenzene 1,1,2,2-tetrachloroethane
1,4-dichlorobenzene toluene
1,2-dichloroethane 1,1,1-trichloroethane

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30022	30022-510	—
w/data pack		
30022-500	30022-520	30122

624 Kit

30020: 624 Calib. Mix #1 30022: 624 Calib. Mix #3
30021: 624 Calib. Mix #2 30023: 624 Int. Standard Mix

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30055	30055-500

624 Complete Kit

30020: 624 Calib. Mix #1 30023: 624 Int. Standard Mix
30021: 624 Calib. Mix #2 30243: 624 Surrogate
30022: 624 Calib. Mix #3 Standard Mix

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30244	30244-500

Method 601 & 602

601/602 Purgeable Halocarbons Kit

30042: 502.2 Calib. Mix #1 30022: 624 Calib. Mix #3
30021: 624 Calib. Mix #2

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30061	30061-500

602 Purgeable Aromatics Calibration Mix

benzene 1,4-dichlorobenzene
chlorobenzene ethylbenzene
1,2-dichlorobenzene toluene
1,3-dichlorobenzene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30035	30035-510	—
w/data pack		
30035-500	30035-520	30135

Method 502.2

502.2 CAL2000 MegaMix™ Mixture

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

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30431	30431-510	—
w/data pack		
30431-500	30431-520	30531

200µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30432	30432-510	—
w/data pack		
30432-500	30432-520	30532

1-Chloro-2-fluorobenzene

1-chloro-2-fluorobenzene
2,000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30040	30040-510	—
w/data pack		
30040-500	30040-520	30140

502.2 Internal Standard Mix #2

2-bromo-1-chloropropane fluorobenzene
2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30041	30041-510	—
w/data pack		
30041-500	30041-520	30141

8021/502.2 Surrogate Mix #1

1-bromo-2-chloroethane fluorobenzene
1-chloro-3-fluorobenzene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30463	30463-510	—
w/data pack		
30463-500	30463-520	30563

8021/502.2 Surrogate Mix #2

1-bromo-2-chloroethane 1-chloro-3-fluorobenzene
4-bromochlorobenzene fluorobenzene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30464	30464-510	—
w/data pack		
30464-500	30464-520	30564

502.2 VOA Calib. Kit #2 (2000ppm)

30442: 502.2 Calib. Mix #1
30431: 502.2 CAL2000 MegaMix™

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30445	30445-500

502.2 VOA Calib. Kit #3 (200ppm)

30439: 502.2 Calib. Mix #1A
30432: 502.2 CAL200 MegaMix™

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30446	30446-500

502.2 Calibration Mix #1 (gases)

bromomethane dichlorodifluoromethane
chloroethane trichlorofluoromethane
chloromethane vinyl chloride

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30042	30042-510	—
w/data pack		
30042-500	30042-520	30142

502.2 Calibration Mix #1A

bromomethane dichlorodifluoromethane
chloroethane trichlorofluoromethane
chloromethane vinyl chloride

200µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30439	30439-510	—
w/data pack		
30439-500	30439-520	30539

04.2, 04.1, & 3/90 SOW

CLP 04.1 VOA Internal Standard/SMC Spike Mix

bromochloromethane 1,2-dichloroethane-d4
4-bromofluorobenzene 1,4-difluorobenzene
chlorobenzene-d5 toluene-d8

2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30457	30457-510	—
w/data pack		
30457-500	30457-520	30557

VOA Internal Standard Mix

bromochloromethane chlorobenzene-d5
1,4-difluorobenzene

2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30011	30011-510	—
w/data pack		
30011-500	30011-520	30111

VOA Surrogate Spike Mix

4-bromofluorobenzene toluene-d8
1,2-dichloroethane-d4

2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30004	30004-510	—
w/data pack		
30004-500	30004-520	30104

VOA Matrix Spike Mix

benzene toluene
chlorobenzene trichloroethene
1,1-dichloroethene

2,500µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30005	30005-510	—
w/data pack		
30005-500	30005-520	30105

VOA Screening Mix #1

benzene o-xylene
ethylbenzene p-xylene
toluene

1,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30001	30001-510	—
w/data pack		
30001-500	30001-520	30101

VOA Screening Mix #2

n-dodecane n-nonane
1,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30002	30002-510	—
w/data pack		
30002-500	30002-520	30102

VOA Tuning Compound

4-bromofluorobenzene
5,000µg/mL in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30003	30003-510	—
w/data pack		
30003-500	30003-520	30103

PFTBA (MS Tuning Compound)

perfluorotributylamine (PFTBA)
1mL/ampul, neat

Each
30482

DID YOU KNOW?

Restek reference materials include a silanized vial for sample transfer.



3/90 SOW

CLP VOA CAL2000

MegaMix™ Mixture (29 components)

Note: This product is provided as a two-ampul set:

benzene	<i>cis</i> -1,3-dichloropropene
bromodichloromethane	<i>trans</i> -1,3-dichloropropene
bromoforn	ethylbenzene
carbon disulfide	methylene chloride
carbon tetrachloride	styrene
chlorobenzene	1,1,2,2-tetrachloroethane
chloroform	tetrachloroethene
dibromochloromethane	toluene
1,1-dichloroethane	1,1,1-trichloroethane
1,2-dichloroethane	1,1,2-trichloroethane
1,1-dichloroethene	trichloroethene
<i>cis</i> -1,2-dichloroethene	<i>m</i> -xylene
<i>trans</i> -1,2-dichloroethene	<i>o</i> -xylene
1,2-dichloropropane	<i>p</i> -xylene

2,000µg/mL each in P&T methanol, 1mL/ampul

vinyl acetate

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30438	30438-510	—
w/data pack		
30438-500	30438-520	30538

CLP VOA Calibration Kit #2

30006: VOA Calibration Mix #1 (ketones)

30010: VOA Calibration Mix #5 (gases)

30438: CLP VOA CAL2000 MegaMix™

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30442	30442-500

VOA Calibration Mix #5 (gases)

bromomethane

chloroethane

chloromethane

vinyl chloride

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30010	30010-510	—
w/data pack		
30010-500	30010-520	30110

VOA Calibration Mix #2

benzene

carbon disulfide

ethylbenzene

toluene

vinyl acetate

o-xylene

p-xylene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30007	30007-510	—
w/data pack		
30007-500	30007-520	30107

VOA Calibration Mix #3

carbon tetrachloride

chlorobenzene

chloroform

1,1-dichloroethane

1,1-dichloroethene

1,2-dichloropropane

methylene chloride

1,1,2-trichloroethane

trichloroethene

m-xylene

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30008	30008-510	—
w/data pack		
30008-500	30008-520	30108

VOA Calibration Mix #4

bromodichloromethane

bromoform

dibromochloromethane

1,2-dichloroethane

cis-1,2-dichloroethene

trans-1,2-dichloroethene

cis-1,3-dichloropropene

trans-1,3-dichloropropene

styrene

1,1,2,2-tetrachloroethane

tetrachloroethene

1,1,1-trichloroethane

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30009	30009-510	—
w/data pack		
30009-500	30009-520	30109

CLP Volatile Organics Kit #2

30001: VOA Screening Mix #1

30002: VOA Screening Mix #2

30003: VOA Tuning Comp.

30004: VOA Surr. Spike Mix

30005: VOA Matrix Spike Mix

30006: VOA Calib. Mix #1

(ketones)

30010: VOA Calib. Mix #5

(gases)

30011: VOA Int'l Standard Mix

30438: CLP VOA CAL2000

MegaMix™

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30440	30440-500

Volatile Organics Kit

30001: VOA Screening Mix #1

30002: VOA Screening Mix #2

30003: VOA Tuning Comp.

30004: VOA Surr. Spike Mix

30005: VOA Matrix Spike Mix

30006: VOA Calib. Mix #1

(ketones)

30007: VOA Calib. Mix #2

30008: VOA Calib. Mix #3

30009: VOA Calib. Mix #4

30010: VOA Calib. Mix #5

(gases)

30011: VOA Internal

Standard Mix

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30050	30150

04.2 & 04.1 SOW

CLP 04.1 VOA CAL2000

MegaMix™ Mixture (40 components)

benzene	<i>trans</i> -1,3-dichloropropene
bromodichloromethane	ethylbenzene
bromoform	isopropylbenzene
carbon disulfide	methyl acetate
carbon tetrachloride	methylcyclohexane
chlorobenzene	methylene chloride
chloroform	styrene
1,2-dibromo-3-chloropropane	methyl <i>tert</i> -butyl ether (MTBE)
cyclohexane	1,1,2,2-tetrachloroethane
dibromochloromethane	tetrachloroethene
1,2-dibromoethane	toluene
1,2-dichlorobenzene	1,2,4-trichlorobenzene
1,3-dichlorobenzene	1,1,1-trichloroethane
1,4-dichlorobenzene	1,1,2-trichloroethane
1,1-dichloroethane	trichloroethylene
1,2-dichloroethane	1,1,2-trichlorotrifluoroethane
1,1-dichloroethene	(Freon® 113)
<i>cis</i> -1,2-dichloroethene	<i>m</i> -xylene
<i>trans</i> -1,2-dichloroethene	<i>o</i> -xylene
1,2-dichloropropane	<i>p</i> -xylene
<i>cis</i> -1,3-dichloropropene	

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30456	30456-510	—
w/data pack		
30456-500	30456-520	30556

502.2 Calibration Mix #1 (gases)

bromomethane	dichlorodifluoromethane
chloroethane	trichlorofluoromethane
chloromethane	vinyl chloride

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30042	30042-510	—
w/data pack		
30042-500	30042-520	30142

502.2 Calibration Mix #1A

bromomethane	dichlorodifluoromethane
chloroethane	trichlorofluoromethane
chloromethane	vinyl chloride

200µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30439	30439-510	—
w/data pack		
30439-500	30439-520	30539

CLP 04.1 VOA Kit #3

30006: VOA Calibration Mix #1 (ketones)
30042: 502.2 Calibration Mix #1 (gases)
30456: CLP 04.1 VOA CAL2000 MegaMix™

Contains 1mL each of these mixtures.

Kit	Kit w/Data Pack
30460	30460-500

OLC 03.2 VOA MegaMix™ Mixture

(42 components)

benzene	<i>trans</i> -1,3-dichloropropene (E)
bromochloromethane	ethylbenzene
bromodichloromethane	isopropylbenzene (cumene)
bromoform	methyl acetate
carbon disulfide	methylcyclohexane
carbon tetrachloride	methyl <i>tert</i> -butyl ether (MTBE)
chlorobenzene	methylene chloride
chloroform	(dichloromethane)
cyclohexane	styrene
dibromochloromethane	1,1,2,2-tetrachloroethane
(chlorodibromomethane)	tetrachloroethylene
1,2-dibromo-3-chloropropane	toluene
1,2-dibromoethane (EDB)	1,2,3-trichlorobenzene
1,2-dichlorobenzene	1,2,4-trichlorobenzene
1,3-dichlorobenzene	1,1,1-trichloroethane
1,4-dichlorobenzene	1,1,2-trichloroethane
1,1-dichloroethane	trichloroethylene
1,2-dichloroethane	1,1,2-trichlorotrifluoroethane
1,1-dichloroethylene	(Freon® 113)
<i>cis</i> -1,2-dichloroethylene	<i>m</i> -xylene
<i>trans</i> -1,2-dichloroethylene	<i>o</i> -xylene
1,2-dichloropropane	<i>p</i> -xylene
<i>cis</i> -1,3-dichloropropene(Z)	

2,000µg/mL each in P&T methanol, 1mL/ampul

Each	5-pk.	10-pk.
30492	30492-510	—
w/data pack		
30492-500	30492-520	30592

OLC 03.2 VOA Deuterated Monitoring Compounds (DMC)

(14 components)

Note: This product is provided as a two-ampule set.

Non-Ketones:

benzene-d6	1,1-dichloroethene-d2
bromoform-d	1,2-dichloropropane-d6
chloroethane-d5	<i>trans</i> -1,3-dichloropropene-d4
chloroform-d	1,1,2,2-tetrachloroethane-d2
1,2-dichlorobenzene-d4	toluene-d8
1,2-dichloroethane-d4	vinyl chloride-d3

100µg/mL each in P&T methanol, 1mL/ampul

Ketones:

2-butanone-d5	2-hexanone-d5
---------------	---------------

200µg/mL each in P&T methanol, 0.5mL/ampul

Each	5-pk.
30493	30493-510

EPA Contract Lab Program (CLP)

Statement of Work (SOW) OLC03.2 is an analytical method for the detection of low concentration volatile, semivolatile, and pesticide/Arclor® organics in aqueous samples. For VOA and SVOA GC/MS analysis OLC03.2 introduces Deuterated Monitoring Compounds (DMCs) as a sample-by-sample accuracy indicator.

Can't locate the exact mixture you need?



With thousands of compounds in our inventory, we can make any mixture to your specifications.

Reach for Restek

Plus 1™ Restek's Customer Commitment

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