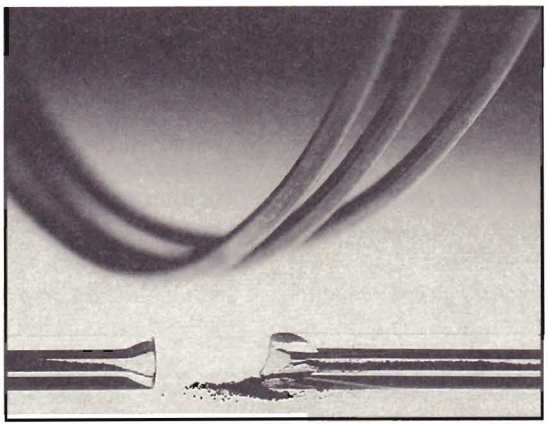
THE RESTEK ADVANTAGE

Analyze Flavor Volatiles in Alcoholic Beverages

Using Restek's New Silcosteel® CarboBlack™ Packed Columns

The overall flavor and quality of alcoholic beverages is determined by the amount of acids, alcohols, and aldehydes present in distilled spirits. The Association of Official Analytical Chemists (AOAC) methods provide information on acetic acid analysis and determinations for fusel oils, methanol, ethanol, and higher alcohols.[1] Gas chromatography provides a rapid and simplified method to analyze all of these components simultaneously without performing any preliminary extractions. Glass packed columns are commonly used for such analyses, but they are prone to breakage and adsorption. Restek's new Silcosteel® CarboBlack™ columns are made from stainless steel coated with a deactivated fused silica inner layer, providing improved inertness, durability and flexibility compared to traditional glass packed columns. CarboBlack™ columns yield excellent separation and reproducible quantitation of the important volatile components in alcoholic beverages such as scotch whiskey and rum.



No more mess from broken glass packed columns when you use Silcosteel® CarboBlack™ columns.

Silcosteel® CarboBlack™ columns offer greater versatility over glass packed columns

Stainless steel tubing provides greater flexibility, durability, and easier handling than glass columns. These columns also offer superior column reproducibility due to more stringent tolerances for inside diameters. The typical ID tolerance for stainless steel is 0.001" compared to .0025" for glass. In addition, the ID tolerance of glass tubing is further affected during the coiling process. This is attributable to burner temperature inconsistencies and coiling speed. This results in poor column-to-column reproducibility which is not a concern with Silcosteel® tubing. The flexibility and strength of stainless steel allows columns to be coiled to a 3" diameter without fear of breakage.

Five percent Carbowax® 20M on 80/120 mesh CarboBlack™ B columns are available in 1/8" OD with a 2mm ID. A new Packed Column Inlet Adaptor Fitting is available to convert 1/4"

in this issue...

The Restek Advantage is printed on recycled paper.

injection ports for use with this 1/8" tubing. This adaptor allows on-column injection with minimal sample backflash. The column can be installed directly into the detector port using a 1/4" to 1/8" reducing ferrule. Unlike traditional packed columns, the new CarboBlack™ columns have been pre-conditioned, eliminating the need for overnight conditioning. Only 20-30 minutes of conditioning at 170°C following column installation is required.

CarboBlack™ columns permit accurate acid determination

Acid concentration is indicative of maturity in distilled spirits such as Scotch whiskey. Fresh-distilled whiskey contains a low concentration of acids, whereas total acid content increases as whiskey is aged.[2] The predominant acid in whiskey is acetic acid. AOAC Method #945.08 recommends a titration method for acetic acid determination.[1] Packed column chromatography is a practical alternative since it provides accurate qualitative and quantitative results for acids. The glass wool end plugs are treated with phosphoric acid to prevent adsorption of these acidic components. To ensure minimal acid adsorption, new columns can be pre-treated by injecting several microliters of a 0.1% phosphoric acid solution. Note the good peak shape of the acetic acid in the scotch sample on the CarboBlack™ column in Figure 1.

Achieve excellent peak symmetry and resolution of alcohols

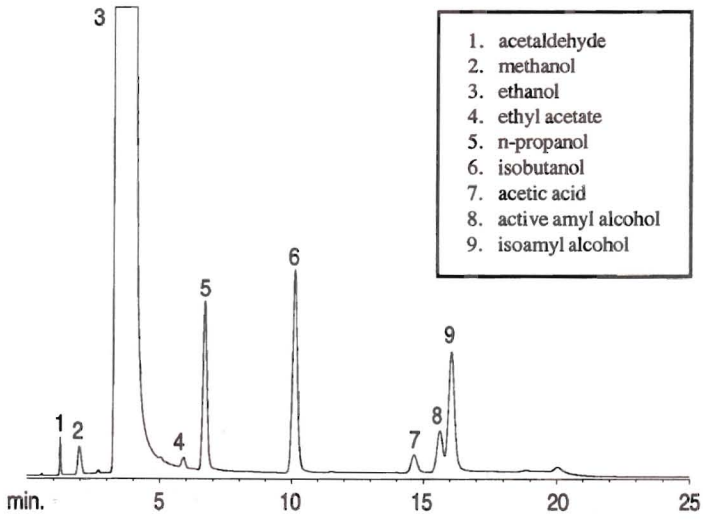
Percent alcohol concentration can be accurately determined on CarboBlack™ columns. Methanol, ethanol, and fusel oils (aliphatic alcohols between ethanol and isoamyl alcohol) can be analyzed for individual concentrations or for total alcohol content. Ethanol has the highest concentration of the alcohols, and determines proof value. Methanol and isopropanol determination allow detection of denaturants in alcoholic beverages.[3] Beverage analysts often experience poor methanol peak shape. Note the excellent peak shape of methanol and its resolution from ethanol on the CarboBlack™ columns as shown in Figures 1 and 2.

The two predominate fusel oils, active-amyl and isoamyl alcohol, are difficult to separate. These compounds, along with isobutanol, predominately influence the aroma strength of the alcoholic beverages.[2] Resolution of these components is difficult to achieve even with capillary columns. CarboBlack™ packed columns yield 60% resolution between active-amyl and isoamyl alcohols as shown in Figures 1 and 2.

Accurately determine carbonyl concentration in alcoholic beverages

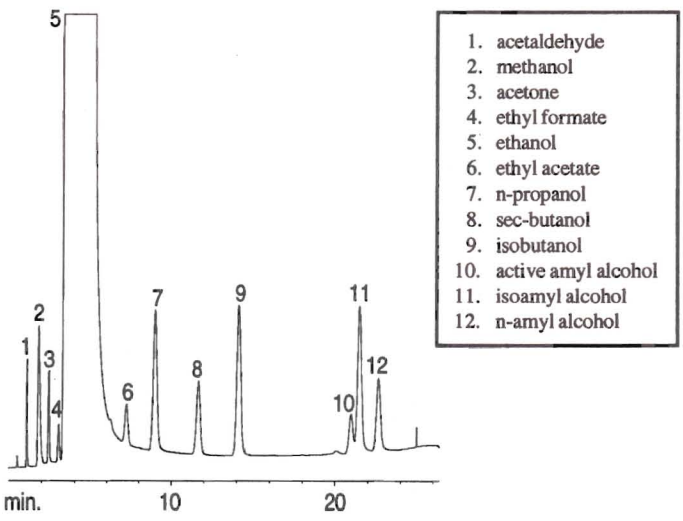
Recognition of aldehydes, ketones, and esters is important in analyses of distilled and fermented beverages. Acetaldehyde, the predominate aldehyde in most alcoholic beverages, exhibits a pungent odor that is softened by acetal formation during the maturation process.[2] Ethyl formate and ethyl acetate concentrations increase consistently with age and are reliable markers for age determination, especially of whiskeys. Eight-year-old Bourbon contains about 19 times more ethyl acetate and 10 times more ethyl formate as unaged Bourbon.[3] Acetone determination detects any adulteration or contamination present.[3] All of these carbonyl compounds in distilled spirits are completely resolved on a CarboBlack™ column, as shown in Figure 2.

Figure 1 - Unbreakable Silcosteel® CarboBlack™ B packed columns provide excellent resolution of Scotch whiskey.



2m, 1/8" OD x 2mm ID, 5% Carbowax 20M 80/120 CarboBlack™ B column (cat.# 80105)
0.5µl on-column injection of Scotch, on-column concentration: neat
Oven temp.: 70°C to 150°C @ 4°C/min.
Inj./det. temp.: 200°C/250°C **Detector:** FID
Carrier gas: nitrogen **Column flow rate:** 20ml/min.

Figures 2 - CarboBlack™ B columns resolve important flavor volatiles in fresh-distilled and aged rum.



2m, 1/8" OD x 2mm ID, 5% Carbowax 20M 80/120 CarboBlack™ B column (cat.# 80105)
0.5µl on-column injection of fusel oils in rum, on-column concentration: neat
Oven temp.: 65°C (hold 5 min.) to 150°C @ 4°C/min.
Inj./det. temp.: 200°C/250°C **Detector:** FID
Carrier gas: nitrogen **Column flow rate:** 20ml/min.

The new Silcosteel® CarboBlack™ columns can be used to effectively analyze the important flavor volatiles in distilled spirits such as scotch and rum. All components are resolved to provide accurate and reproducible quantitation. Even active compounds such as alcohols and acetic acid exhibit excellent response. These new columns provide more versatility over glass packed columns traditionally used for alcoholic beverage analysis. Stainless steel tubing provides easier handling, eliminates breakage, and offers greater precision of column dimensions. The fused silica inner layer ensures inertness. Flexibility of stainless steel allows these packed columns to be wound to smaller diameters to accommodate ovens of various sizes. A new adaptor allows for on-column injection into any 1/4" injection port.

References

1. AOAC, *Official Book of Methods of AOAC, 15th ed.*, 1990.
2. Suomalainen, H. and L. Nykäne, "Composition of Whiskey Flavour", *Process Biochemistry*, July, 1970.
3. Deman, *Principles of Food Chemistry*, 1990. pp. 48-50.

CarboBlack™ Packed Columns Product Listing

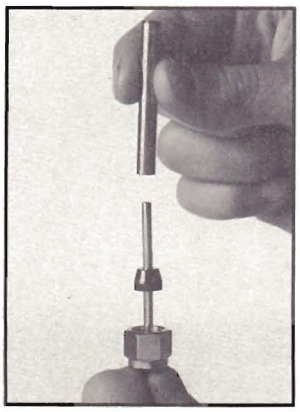
To order, specify Restek catalog number and instrument configuration. For example, a 2m, 1/8" OD, 2mm ID 1% Rt-1000 on 60/80 CarboBlack™ B, used in an HP 5890 GC would be part number: 80207-810.

Phase	Packing	Length	OD	ID	Catalog #	Application
5% Carbowax® 20M	80/120 CarboBlack™ B	2m	1/8"	2mm	80105-???	Flavor volatiles in alcoholic beverages
5% Carbowax® 20M	60/80 CarboBlack™ B	1.8m	1/8"	2mm	80106-???	Blood alcohols
6.6% Carbowax® 20M	80/120 CarboBlack™ B	2m	1/8"	2mm	80107-???	Alcoholic beverages & fermentation components
0.3% Carbowax® 20M, 0.1% H3PO4	60/80 CarboBlack™ C	0.75m	3/16"	4mm	80111-???	Free acids
4% Carbowax® 20M, 0.8% KOH	60/80 CarboBlack™ B	2m	1/8"	2mm	80116-???	Amines
0.2% Carbowax® 1500	60/80 CarboBlack™ C	2m	1/8"	2mm	80121-???	Alcohols, esters, ketones
0.2% Carbowax® 1500	80/100 CarboBlack™ C	2m	1/8"	2mm	80122-???	Alcohols, esters, ketones
0.1% Rt-1000	80/100 CarboBlack™ C	1.8m	1/8"	2mm	80205-???	Phenols, solvents
1% Rt-1000	60/80 CarboBlack™ B	2.4m	1/8"	2mm	80206-???	EPA 601
1% Rt-1000	60/80 CarboBlack™ B	2m	1/8"	2mm	80207-???	EPA 624
3% Rt-1500	80/120 CarboBlack™ B	3.05m	1/8"	2mm	80211-???	Solvents
1% Rt-1510	60/80 CarboBlack™ B	3.05m	1/8"	2mm	80216-???	Solvents
1.5% XE 60, 1% H3PO4	60/80 CarboBlack™ B	1.8m	1/8"	2mm	80305-???	Sulfur compounds
0.19% Picric Acid	80/100 CarboBlack™ C	2m	1/8"	2mm	80311-???	C4 unsaturates

Instrument Configuration	Restek Part #
General Configuration, fits most GCs	-800
HP 5880, 5890, 5987	-810
Varian 3700, Vista series, FID	-820
Perkin Elmer 900-3920, Sigma 1,2,3	-830

(other configurations available, please call your local distributor)

*Restek's CarboBlack™ B is equivalent to Carbopack™ B and Carbograph™ 1. Restek's CarboBlack™ C is equivalent to Carbopack™ C and Carbograph™ 2.



Packed Column Inlet Adapter Fittings

Many GCs are shipped with an adapter sleeve to allow 1/8" or 3/16" columns to be used in 1/4" injection ports for on-column analysis. This inlet adapter sleeve is bulky and adds mass to the injection port. Restek's low mass inlet adapters fit over the top of a 1/8" or 3/16" Silcosteel® packed column to center the column perfectly in a 1/4" injection port. A slot is positioned at the top of the adapter to prevent carrier gas flow occlusion and a chamfered guide directs the syringe needle to the center of the packed column. The adapter fitting seals at the base of the 1/4" injection port using a 1/4" to 1/8" or 1/4" to 3/16" reducing ferrule (supplied). A reducing ferrule is provided for column installation into the detector.

Packed Column Inlet Adapter Fitting:
(1/4" to 1/8"): cat.# 21651
(1/4" to 3/16"): cat.# 21650

1/4" to 1/8" Reducing Ferrules:
Vespel®/graphite: cat.# 20222, 10-pk.
Graphite: cat.# 20225, 10-pk.

Detection of Antioxidants in Food by Capillary GC

Foods containing fats and oils are prone to lipid oxidation which promotes off-flavors and limits shelf-life. In order to inhibit the oxidation process, food preservatives, or antioxidants, are often added. Antioxidant food additives retard oxidative rancidity caused by the atmosphere and delay discoloration in meats, meat products, and fruits and vegetables. Commonly used antioxidants include phenolic compounds such as BHA(butylated hydroxy anisole), BHT(butylated hydroxy toluene), PG(propyl gallate), and TBHQ(tert-butyl-hydroquinone). Recent attention has focused on "natural" antioxidants such as tocopherols and tocotrienols. These antioxidants possess Vitamin E activity and are currently very popular for their dual function in both food and health preservation.

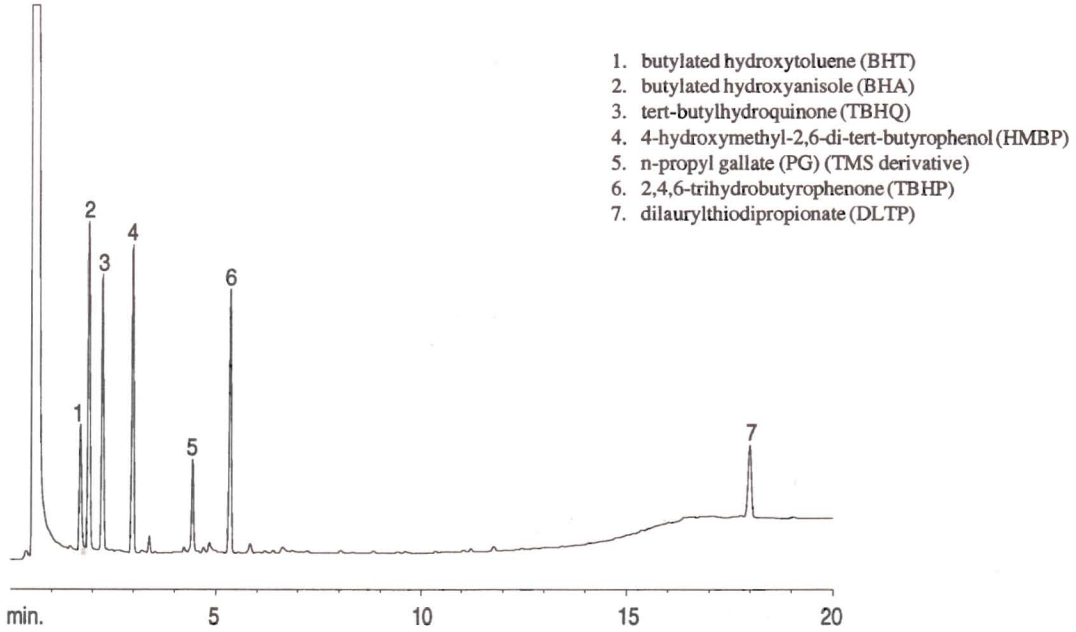
Capillary GC is often used for food antioxidants analysis since capillary columns provide rapid, accurate detection. Methods typically recommend non-polar columns such as the Rtx®-1 or Rtx®-5. However, when analyzing several different antioxidants simultaneously, coelutions can occur when using non-polar columns. By using an intermediate polarity column, co-elution problems can be eliminated and excellent resolution achieved. The selectivity and thermal stability of the Rtx®-50 column provides an ideal choice for the analysis of antioxidants, providing excellent resolution and peak shape.

Synthetic Antioxidants

Primary antioxidants such as BHA, BHT, TBHQ, and PG, terminate the free radical chains susceptible to lipid oxidation. Secondary antioxidants such as DLTP (dilauryl thiopropionate) function by decomposing the lipid hydroperoxides into stable end products.[1] The United States Food and Drug Administration (FDA) has specific regulations on phenolic antioxidant addition, because many, like BHT, are toxic above certain levels. The GRAS (Generally Recognized As Safe) limit for direct addition to food is 0.02% (200ppm), based on the fat content of the food.[1] If added to food packaging, which is an indirect addition to food, the maximum allowable limit is 50ppm or 0.005% in the food item.[1] This limit applies to a maximum concentration allowable for antioxidants used alone or a total concentration for combinations of antioxidants. As mandated by the FDA, antioxidant concentration levels in food samples must be monitored to ensure such regulatory standards.

Several methods have been developed for the analysis of regulated antioxidants in food.[1,2,3] Although analysis of primary or secondary antioxidants can be performed on non-polar columns, simultaneous determination requires the selectivity of a more polar column. Figure 1 shows the analysis of seven regulated antioxidants, including silylated propyl

Figure 1 - The Rtx®-50 capillary column provides rapid analysis of 7 FDA-regulated food antioxidants.



30m, 0.53mm ID, 0.50µm Rtx®-50 (cat.# 10540)
1.0µl direct injection of 7 regulated food antioxidants using a Uniliner®
on-column concentration: 100ppm

Oven temp.:	165°C (hold 1 min.) to 310°C @ 10°C/min. (hold 10 min.)		
Inj./det. temp.:	290°C/310°C	**Detector:**	FID
Carrier gas:	helium	**Linear velocity:**	89cm/sec. set @ 100°C
Detector sensitivity:	8 x 10^{-11} AFS	**Column flow:**	11.7cc/min.

gallate on an Rtx®-50 column. The GRAS limits can be easily achieved on the Rtx®-50 by using the direct injection mode. Baseline resolution of all components is achieved in less than 20 minutes due to the 310°C maximum temperature limit of the Rtx®-50 column.

Tocopherol Antioxidants

Tocopherols are primary antioxidants that quench free radicals created by oxidation of unsaturated bonds in fats.[4] They can be extracted from natural sources such as nuts, seed oils, and soybeans, or created synthetically. Alpha-tocopherol has the most biological potency, however, δ-tocopherols have the most antioxidant potency. Therefore, concentrates used as food antioxidants contain high levels of γ- and δ-tocopherols, and smaller amounts of α-tocopherol.[4] Usage levels of mixed tocopherols range from 0.015% to 0.045% based on oil or fat content.[5] The use of mixed tocopherols as antioxidants are generally regarded as safe by the FDA.[6] Tocopheryl acetate, a stable form of Vitamin E, is also added to food products. Although tocopheryl acetate is not an antioxidant itself, in an acidic environment, it slowly hydrolyzes and tocopherol is released.[4]

Because tocopherols are found in a variety of sample matrices, labs may be required to perform sample preparation including saponification, solvent extraction, and silylation. Tocopherols are commonly analyzed as derivatives, but can also be chromatographed in their free form. Figure 2 shows the analysis of underivatized α-, γ-, and δ-tocopherols, γ-tocotrienol and tocopheryl acetate on a 30 meter, 0.53mm ID, 0.5μm Rtx®-50 column. All components exhibit good peak shape and are baseline resolved in less than 20 minutes.

The Rtx®-50 is an excellent choice for phenolic and tocopherol antioxidants. The inertness and high thermal stability of the Rtx®-50 permits all components to be analyzed, with the exception of propyl gallate, in their free form. All components are well resolved to provide qualitative and quantitative accuracy. The 310°C maximum operating temperature of the Rtx®-50 column reduces analysis time while maintaining a stable baseline during temperature programming.

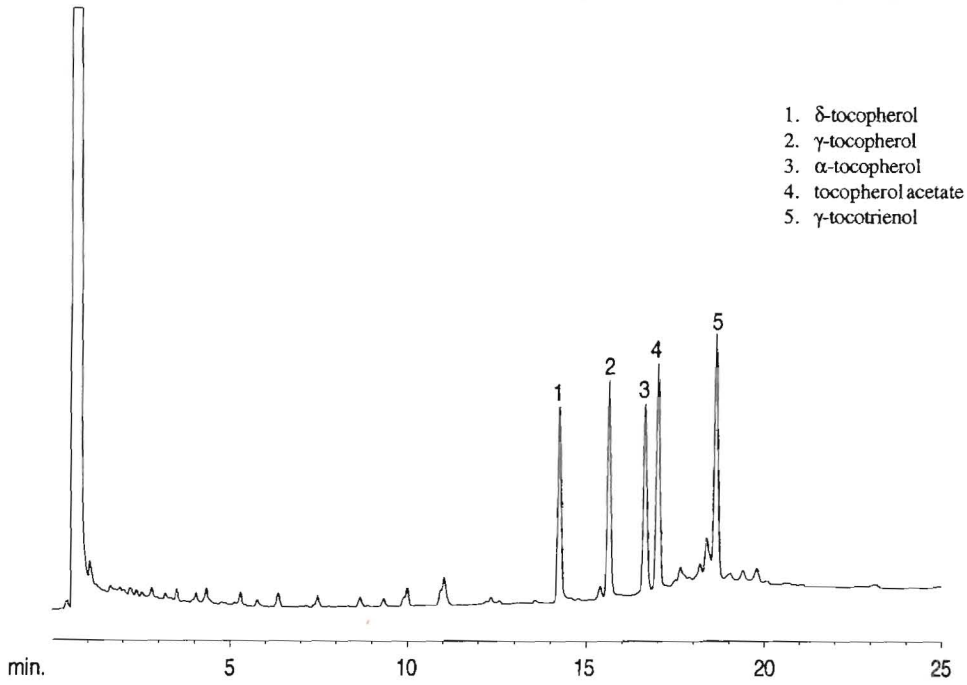
References

1) AOAC, *Food Additives Analytical Manual: Volumes I and II*, 1992.
2) AOAC, *Official Methods of Analysis, Volumes I and II*, 1990.
3) AOCS, *Official Methods and Recommended Practices*, 1994.
4) Hudson, B.F.J., *Food Antioxidants*, 1990.
5) Han, D., O.S. Yi and H.K. Shin, *Journal of Food Science*, Vol. 55, pp.247, 1990.
6) Food and Drug Administration proposal 184.1894, Oct. 27, 1978.

Product Listing

Rtx®-50
30 meter, 0.53mm ID, 0.50μm
cat.# 10540

Figure 2 - Tocopherol antioxidants can be analyzed on the Rtx®-50 capillary column.



30m, 0.53mm ID, 0.50μm Rtx®-50 (cat.# 10540)
1.0μl direct injection of Vitamin E food antioxidants
on-column concentration: 50ppm

Oven temp.:	200°C to 290°C @ 5°C/min. (hold 15 min.)		
Inj./det. temp.:	290°C/310°C	**Detector:**	FID
Carrier gas:	helium	**Linear velocity:**	89cm/sec. set @ 100°C
Detector sensitivity:	8×10^{-11} AFS	**Column flow:**	11.7cc/min.

Aromatics in Reformulated Gasoline by GC/MS

The US Clean Air Act amendments of 1990 require that after 1994 only reformulated gasoline be sold in certain areas which have not attained specified ozone levels. The reformulation of gasoline is designed to reduce air pollution by lowering the emission of volatile organic compounds (VOCs), nitrogen oxides (NO_x) and toxic compounds. In order to meet the reduction of air pollution, a new set of specifications[1,2] will go into effect on January 1, 1995 setting limits on the volatility and composition of gasoline. As part of these new regulations the Environmental Protection Agency (EPA) has specified that GC/MS be used for the determination of total aromatics in gasoline. In addition, this test method can also be used to measure the volume % of benzene as an alternative to ASTM test method D3606.

In order to meet the EPA requirements, the American Society of Testing and Materials Committee on Petroleum Products and Lubricants (ASTM D2) is developing a standard test method, which is currently under evaluation by an intralaboratory cooperative test program. In addition to a GC/MS and column, the procedure requires specific mixtures of aromatic hydrocarbons for qualitative and quantitative calibration of the system prior to sample analysis. It would be time consuming and expensive if each laboratory had to obtain the 28 individual hydrocarbons, perform purity analyses on each, and prepare two sets of the 5 calibration levels. Restek now offers kits containing the aromatic standards necessary for calibration using the proposed ASTM method. These standards undergo extensive quality control to ensure the purity of the starting materials and the accuracy of the final mixtures.

Peaks are Integrated Using Ions Specific for Aromatic Hydrocarbons

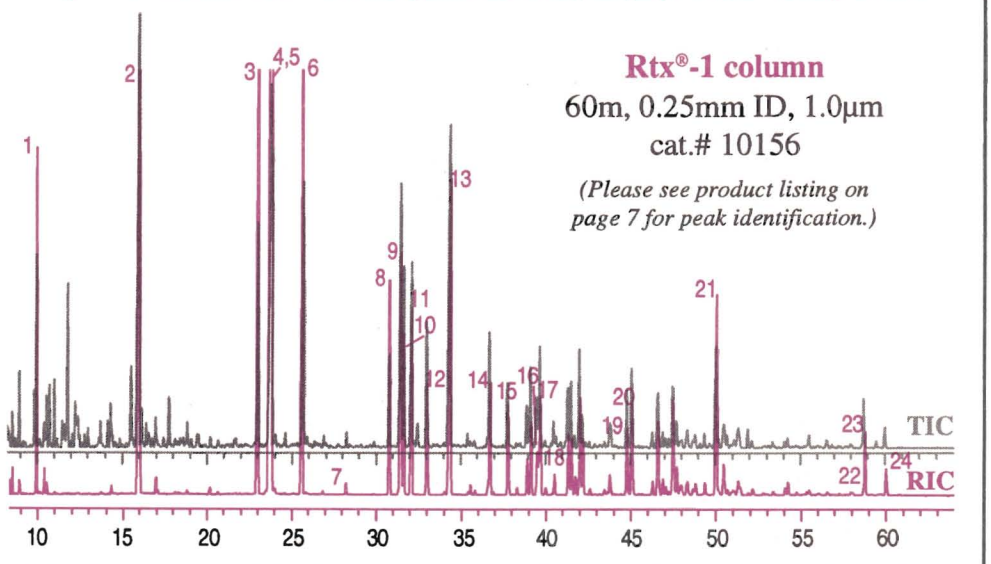
The ASTM proposed method specifies the analysis of gasoline using a 60 meter methyl silicone capillary column and a quadrapole GC/MS system operated in the scan mode from 45-300 dalton. Either a split injection with direct interface to the GC/MS or a cool on-column injection with an open split interface may be used. For the purpose of this study, the split injector was selected with a 60m, 0.25mm ID, 1.0µm Rtx®-1 column. Figure 1 was obtained from the analysis of a gasoline sample, showing both the total ion chromatogram (TIC) and a reconstructed chromatogram (RIC) with specific ions for aromatics extracted. The 60 meter column provides resolution of the aromatic hydrocarbons in the complex gasoline sample. Aromatics are identified, with the aid of the mass spectra, and appropriate response factors are applied. The Mass % of each aromatic detected is calculated using the internal standard technique and the individual amounts are summed to report a total aromatic concentration.

Pure Calibration Materials for Quantitative Accuracy

Since the proposed test method will be used by refineries and regulators to determine the aromatic concentration in gasoline, it is crucial that the method be accurate. Response factor calibration, using an accurate standard, is a critical part of the analytical procedure. However, before a calibration mixture can be prepared, the chemical purity of the aromatic components must be determined. Restek uses several complimentary analytical techniques including gas chromatography with flame ionization detection (GC/FID), GC/MS, refractive index (RI), melting point (Tmp) and differential scanning calorimetry (DSC) to determine the identity and purity of raw materials used in standards preparation.

Gas chromatography is a valuable tool for calculating an aromatic compound's purity, but only if the column and conditions are optimized to resolve possible organic impurities. All volatile hydrocarbons in Restek's reformulated gasoline standard mixes are analyzed using a 105m, 0.53mm ID, 3.0µm Rtx®-502.2 column (cat.# 10910), which has good selectivity for resolving aromatics and high capacity for the major component. A second GC/FID analysis for substituted benzenes is also performed using a more polar Stabilwax® column which gives better resolution of possible coeluting isomers. GC/MS analysis is also done on all raw materials to confirm identity in case of mislabeling.

Figure 1 - Extracted ion chromatogram selectively displays aromatics in gasoline.



1.0µl split injection of neat gasoline
Oven temp.: 50°C (hold 1 min.) to 190°C @ 2°C/min.
Inj. temp.: 250°C **Det. temp.:** 280°C
Linear velocity: 35cm/sec. set @ 50°C
Split ratio: 200:1
Mass Spec: 45-300 m/e, 1 scan/sec.

1. 40 CFR Part 80, *Federal Register*, 59(32):7716-7878, Feb.16, 1994.
2. W.H. Keesom, M.J. Humbach; "Effective Gasoline Reformulation", *Standardization News*, 22 (6) June 1994.

Refractive index provides a complementary qualitative technique and is especially informative when combined with GC/MS and DSC. The melting point of an organic compound provides another qualitative analysis in addition to RI and MS but most importantly the presence of water and inorganic impurities can be determined[3]. DSC provides a very powerful tool for purity analysis because it measures impurities which are not detectable by GC. DSC analysis was particularly helpful for the analysis of 1,2-diethyl benzene containing an impurity of 1,4-diethyl benzene. These compounds have boiling points which differ by only 0.4°C and are therefore difficult to purify, but the melting points differ by 12°C. In this case, DSC provides a better qualitative identification than GC/MS, because the isomers have similar spectra and retention times. All of these individual tests are important quality control checks on the raw materials because it is very difficult to determine the purity of the mixture after the standard has been prepared.

Construct a 5 Point Calibration Curve with Convenient Standards Kits

Two sets of calibration blends are available containing the aromatics specified in the ASTM proposed test method. The appropriate internal standards are included in the mixtures for calculation of relative response factors. Each kit contains five ampuls of standards at the correct concentration to construct a five point calibration curve and complete the calibration of the GC/MS system. A data pack containing complete quality assurance documentation is available for each of these standards. The data pack includes the raw material purities by each of the tests mentioned, gravimetric amounts determined by NIST traceable balance, and quantitative verification by GC/MS. In addition to these kits, individual standards at each concentration level are also available.

3. ASTM Test Method E-928 "Mol Percent Purity by Differential Scanning Calorimetry" *ASTM Book of Standards* Vol14.02.

ASTM/EPA Aromatics Standards Product Listing

Peak #	Compound	Mix #1	Mix #2	Mix #3	Mix #4	Mix #5
1	benzene	30mg/ml	15mg/ml	10mg/ml	5mg/ml	1mg/ml
2	toluene	150	100	50	30	10
3	ethylbenzene	50	25	10	5	1
4	*m*-xylene	60	30	10	5	1
5	*p*-xylene	60	30	10	5	1
6	*o*-xylene	60	30	10	5	1
IS1	benzene-d6	20	20	20	20	20
IS2	ethylbenzene-d10	20	20	20	20	20
	Cat. #					
	single ampul	36200	36201	36202	36203	36204
	single ampul w/dp	36200-500	36201-500	36202-500	36203-500	36204-500
	5-pack	36200-510	36201-510	36202-510	36203-510	36204-510
	5-pack w/dp	36200-520	36201-520	36202-520	36203-520	36204-520
	10-pack w/dp	36300	36301	36302	36303	36304

Complete ASTM/EPA Aromatics Set #1 Kit (1ml each of above 5 mixes): 36205, per kit 36205-500, per kit with data pack

Peak #	Compound	Mix #1	Mix #2	Mix #3	Mix #4	Mix #5
7	isopropylbenzene	30	15	10	5	1
8	n-propylbenzene	30	15	10	5	1
9	1-methyl-3-ethylbenzene	30	15	10	5	1
10	1-methyl-4-ethylbenzene	30	15	10	5	1
11	1,3,5-trimethylbenzene	30	15	10	5	1
12	1-methyl-2-ethylbenzene	30	15	10	5	1
13	1,2,4-trimethylbenzene	50	25	10	5	1
14	1,2,3-trimethylbenzene	30	15	10	5	1
15	indan	30	15	10	5	1
16	1,4-diethylbenzene	30	15	10	5	1
17	butylbenzene	30	15	10	5	1
18	1,2-diethylbenzene	30	15	10	5	1
19	1,2,4,5-tetramethylbenzene	20	10	5	2.5	1
20	1,2,3,5-tetramethylbenzene	20	10	5	2.5	1
21	pentamethylbenzene	20	10	5	2.5	1
22	naphthalene	20	10	5	2.5	1
23	2-methylnaphthalene	20	10	5	2.5	1
24	1-methylnaphthalene	20	10	5	2.5	1
IS2	ethylbenzene-d10	20	20	20	20	20
IS3	naphthalene-d8	10	10	10	10	10
	Cat. #					
	single ampul	36206	36207	36208	36209	36210
	single ampul w/dp	36206-500	36207-500	36208-500	36209-500	36210-500
	5-pack	36206-510	36207-510	36208-510	36209-510	36210-510
	5-pack w/dp	36206-520	36207-520	36208-520	36209-520	36210-520
	10-pack w/dp	36306	36307	36308	36309	36310

Complete ASTM/EPA Aromatics Set #2 Kit (1ml each of above 5 mixes): 36211, per kit 36211-500, per kit with data pack

Standards Spotlight

XHc Underground Storage Tank Monitoring Standards and JP-8 Military Fuel Standards

• Suitable for matrix spiking & recovery studies • Highest concentration commercially available • Full data pack for audit compliance • Two convenient volumes for every product • Produced under an ISO 9001 registered quality system •

Restek is pleased to introduce the highest concentration underground storage tank monitoring standards available today - our XHc (eXtra High Concentration) product line. These standards are prepared at a concentration of 50,000µg/ml in a solvent suitable for the type of analysis being performed.

Each standard is packaged in either 1ml or 5ml volumes. The mixtures are placed in an amber ampul, purged with nitrogen, and flame sealed for extended shelf life. The standards are shipped with an ampul breaker and extra label for your convenience. In addition, the 1ml ampuls are that supplied are silane treated and come with a screw cap vial for storage of any unused standard.

All the products are available with a full data pack for audit compliance. Each data pack contains detailed information on product manufacture and testing to easily pass the most stringent quality audit a laboratory might encounter. Our customer choice packaging offers either a 10% discount or a FREE data pack for 5-pack purchases, and a 10% discount PLUS FREE data pack for 10-pack purchases.

XHc Unleaded Gasoline Composite Standard

50,000µg/ml in purge & trap grade methanol.

Packaged 1ml per ampul:

cat.# 30205	each
30205-500	each w/data pack
30205-510	5-pack
30205-520	5-pk. w/data pack
30305	10-pk. w/data pack

Packaged 5ml per ampul:

cat.# 30206	each
30206-500	each w/data pack
30206-510	5-pack
30206-520	5-pack w/data pack
30306	10-pack w/data pack

XHc Aviation Gas Standard

50,000µg/ml in purge & trap grade methanol.

Packaged 1ml per ampul:

cat.# 30207	each
30207-500	each w/data pack
30207-510	5-pack
30207-520	5-pack w/data pack
30307	10-pack w/data pack

Packaged 5ml per ampul:

cat.# 30208	each
30208-500	each w/data pack
30208-510	5-pack
30208-520	5-pack w/data pack
30308	10-pack w/data pack

XHc Jet Fuel A Standard

50,000µg/ml in methylene chloride.

Packaged 1ml per ampul:

cat.# 31242	each
31242-500	each w/data pack
31242-510	5-pack
31242-520	5-pack w/data pack
31342	10-pack w/data pack

Packaged 5ml per ampul:

cat.# 31243	each
31243-500	each w/data pack
31243-510	5-pack
31243-520	5-pack w/data pack
31343	10-pack w/data pack

XHc Fuel Oil #4 Standard

50,000µg/ml in methylene chloride

Packaged 1ml per ampul:

cat.# 31244	each
31244-500	each w/data pack
31244-510	5-pack
31244-520	5-pack w/data pack
31344	10-pack w/data pack

Packaged 5ml per ampul:

cat.# 31245	each
31245-500	each w/data pack
31245-510	5-pack
31245-520	5-pack w/data pack
31345	10-pack w/data pack

XHc Fuel Oil #5 Standard

50,000µg/ml in methylene chloride

Packaged 1ml per ampul:

cat.# 31246	each
31246-500	each w/data pack
31246-510	5-pack
31246-520	5-pack w/data pack
31346	10-pack w/data pack

Packaged 5ml per ampul:

cat.# 31247	each
31247-500	each w/data pack
31247-510	5-pack
31247-520	5-pack w/data pack
31347	10-pack w/data pack

XHc Fuel Oil #6 Standard

50,000µg/ml in methylene chloride

Packaged 1ml per ampul:

cat.# 31248	each
31248-500	each w/data pack
31248-510	5-pack
31248-520	5-pack w/data pack
31348	10-pack w/data pack

Packaged 5ml per ampul:

cat.# 31249	each
31249-500	each w/data pack
31249-510	5-pack
31249-520	5-pack w/data pack
31349	10-pack w/data pack

XHc JP-4 Standard

50,000µg/ml in methylene chloride

Packaged 1ml per ampul:

cat.# 31250	each
31250-500	each w/data pack
31250-510	5-pack
31250-520	5-pack w/data pack
31350	10-pack w/data pack

Packaged 5ml per ampul:

cat.# 31251	each
31251-500	each w/data pack
31251-510	5-pack
31251-520	5-pack w/data pack
31351	10-pack w/data pack

XHc JP-5 Standard

50,000µg/ml in methylene chloride

Packaged 1ml per ampul:

cat.# 31252	each
31252-500	each w/data pack
31252-510	5-pack
31252-520	5-pack w/data pack
31352	10-pack w/data pack

Packaged 5ml per ampul:

cat.# 31253	each
31253-500	each w/data pack
31253-510	5-pack
31253-520	5-pack w/data pack
31353	10-pack w/data pack

XHc JP-8 Standard

50,000µg/ml in methylene chloride

Packaged 1ml per ampul:

cat.# 31254	each
31254-500	each w/data pack
31254-510	5-pack
31254-520	5-pack w/data pack
31354	10-pack w/data pack

XHc JP-8 Standard (cont.)

Packaged 5ml per ampul:

cat.# 31255	each
31255-500	each w/data pack
31255-510	5-pack
31255-520	5-pack w/data pack
31355	10-pack w/data pack

XHc Kerosene Composite Standard

50,000µg/ml in methylene chloride

Packaged 1ml per ampul:

cat.# 31256	each
31256-500	each w/data pack
31256-510	5-pack
31256-520	5-pack w/data pack
31356	10-pack w/data pack

Packaged 5ml per ampul:

cat.# 31257	each
31257-500	each w/data pack
31257-510	5-pack
31257-520	5-pack w/data pack
31357	10-pack w/data pack

XHc Diesel Fuel #2 Composite Standard

50,000µg/ml in methylene chloride

Packaged 1ml per ampul:

cat.# 31258	each
31258-500	each w/data pack
31258-510	5-pack
31258-520	5-pack w/data pack
31358	10-pack w/data pack

Packaged 5ml per ampul:

cat.# 31259	each
31259-500	each w/data pack
31259-510	5-pack
31259-520	5-pack w/data pack
31359	10/pack w/data pack

XHc Mineral Spirits Standard

50,000µg/ml in methylene chloride

Packaged 1ml per ampul:

cat.# 31260	each
31260-500	each w/data pack
31260-510	5-pack
31260-520	5-pack w/data pack
31360	10-pack w/data pack

Packaged 5ml per ampul:

cat.# 31261	each
31261-500	each w/data pack
31261-510	5-pack
31261-520	5-pack w/data pack
31361	10-pack w/data pack

JP-8 Standard

5000µg/ml in 1ml methylene chloride, 1ml per ampul.

cat.# 31262	each
31262-500	each w/data pack
31262-510	5-pack
31262-520	5-pack w/data pack
31362	10-pack w/data pack

Restek chemical standards are produced under an ISO 9001 registered quality system!

Additional EPA Drinking Water Monitoring Standards

The EPA is now requiring laboratories to monitor and report three additional compounds in drinking water. These compounds are: methylene chloride, 1,1,2-trichloroethane, and 1,2,4-trichlorobenzene. To meet these requirements, Restek is offering an additional calibration mixture and revised kit for drinking water analysis. See Restek's *Chromatography Products 1994/95* catalog (page 43) for a complete description of the DW-VOC Mix #1 & #2, plus Trihalomethane Mix.

DW-VOC Mix #3

200µg/ml each in purge & trap grade methanol, 1ml per ampul.

methylene chloride
1,1,2-trichloroethane
1,2,4-trichlorobenzene

cat.# 30209	each
30209-500	each w/data pack
30209-510	5-pack
30209-520	5-pack w/data pack
30309	10-pack w/data pack

Revised DW-VOC Kit

Contains 1ml each of these products:

501 Trihalomethane Mix (cat.# 30036)
DW-VOC Mix #1 (cat.# 30037)
DW-VOC Mix #2 (cat.# 30038)
DW-VOC Mix #3 (cat.# 30209)

cat.# 30210	each kit
30210-500	each kit w/data pack

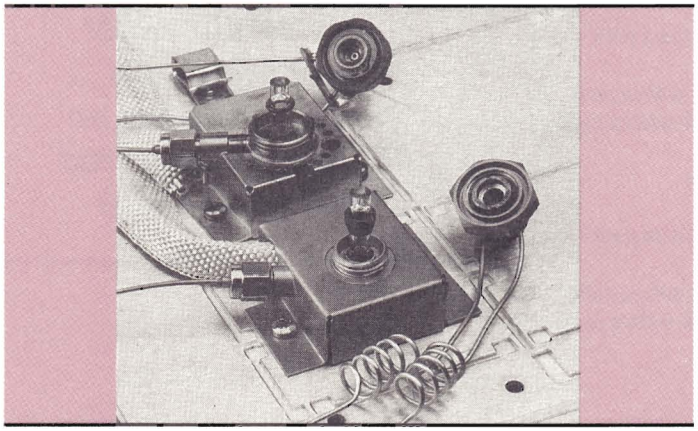
New - Restek's Split/Splitless Injection Port for HP 5890 GCs

After many years of using HP 5890 GCs in our Applications, R&D, and Quality Assurance labs and getting feedback from our customers, Restek has re-engineered the HP split/splitless injector. Our new injector offers several features not available with the original design.

Improved Split/splitless Weldment Assembly

- Split/splitless weldment re-design allows the use of 1/4" type graphite or Vespel®/graphite ferrules instead of rubber or graphite o-rings.*
- Locking pin and slot assembly eliminates the need for a metal bracket to prevent the inlet lines from snapping.
- Narrow weldment recess prevents trapped septa particles.

Figure 1 - Restek's injection port seals the sleeve tightly with a Swagelok®-type 1/4" ferrule eliminating rubber o-ring melt down.



The o-ring that seals the sleeve in the top of the injector is a potential source for leaks. Although rubber o-rings seal well at temperatures below 200°C, they can become brittle and leak when operated for prolonged periods above 200°C. Graphite o-rings can handle higher temperatures, but do not seal as easily as rubber o-rings. We experimented with various designs and determined that a 1/4" type ferrule, installed tapered end down, gave the most consistent, leak-free results. Our new design accepts either Vespel® or graphite ferrules and delivers a leak-free seal, even at injection port temperatures as high as 400°C. The photo in Figure 1 illustrates the taper inside the injection port weldment utilizing standard ferrules to ensure a leak tight seal.*

The original equipment incorporates a metal bracket that locks into a slot on the top of the injection port. This design prevents the assembly from spinning and breaking off the inlet gas lines. Our new design incorporates a pin and slot assembly that prevents the split/splitless weldment from spinning and eliminates the bulky metal bracket assembly. The original split/splitless weldment incorporates a multi-piece assembly that permits a large recess between the top and bottom orifice. Septum particles can be trapped in this recess, causing ghost peaks and baseline disturbances. Restek's new design significantly reduces this recess and eliminates trapped septa particles.

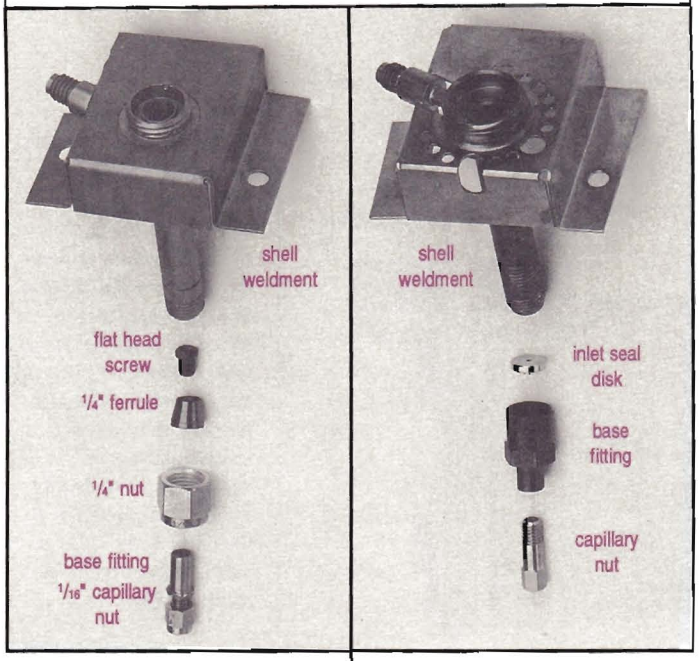
Re-designed Injector Base Improves Seal and Simplifies Column Installation

- Base inlet sealing disk eliminated and replaced with a standard 1/4" ferrule.
- Sleeve supported with an easy to remove, inexpensive slotted flat head stainless steel screw.
- Base fitting designed to use standard 1/16" Swagelok®-type ferrules.
- Base fitting protrudes from the insulated cup making column installation easier.
- Original sleeve dimensions and column insertion distances maintained (from back of nut).

In the original equipment injector design, the sleeve is supported in the injector by a metal inlet seal that sits inside of the injector fitting. When the injector nut is tightened, a knife edge at the base of the injection port cuts into the metal inlet seal and creates a seal by compressing metal against metal. The inlet seals require frequent replacement due to sample contamination and cannot be re-used, otherwise a leak will occur. Restek has re-designed the base of the injection port to accept a Swagelok®-type ferrule to seal the injector fitting to the injector shell weldment. An inexpensive slotted, stainless steel flat head screw fits into the base fitting, supporting the sleeve in the injection port and maintaining the same flow path as the original design. This screw can easily be cleaned and re-installed without fear of leakage and is inexpensive enough to replace when severely contaminated. Both gold plated and Silcosteel® versions are available to minimize decomposition of active compounds. Figure 2 shows a comparison between the original equipment design and Restek's design. Restek's design eliminates the inlet seal while maintaining the original column insertion distances from the back of HP's standard capillary inlet nut. The injector fitting is designed to seal with standard 1/16" graphite or Vespel® capillary ferrules with the threads protruding from the insulated cover. This allows visual confirmation of column installation.

**Standard 1/4" ID graphite and Vespel®/graphite ferrules fit over split sleeves (OD ~ 6.35mm) but a slightly enlarged ferrule ID is required for splitless sleeves (OD 6.5mm) and is available from Restek.*

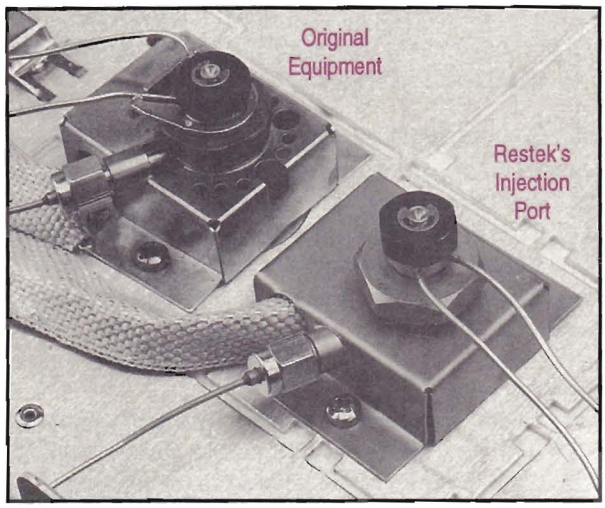
Figure 2 - The original equipment design requires a metal inlet seal which presses against a knife edge at the base of the injector shell weldment. Restek's design seals the inlet base fitting with a standard 1/4" Swagelok®-type ferrule and utilizes an inexpensive flat head machine screw to achieve the same flow profile at the base of the injector.



Restek's Injection Port | *Original Equipment*

Restek's Split/Splitless Injection Port for HP 5890 GCs:

- Swagelok®-type ferrules seal around sleeve.
- Narrower needle gap eliminates trapped septum particles.
- Locking pin guide.
- Standard 1/4" ferrule used instead of sealing disk.
- Easily removable and re-useable slotted base screw utilized to maintain flow profile.
- Original sleeve dimensions and column insertion distances maintained (26mm from back of nut).



Product Listing

Product	Quantity	Cat.#
Complete Injection Port Assembly includes*: base fitting, split/splitless weldment, shell weldment, stainless steel base screw, septum nut, 1/16" and 1/4" stainless steel nuts, 1/4" graphite ferrules		
Injection Port Kit for HP 5890 GCs	kit	21625
Ferrules for split sleeves (6.35mm OD):		
1/4" graphite ferrules	10-pk.	20210
1/4" Vespel®/graphite ferrules	10-pk.	20221
Ferrules for splitless sleeves (6.5mm OD):		
6.55mm ID Graphite Splitless Ferrules (1/4" type)	10-pk.	20260
6.55mm ID Vespel®/Graphite Splitless Ferrule (1/4" type)	10-pk.	20261
Replacement Parts		
Flat head base screws:		
S.S. Base Screws for Restek 5890 Injection Port	10-pk.	21633
	50-pk.	21634
Silcosteel® Base Screws for Restek 5890 Injection Port	10-pk.	21631
	50-pk.	21632
Gold-plated Base Screws for Restek 5890 Injection Port	2-pk.	21629
	10-pk	21630
Needle Guide Septa Nut / 26 gauge	each	21309
Base Fitting for Restek 5890 Injection Port	each	21626
Split/Splitless Weldment for Restek 5890 Injection Port	each	21627
Shell Weldment for Restek 5890 Injection Port	each	21628

**Does not include inlet sleeve, 1/16" type capillary ferrule, or split/splitless sleeve ferrules. Order separately.*

Hints for the Capillary Chromatographer

Using Flame Ionization Detectors

The flame ionization detector (FID) is the most widely used GC detector. The FID is a destructive, mass flow dependent detector that utilizes a flame source to ionize compounds. Compounds responding to FIDs include all organic compounds that ionize in a hydrogen and air flame. Many of these compounds are found in petrochemical, industrial, environmental, clinical, and flavor and fragrance analyses.

The FID is considered a universal detector since it responds to a wide variety of organic compounds. However, carbon dioxide, carbon monoxide, nitrogen, oxygen, carbon disulfide, and inert gases produce little or no response by FID. Formaldehyde, formic acid and heavily halogenated compounds produce a low or non-linear response, because of the few carbon/hydrogen bonds present.

Detector Operation

A signal for an FID is produced when a sample combusts in a hydrogen/air flame and ionizes (Figure 1). Because the FID is a mass sensitive detector, it will respond to the amount of material passing through the flame at a given time. The response of a component is directly proportional to the amount injected.

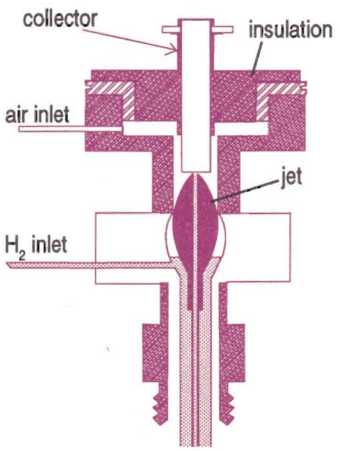
FIDs can detect concentrations of saturated compounds as low as 20pg. However, FIDs are not practical for quantitating compounds below 0.1ng because baseline noise from air movement around the collector, electrical noise, gas contaminants, and column bleed reduces FID sensitivity (signal/noise ratio). In addition, the presence of compounds with minimal carbon/hydrogen bonds (i.e., carbonyl, alcohols, halogens, amines, etc.) also make detectability below 0.1ng difficult. When an increase in concentration no longer produces an increase in response, the detector has reached its maximum linear range. The FID has the largest linear range of all the common GC detectors, 10^7 times the minimum detectable level of a hydrocarbon.

FID jet orifices range from 0.011" to 0.030". In general, the smaller the jet orifice, the more sensitive an FID. Internal diameters of 0.011" are used for narrow bore capillary columns (< 0.25mm ID) when high sensitivity is desired. However, smaller orifices are prone to plugging and are impractical for packed columns or thick film capillary columns. Some packed columns with high phase loadings require a 0.030" jet. In most cases, jet orifices of 0.018" are used.

Detector Gases

FID gas flow rates are generally optimized at 30cc/min. hydrogen, 30cc/min. carrier plus make-up gas, and 300cc/min. air.

Figure 1 - Flame Ionization Detector Design



Hydrogen gas is heated as it flows around the side of the jet and is mixed with the make-up gas and sample effluent exiting from the column. The combined gases travel through the jet tail and combust as they exit the jet orifice. Because there is an applied voltage across the jet, a current is produced by the ionized particles as they reach the collector and is measured by an electrometer.

Sensitivity can be increased by decreasing the hydrogen flow rate relative to the carrier and make-up gas. Usually, a 1:1.25 ratio of hydrogen to carrier gas (25cc/min H_2, 30cc/min carrier + make-up) increases sensitivity. However, at lower hydrogen flow rates, the flame is prone to extinguishing as large volumes (>2µl) of water elute. A higher than optimum hydrogen flow rate is usually necessary when using water as a solvent to ensure the flame remains lit at all times. Increasing the air flow rate above 300cc/min. has little effect on FID sensitivity. However, increasing the air flow rate to 400-450 ml/min. will extend the linearity of the FID when high concentrations of analytes are injected. Since there are variations in detector design, consult your instrument manual for optimum gas settings.

Carrier, hydrogen, and air should be of high purity or the FID background noise will increase. Excessive moisture in the hydrogen or air is particularly a problem. Make-up gas is frequently recommended with capillary columns to optimize detector performance, reduce dead volume, and minimize band broadening. Without make-up gas to augment the typical 5cc/min. carrier gas flow rate of a capillary column, the flame burns rich which results in increased noise and non-linearity.

Detector Maintenance

FIDs can become contaminated from stationary phase bleed and sample residue. Most stationary phase bleed consists of low molecular weight silicone polymer fragments that form a white silica powder when burned in the flame. This contamination can deposit on the jet tip and collector increasing background

noise. In cases of severe contamination, noise spikes occur as the silica particles are carried into the flame. Other stationary phases such as Carbowax® and high molecular weight sample contaminants form a residue that coats the detector surfaces. As the coating builds, noise increases and sensitivity diminishes.

Figure 2 shows a chromatogram obtained before and after a new jet was installed. In the first chromatogram, active compounds such as benzoic acid and 2,4-dinitrophenol (peaks 3 & 5) are tailing and produce a reduced response. By installing a new jet, peak shape and response could be restored. The problem was identified as stationary phase contamination from a Carbowax® column that was previously installed in the GC. Because the Carbowax® column has a lower maximum temperature than an XTI®-5 column (250°C vs. 360°C), the stationary phase decomposed when the detector temperature was increased to elute higher molecular weight compounds.

In addition to stationary phase bleed and sample residue, glass fragments or short pieces of fused silica tubing can break off and become lodged in the small jet orifice causing peak tailing or adsorption. This can occur during installation as the column is inserted into the jet and pushed as close to the orifice as possible. Often, excessive tailing solvent or sinusoidal baseline signals indicate a fused silica fragment lodged in the jet.

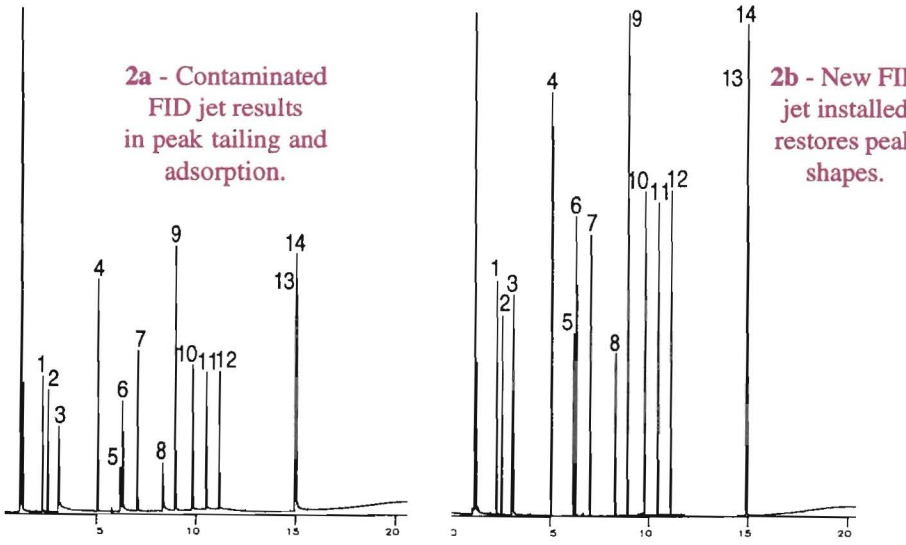
Inexpensive FID cleaning kits are available from most chromatography suppliers and contain jet reamers, tube brushes, and emery cloth to make cleaning easy. Avoid using kits with brass brushes which leave metal fragments inside the FID. To clean an FID, cool the detector, disassemble the collector assembly, and remove the jet. It is important to inspect the FID base for particles by shining a flashlight into the oven and through the fitting until the light exits the top of the FID where the jet is attached. Rotate the light to look for particles or contamination that might be on the FID base wall. Clean the detector base with a 1/4" brush. Next clean the jet orifice with a jet reamer and a stainless steel toothbrush. Shine a flashlight through the jet bore and look for dull spots or particles lodged in the bore. Clean the jet in an ultrasonic bath with solvents such as methylene chloride to remove the contamination. Discard the jet if it can not be cleaned. Finally, clean the electrometer metal contacts and spring clips with emery cloth to ensure a noise free signal. Wipe off the metal dust by using a Kimwipes® or cloth moistened with solvent. Reassemble and allow the detector temperature to reach 150°C before installing a column or lighting the flame.

FID Operating Hints

The FID temperature should always be operated above the final oven temperature to prevent stationary phase or sample components from condensing in the detector. Since the column is installed in the detector's heated zone, never heat the FID above the stationary phases maximum operating temperature or decomposition and excessive bleed will occur. Never set an FID temperature below 100°C or water vapor from the flame will condense in the detector. Always install the capillary column as close to the jet orifice as possible to prevent sample adsorption or decomposition on the metal surfaces of the jet. Inert Silcosteel® FID jets should be used with active samples to prevent adsorption or decomposition. In addition, ceramic jets for high temperature applications are used by some instrument companies for MXT® or fused silica lined metal capillary columns to prevent gounding of the electrical signal.

Due to its ease of use and nearly universal response to most organic compounds, the FID has become the most commonly used GC detector. Proper care and routine maintenance is required to keep an FID working at maximum sensitivity with minimum background noise.

Figure 2 - Peak tailing and adsorption occur when sample residue or stationary phase bleed deposits in the FID jet tail and interacts with the sample stream.



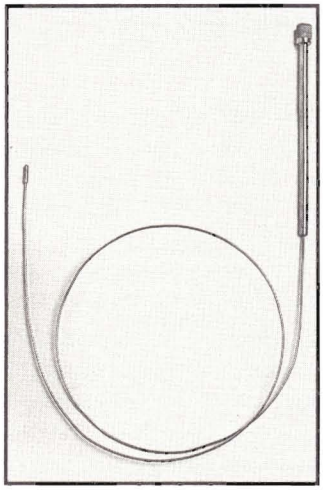
1. 1,2-hexanediol
2. nitro-di-nipropylamine
3. benzoic acid
4. C14
5. 2,4-dinitrophenol
6. nitrophenol
7. nitroaniline
8. pentachlorophenol
9. carbazole
10. C20
11. C21
12. C22
13. benzo(*b*)fluoranthene
14. benzo(*k*)fluoranthene

30m, 0.25mm ID, 0.50µm XTI®-5 (cat.# 12238)
1.0µl split injection. 7-17ng/compound on-column.

Oven temp.:	100°C to 300°C @ 6°C/min., then to 360°C @ 15°C/min. (hold 15 min.)
Inj. & det. temp.:	360°C
Carrier gas:	hydrogen
Linear velocity:	40cm/sec. set @ 100°C
FID sensitivity:	8×10^{-11} AFS
Split vent:	40:1

Peak Performers

Replacement Chemical Trap for HP 5890 GCs



GC carrier gas line contamination is a difficult problem to diagnose and correct. Carrier gas line contamination can occur from carrier gas impurities or by sample backflash. Contamination can appear as ghost peaks or a rising baseline not attributed to the inlet, column, or detector. HP 5890 GCs incorporate a small chemical filter in the carrier gas line to reduce the possibility of contamination. However, the trap capacity is low and must be periodically changed or contamination will occur. It is recommended the traps be replaced annually under normal operating conditions or more frequently under heavy usage.

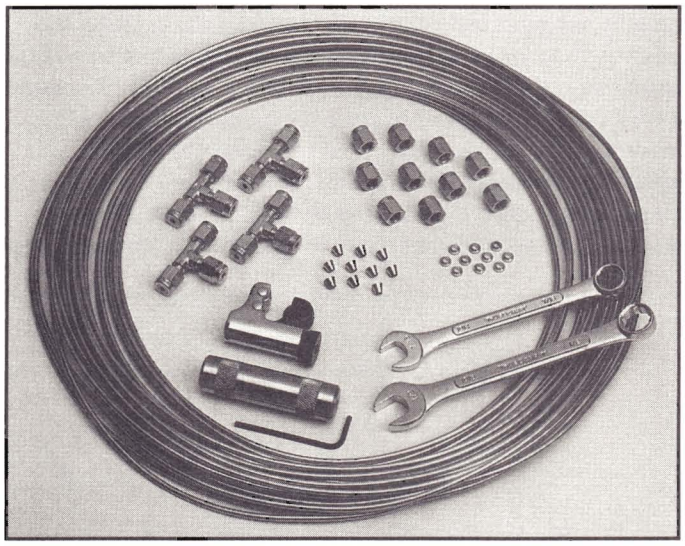
Restek's chemical trap is easy to install and attaches to the same fittings as HP's chemical trap. It incorporates built-in frits and adsorbents to remove both moisture and hydrocarbons. Additionally, Restek's trap can be regenerated to remove trapped contaminants and restored to its original performance.

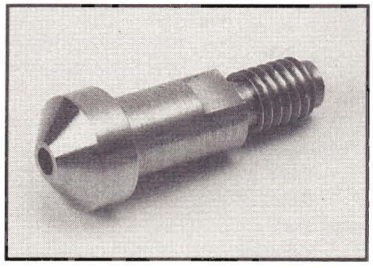
Replacement Chemical Trap for HP 5890 GCs
cat.# 21610, each

GC Installation Kit

Now, a GC Installation kit to make your life easier! This kit contains the necessary tubing and fittings to add an additional GC to your lab bench. Also included in the kit are four 1/8" tees, so GC gases like hydrogen, helium, nitrogen, and air can be routed to the new inlet or detector from the existing gas lines. Additional parts, such as purifiers or regulators, may be ordered separately to customize the GC installation to your labs specifications.

Kit includes: one Imp™ tubing cutter, one 1/8" x 1/4" reamer, one 7/16" wrench, one 1/2" wrench, four 1/8" brass tees, ten 1/8" brass nuts, ten brass front and back ferrules, and 50' of pre-cleaned 1/8" copper tubing.
GC Installation Kit: cat.# 21325





Injector Nut for Shimadzu 17A GCs

Uses standard 1/16" Swagelok®-type ferrules

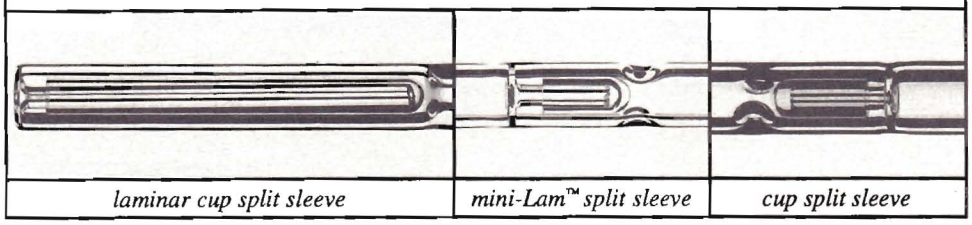
Restek has developed a special inlet fitting for Shimadzu 17A GCs that allows the use of standard 1/16" Swagelok®-type ferrules. Either Vespel® or graphite type ferrules can be used. (Similar to Shimadzu part# 221-32509.)
cat.# 21895, kit

mini-Lam™ Split Sleeve for HP 5890 GCs

Same benefits as the laminar cup at a lower price!

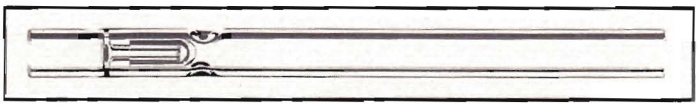
Grob[1] has published results showing the laminar cup splitter as the best split inlet sleeve to prevent un-vaporized sample from reaching the column. Injection volumes as large as 5µl can be made without having un-vaporized sample reach the column. Un-vaporized sample will result in molecular weight discrimination or non-linear response. Grob has determined that the narrow channels between the laminar cup and sleeve wall are responsible for enhanced evaporation since the gap at the bottom of the laminar cup traps the liquid sample until it is fully vaporized. Other sleeve designs are not as effective in completely vaporizing the sample, even with volumes as small as 2µl.

Figure 1 - The *mini*-Lam™ split sleeve offers the benefits of a laminar cup sleeve (complete sample vaporization) while reducing trapped septa particles and sample residue common with a standard inverted cup split sleeve.



laminar cup split sleeve	*mini-Lam™ split sleeve*	*cup split sleeve*

Although the laminar cup provides the best sample vaporization and minimizes discrimination, it is costly to manufacture. The complexity of the sleeve design and precision required to seal several glass chambers at the outlet of the sleeve (Figure 1) greatly increases the price when compared to a simple splitless sleeve. Restek has recently developed a new sleeve, the *mini*-Lam™, that maintains the same benefits of a laminar cup, but at a lower cost. The design of the new *mini*-Lam™ utilizes a shortened, inverted laminar cup. Because the head of the shortened laminar cup faces upward, septa particles and sample residue are not trapped in the cup body as seen with standard cup sleeves. The *mini*-Lam™ sleeve can also be used for dual column analysis in a split/splitless injection port using a two-hole ferrule. The wider base of the *mini*-Lam™ provides enough room for the installation of two capillary columns, whereas the standard laminar cup splitter could not.



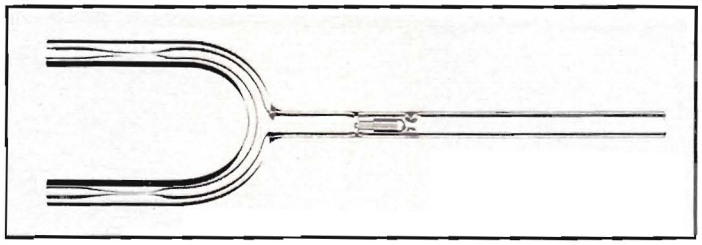
***mini*-Lam™ Split Sleeve for HP 5890 GCs**
cat.# 20990, each
cat.# 20991, 5-pk.

Dual Column *mini*-Lam™ Direct Injection Tee for 1/4" Packed Column Inlets

Complete vaporization of the sample and visual confirmation of the column connection

Direct injection into two columns simultaneously has become increasingly popular for dual column confirmational analysis. Previously, Restek only offered a cyclo dual column direct injection tee that utilized a glass screw to vaporize the sample prior to reaching the tee split point. Based on Dr. Grob's work[1], we now have a *mini*-Lam™ direct injection tee which allows complete vaporization and permits larger sample volumes.

The *mini*-Lam™ direct injection sleeve is designed to fit into 1/4" packed column injectors. The tee incorporates a press-tight taper in each outlet leg that delivers a perfect, dead volume free connection to each 0.32 or 0.53mm ID analytical column (OD ranging from 0.4 to 0.8mm) and allows visual confirmation of the column connection. The open top design makes it easy to pack with glass wool to keep dirty sample residue from contaminating the cup. A complete *mini*-Lam™ dual column direct injection kit is available that includes a deactivated 1/4" glass tee, 1/4" nut and ferrule, two 1/4" to 1/16" reducing unions, and ferrules. Deactivated replacement glass tees are also available.



***mini*-Lam™ Direct Injection Tee Kit:** cat.# 20436, kit
***mini*-Lam™ Direct Injection Tee:** cat.# 20435, each

1. K. Grob, *HRC & CC*, 15 (1992) 190.