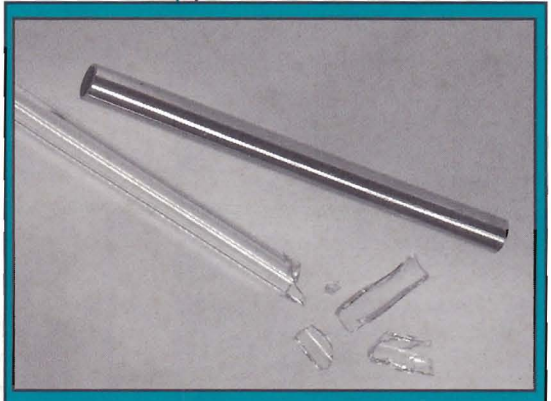
THE RESTEK ADVANTAGE

Innovative Silcosteel® Metal Inlet Sleeves

Restek has developed a new low cost, high performance inlet sleeve for the HP 5890/6890 split/splitless injector. Advances made by our manufacturing group enable us to introduce a stainless steel inlet sleeve with inertness equivalent to deactivated glass. Utilizing our advanced Silcosteel® process, metal sleeves are coated with a fused silica-like layer and then deactivated with a high temperature silanization process. These sleeves are ideal for split/splitless applications that require frequent changing of inlet sleeves to keep the injector contamination free. These sleeves are also excellent for portable GCs since they do not break during transportation or installation.

SILCOSLEEVE™ METAL INLET SLEEVES UTILIZE RESTEK'S SILCOSTEEL® SURFACE

Silcosteel® is a process that bonds a thin, uniform layer of flexible fused silica on the stainless steel. The surface is then deactivated and made inert by the same process used in our high quality fused silica and MXT® capillary columns. Because the Silcosteel® layer is flexible, it will not crack or break off the surface of the stainless steel.



- Equivalent inertness to glass sleeves.
- Excellent response for pesticides, phenols and other active compounds.
- Silcosleeve™ inlet sleeves won't crack, chip, or break like glass sleeves.
- Inexpensive and cost effective.

SILCOSLEEVE™ INLET SLEEVES SHOW EXCELLENT INERTNESS FOR ACTIVE COMPOUNDS

Inertness was demonstrated by injecting DDT and endrin at 50pg/µl in the splitless mode. Total DDT and endrin breakdown was calculated on the metal sleeve (Table 1) and compared to a deactivated glass inlet sleeve. Minimal breakdown was calculated for both sleeves which indicates that potential active sites are covered by the deactivation layer. This same standard was injected onto an untreated metal sleeve to show the breakdown of DDT to DDE and DDD due to active sites present on the surface of the sleeves (Table 1). Figure 1 illustrates the response of trace phenols using a Silcosleeve™ inlet sleeve. Phenols are also excellent indicators for inertness since they are chemically active compounds which adsorb on active surfaces.

STRENGTH AND DURABILITY

Since Silcosleeve™ inlet sleeves are stainless steel they are not prone to breakage when installing or removing the sleeve, or during transportation to a testing site. The fused silica is bonded to the stainless steel so it will not crack or break off the surface if mishandled or accidentally dropped.

SILCOSLEEVE™ INLET SLEEVES ARE COST EFFECTIVE

Since different manufacturing techniques are used in making Silcosleeve™ inlet sleeves, we are able to reduce the process time and pass the savings on to our customers. If you need highly inert and reasonably priced inlet sleeves for your HP 5890/6890 GC, try our new Silcosleeve™ metal inlet sleeves.

Silcosleeve™ Metal Inlet Sleeve for HP GCs

OD/ID & Length
6.35mm/5.2mm & 78.5mm

5-Pack	25-Pack
21700	21701

Add appropriate suffix to Cat. No. to order prepacked sleeves.

Packing	5-Pk. Suffix	25-Pk. Suffix
FS Wool	200.5	200.25
FS Beads	201.5	201.25
Glass Wool	202.5	202.25
CarboFrit™	209.5	209.25

IN THIS ISSUE...

Table 1 - Comparison of Endrin & DDT Breakdown

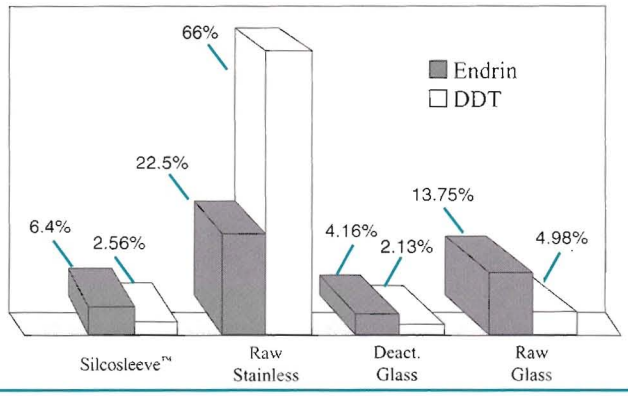
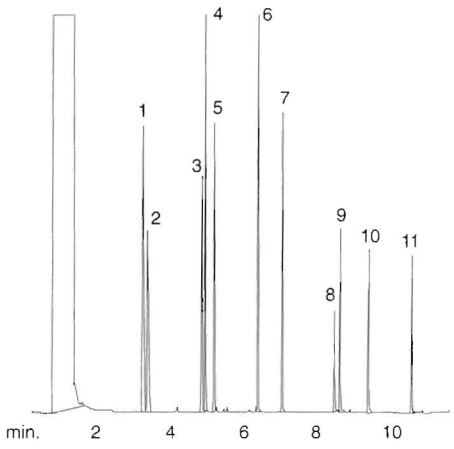


Figure 1 - Phenols are excellent indicators to demonstrate the inertness of Silcosleeve™ metal inlet sleeves.



1. phenol
2. 2-chlorophenol
3. 2-nitrophenol
4. 2,4-dimethylphenol
5. 2,4-dichlorophenol
6. 4-chloro-3-methylphenol
7. 2,4,6-trichlorophenol
8. 2,4-dinitrophenol
9. 4-nitrophenol
10. 2-methyl-4,6-dinitrophenol
11. pentachlorophenol

30m, 0.25mm ID, 0.25µm XTI-5 (12223)
1.0µl splitless inj. 50ng of phenols.
Oven temp.: 40°C to 350°C @ 15°C/min. (hold 15 min.)
Inj. temp.: 250°C
Detector: FID

Note: If you have a specific sleeve application please contact **your local distributor.**

PINNACLE™ TO-11:

The Definitive HPLC Column for Formaldehyde Analysis

EPA method TO-11 describes a procedure for the determination of formaldehyde as well as 14 other aldehydes and ketones in ambient indoor and outdoor air. Formaldehyde is of particular concern due to its widespread occurance and tendency to photochemically react to produce many pollutants such as peroxyacetyl nitrate compounds, peroxides, and ozone. Its major source of emissions is due to incomplete combustion of hydrocarbons such as fossil fuels and waste incineration. It is also released from many chemical and industrial processes in which it is used. Short term exposure to formaldehyde and other carbonyl compounds can cause irritation of the eyes, skin, and mucous membranes, while chronic or high level exposure may damage the lungs and other vital organs.

The TO-11 method outlines a procedure in which the sample is collected by drawing air through cartridges containing a solid adsorbent coated with dinitrophenylhydrazine (DNPH). Following collection the sample is eluted from the cartridge as the DNPH derivative and then analyzed by HPLC with UV detection. Although this procedure was written specifically for formaldehyde, it also lists 14 other aldehydes and ketones which can be analyzed with modification of the basic chromatographic procedure.

For the analysis of formaldehyde alone, the method recommends a single 25cm Zorbax™ ODS column with a water/acetonitrile mobile phase. Using this column and conditions, formaldehyde is retained approximately 7 minutes. Using a 15cm, 5µm Pinnacle™ TO-11 column, the retention is decreased to under 2 minutes. The analysis time can be further reduced by using a shorter, high efficiency 3µm Pinnacle™ column.

To separate all 15 compounds listed in the method the procedure recommends two 25cm Zorbax™ ODS columns in series and a 55 minute gradient with a 15 minute reequilibration. This long gradient and use of two columns is required because a regular ODS column does not have the proper selectivity to easily separate acetone, acrolein, and propionaldehyde as well as the tolualdehyde isomers. By using a Pinnacle™ TO-11 column which has been specifically optimized for this analysis, all 15 compounds can be easily separated isocratically in less than 10 minutes. For greater resolution of the closely eluting triplicates a 15cm x 3µm or 25cm x 5µm may be used.

The knowledgeable applications group at Restek has made similar improvements to other environmental methods as well. Application Briefs on the HPLC analysis of explosives, carbamates, and PAHs are available upon request. Call your local distributor to request more information on these applications.

Figure 1 - PINNACLE™ TO-11 elutes all 15 components in Method TO-11 isocratically in less than 10 minutes.

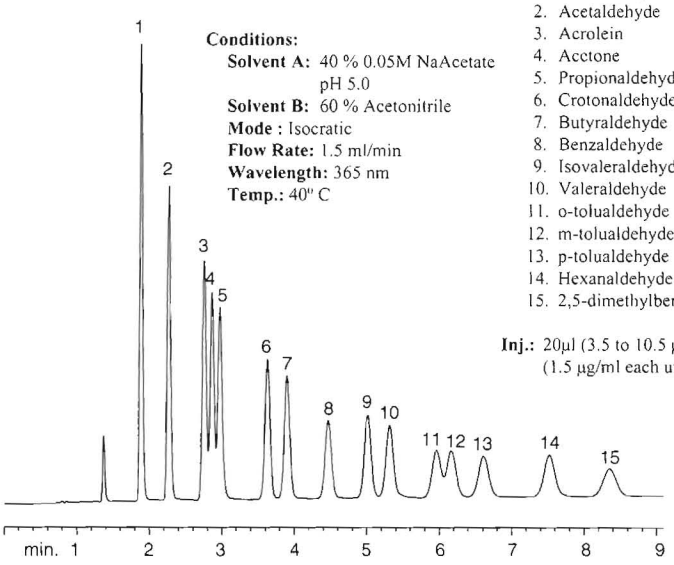
Call your local distributor to request a copy of Restek's HPLC Catalog!

Column: Pinnacle™ TO-11
Catalog Number: 9172565
Dimensions: 150 x 4.6 mm
Particle Size: 5 µm
Pore Size: 120Å

Conditions:
Solvent A: 40 % 0.05M NaAcetate pH 5.0
Solvent B: 60 % Acetonitrile
Mode : Isocratic
Flow Rate: 1.5 ml/min
Wavelength: 365 nm
Temp.: 40° C

DNPH Derivatives of:
1. Formaldehyde
2. Acetaldehyde
3. Acrolein
4. Acetone
5. Propionaldehyde
6. Crotonaldehyde
7. Butyraldehyde
8. Benzaldehyde
9. Isovaleraldehyde
10. Valeraldehyde
11. o-tolualdehyde
12. m-tolualdehyde
13. p-tolualdehyde
14. Hexanaldehyde
15. 2,5-dimethylbenzaldehyde

Inj.: 20µl (3.5 to 10.5 µg/ml of derivative) (1.5 µg/ml each underivatized)



PINNACLE™ TO-11

ID (mm)	Particle Size	Length (mm)	cat. #
4.6	3 µm	100	9172315
	3 µm	150	9172365
	5 µm	150	9172565
	5 µm	250	9172575
3.2	3 µm	100	9172313
	3 µm	150	9172363
	5 µm	150	9172563
	5 µm	250	9172573

— FAST ANALYSIS —
— IMPROVED RESOLUTION —
— ISOCRATIC SEPARATION —
— BINARY MOBILE PHASE —
— DECREASED SOLVENT USAGE —

Get "FAME"ous!

Restek's new FAMEWAX™ column for fast and efficient FAME analysis.

Current methods for the analysis of fatty acid methyl esters (FAMEs) have been designed using Carbowax® columns that provide a specific elution order and separation of polyunsaturated fatty acids (PUFAs) in 35 to 50 minutes. Restek's new 0.25 and 0.32mm ID FAMEWAX™ columns can provide necessary baseline resolution for complex PUFA samples in less than 22 minutes! 0.53mm ID FAMEWAX™ columns are also available for concentrated FAME samples and for conversion from packed to capillary columns.

Capillary column performance requirements for PUFA analysis are specified in AOCS and AOAC methods. The American Oil Chemists Society (AOCS) Method CE 1b-89 "FAMES analysis by capillary GLC" requires baseline resolution of C21:5n3 and C23:0 (internal standard [IS]) and C24:0 and C22:6n3 (DHA). The Association of Official Analytical Chemists (AOAC) Official Method #991.39 "Fatty Acids in Encapsulated Fish Oils and Fish Oil Methyl and Ethyl Esters" requires the same elution pattern as Carbowax® 20M and additional resolution of C23:0(IS) from C22:4n6.

FAMEWAX™ columns meet all the criteria listed in the methods in significantly less time, with faster flow and temperature program rates than other Carbowax® columns. The menhaden oil PUFA analysis in Figure 1 shows that C21:5n3 and C23:0 (IS) are well resolved, as are C24:0, C22:6n3 (DHA) and C24:1n9 with a total analysis time of only 22 minutes. Figure 2 shows the same analysis on the Supelco Omegawax® 250 column with identical GC conditions. Peaks C21:5n3 and C23:0 are not baseline resolved, nor are C22:6n3 and C24:1n9. To achieve resolution of these components on the Omegawax® column, the program rate must be decreased to 2 or 3°C/minutes, increasing analysis time by 59%!

The 30m, 0.32mm, 0.25µm FAMEWAX™ column also meets the criteria for PUFA analysis. Larger diameter 0.32 mm ID col-

Figure 1 - Menhaden Oil PUFA on a FAMEWAX™ Column

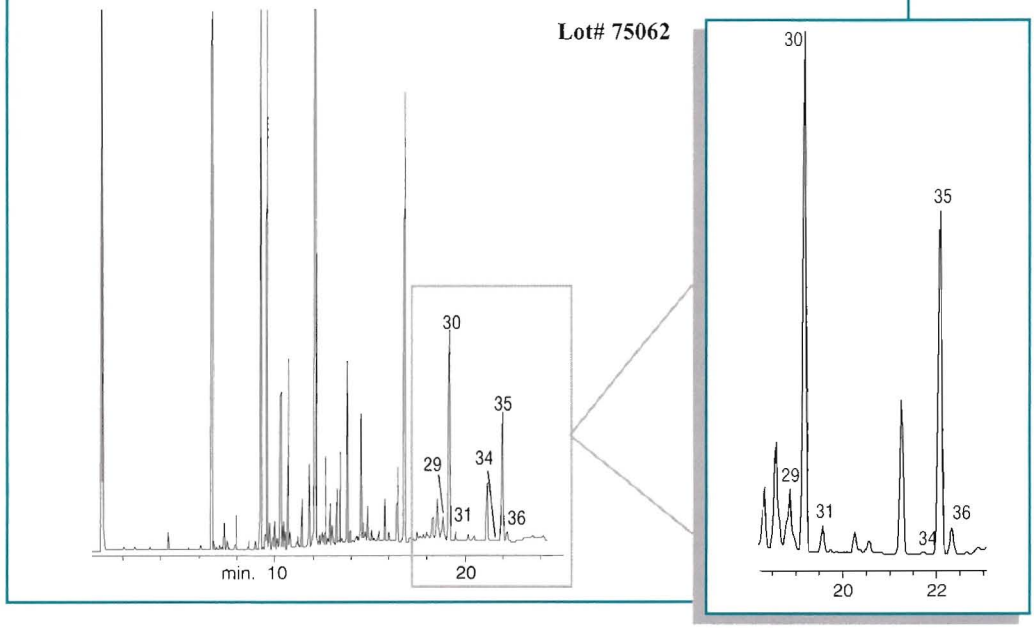
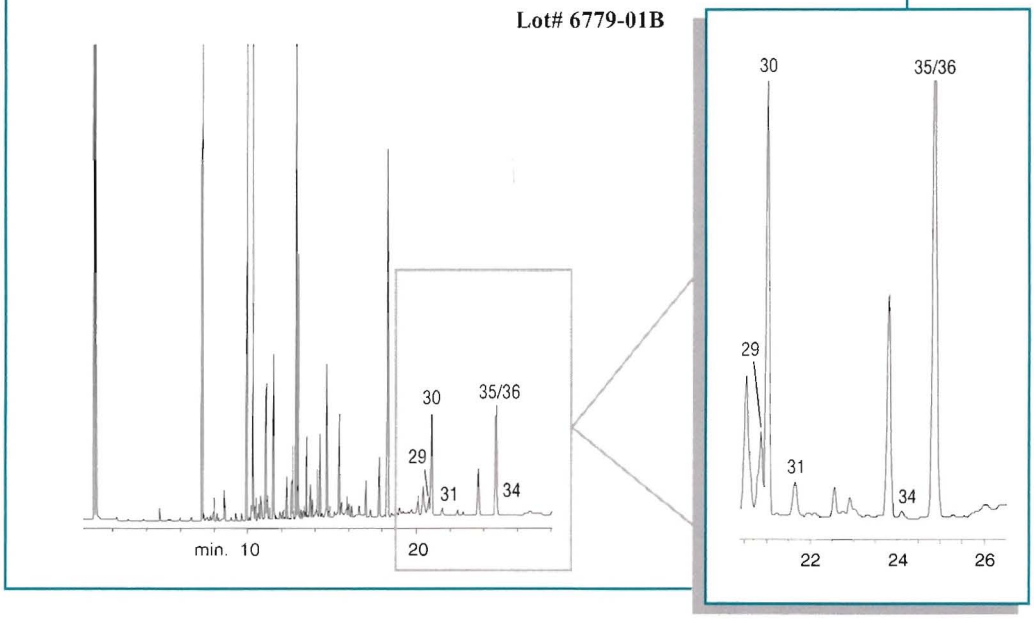


Figure 2 - Menhaden Oil PUFA on an Omegawax™ Column



umns have 4-5 times more sample capacity (400-500ng vs. 50-100ng). The increased sample capacity minimizes overloading of more concentrated samples with minimal loss in column efficiency.

The 30m, 0.53mm, 0.5µm FAMEWAX™ column has the sample capacity to accomodate direct and on-column injections. This wide bore column allows conversion from packed to capillary columns without the expense of adding a capillary injector to your GC. Although attaining the resolution requirements for PUFA analysis is difficult for most wide bore PEGs, Figure 3 illustrates that the 0.53mm ID FAMEWAX™ column can provide sufficient resolution for PUFA analysis.

Unlike similar columns from other manufacturers, all FAMEWAX™ columns are tested with two test mixtures. Each batch of polymer is tested with a menhaden type oil, and each column is tested with a Grob type test mix. The menhaden oil test ensures proper column polarity indicated by the separation and elution order of PUFAs. Also, 0.25mm and 0.32mm ID FAMEWAX™ columns must pass the resolution criteria outlined in the official methods for complex PUFA matrices. The Grob mix ensures inertness, efficiency, film thickness consistency and minimal column bleed.

Save yourself time and money. Try Restek's new FAMEWAX™ columns for fast and efficient FAMEs analyses. The 0.25mm and 0.32mm ID FAMEWAX™ columns provide optimum PUFA analyses in less time than other columns. The 0.53mm ID provides capillary conversion in packed column instruments, maximum sample capacity, and the necessary resolution for a wide variety of FAME analyses, including complex PUFA analysis. All FAMEWAX™ columns are tested with two test mixtures to provide you with the highest quality column for FAME analyses anywhere. We guarantee it!

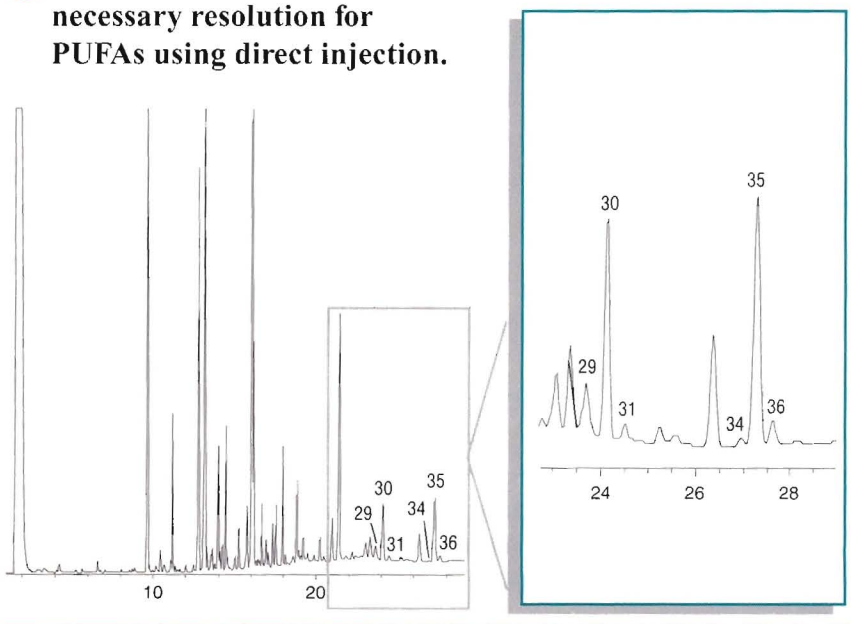
FAMEWAX™ Columns

- **Significantly reduces analysis times.**
- **Specially tested to ensure column reproducibility. We guarantee it!**
- **0.25mm and 0.32mm ID columns available for complex PUFA analysis.**
- **Also available in 0.53mm ID columns.**

FAMEWAX™ Columns

30m, 0.25mm ID, 0.25µm FAMEWAX™: cat.# 12497	30m, 0.32mm ID, 0.25µm FAMEWAX™: cat.# 12498	30m, 0.53mm ID, 0.50µm FAMEWAX™: cat.# 12499

Figure 3 - The 0.53mm ID FAMEWAX™ provides the necessary resolution for PUFAs using direct injection.



Compound List and Conditions for Figures 1-3

29. C21:5n3	34. C24:0
30. C23:0 (IS)	35. C22:6n3
31 C22:4n6	36. C24:1n9

Figure 1
30m, 0.25mm ID, 0.25µm FAMEWAX (75062)
Figure 2
30m, 0.25mm ID, 0.25µm Omegawax (6779-01B)
0.8µl split injection of menhaden oil PUFA with C23:0 (IS)
On-column concentration 100-150ng.

Oven temp.: 120°C to 220°C @ 7°C/min. (hold 20 min.)
Inj. & det. temp.: 220°C
Carrier gas: hydrogen
Linear velocity: 60cm/sec. set @ 120°C
FID sensitivity: 8 x 10^{-11} AFS
Split ratio: 50:1

Figure 3
30m, 0.53mm ID, 0.50µm FAMEWAX (82764B)
1.0µl direct injection of menhaden oil.
On-column concentration 2500ng total.

Oven temp.: 120°C (hold 2 min.) to 220°C @ 6°C/min. (hold 20 min.)
Inj. & det. temp.: 230°C
Carrier gas: hydrogen
Linear velocity: 32cm/sec. set @ 120°C
FID sensitivity: 8 x 10^{-11} AFS

PHARMACEUTICAL

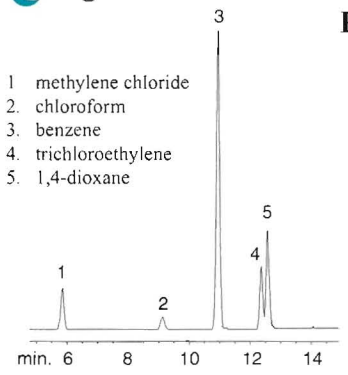
Integra-Guard™ Takes the Frustration Out of Analyzing Residual Solvents

The United States Pharmacopeia (USP) has published several methods for the analysis of residual solvents in pharmaceutical products. USP 467 outlines five methods for Organic Volatile Impurities (OVI) that utilize several different sample introduction techniques and analytical columns. Methods I and V have become the most popular because of the simplified sample introduction technique. Both of these methods involve the direct dissolution of the pharmaceutical product in water followed by direct aqueous injection. Since many pharmaceutical products contain inorganic additives and non-volatile components, the USP methods recommend the use of a 5 meter guard column to protect the analytical column. Although the use of a guard column can greatly increase the life expectancy of your analytical column, making a consistent leak-free connection has been difficult.

To eliminate the headache of making those difficult connections between the guard column and the analytical column, the Wizards at Restek have developed Integra-Guard™ (capillary columns with integrated guard columns). These columns are available with the two most common stationary phases used for OVI analysis. Figure 1 shows the analysis of common solvents on a 30 meter, 0.53mm ID, 5.0µm Rtx®-G27 Integra-Guard™ column (5% phenyl/95% methyl polysiloxane). Figure 2 shows the same analysis on a 30 meter, 0.53mm ID, 3.0µm Rtx®-G43 Integra-Guard™ column (6% cyanopropylphenyl polysiloxane). Both columns are manufactured with high purity, bonded stationary phases for low bleed and unsurpassed inertness. Each column is tested to meet stringent requirements to insure reproducible analysis.

Save time and money and improve your analysis by using Rtx®-G27 and Rtx®-G43 Integra-Guard™columns to analyze residual solvent impurities in pharmaceutical products. In addition, Restek offers several high quality calibration standards for USP 467 methods. Data packages with complete quality assurance information are available for all Restek standards.

Figure 1 - USP 467 analysis on a 30m, 0.53mm ID, 5.0µm Rtx®-G27 Integra-Guard™ column



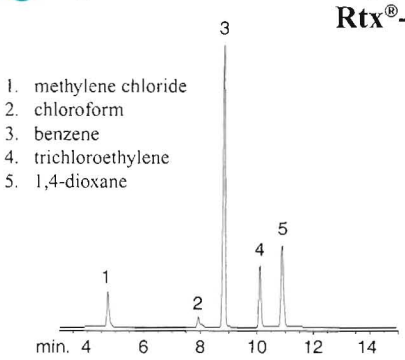
1.0µl direct injection of USP 467 Mix #1 (cat.# 36001).
Oven temp.: 35°C (hold 5 min.) to 175°C @ 8°C/min. to 260°C @ 35°C/min.
Inj. / det. temp.: 200°C / 240°C
Carrier gas: helium
Linear velocity: 30cm/sec. set @ 35°C
FID sensitivity: 1 x 10^{-11} AFS

Columns

30m, 0.53mm ID, 5.0µm
Rtx-G27 Integra-Guard™
cat.# 10279-126

30m, 0.53mm ID, 3.0µm
Rtx-G43 Integra-Guard™
cat.# 16085-126

Figure 2 - USP 467 analysis on a 30m, 0.53mm ID, 3.0µm Rtx®-G43 Integra-Guard™ column



1.0µl direct injection of USP 467 Mix #1 (cat.# 36001).
Oven temp.: 35°C (hold 5 min.) to 175°C @ 8°C/min. to 260°C @ 35°C/min.
Inj. / det. temp.: 200°C / 240°C
Carrier gas: helium
Linear velocity: 34cm/sec. set @ 35°C
FID sensitivity: 1 x 10^{-11} AFS

Standards

USP 467 Mix #1 (in DMSO)
cat.# 36001, ea.
cat.# 36101, 10-pk.

USP 467 Mix #2 (in methanol)
cat.# 36002, ea.
cat.# 36102, 10-pk.

USP 467 Mix #3 (in DMSO)
cat.# 36004, ea.
cat.# 36104, 10-pk.

International USP 467 Mix (in methanol)
cat.# 36003, ea.
cat.# 36103, 10-pk.

See Restek's Product Guide for more information on USP 467 standard mixes.

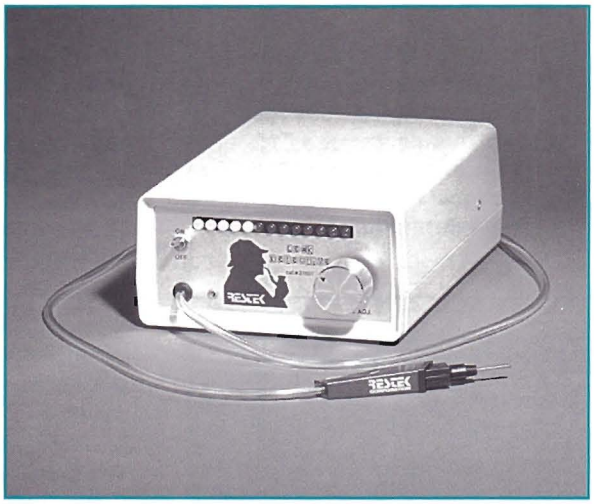
- **Integrated guard column eliminates leaks.**
- **Meets all requirements of USP 467 methods.**
- **Bonded phase columns for low bleed & excellent inertness.**

Restek's Leak Detective™

Restek's Leak Detective™ electronic leak detector is the convenient and affordable solution for GC leak detection. It responds in less than 2 seconds to trace leaks of gases* with thermal conductivities different than air. Helium or hydrogen can be detected at 3 x 10^{-4}cc/sec. or an absolute concentration less than 200ppm. Leaks are indicated by an audible alarm, as well as an LED readout. Two 9-volt batteries provide 10-12 hours of continuous operation, or the unit can be used with an AC adaptor (both included).

Features

- Lowest cost thermal conductivity leak detector available.
- Compact, light weight, hand-held design.
- Contamination-free leak detection.
- Detects helium or hydrogen trace leaks at ≥ 3 x 10^{-4}cc/sec. or ≥ 200ppm.



Detecting Leaks

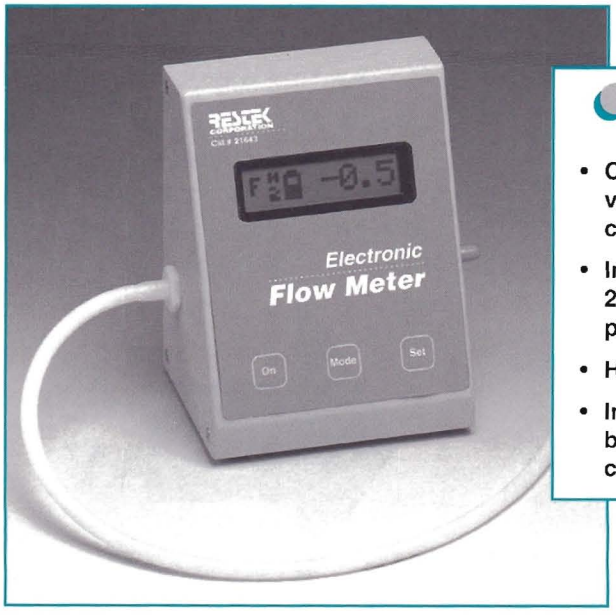
Leaks in a GC system can cause problems ranging from increased detector noise, baseline instability, and short column lifetime, to wasting expensive carrier gases. Electronic leak detectors, like Restek's Leak Detective™ allow analysts to detect minute gas leaks undetected by liquid leak detectors without risk of contamination. Electronic leak detectors are an absolute necessity with capillary GC!

Leak Detective

cat.# 21607 (110 Volts)
cat # 21609 (220 Volts)

** Not designed for use in explosive atmospheres.*

Restek's Veri-Flow 500 Flow Meter



New Features

- Calculates linear velocity based on column ID.
- Includes standard RS 232 ComPort with printer function.
- Has auto zero function.
- Includes rechargeable battery pack and charger.

Other Features

- Reads flow accurately to 500mL/min.
- Measures N_2, He, H_2, 5% Ar/Me, and Air.
- Measures split flow and mass flow.
- Has pulse free operation that will not interfere with EPCs.
- Includes auto "off" battery saver.
- Includes NIST traceable calibration certificate.
- Includes capillary column adaptor.

Veri-Flow 500 Flow Meter

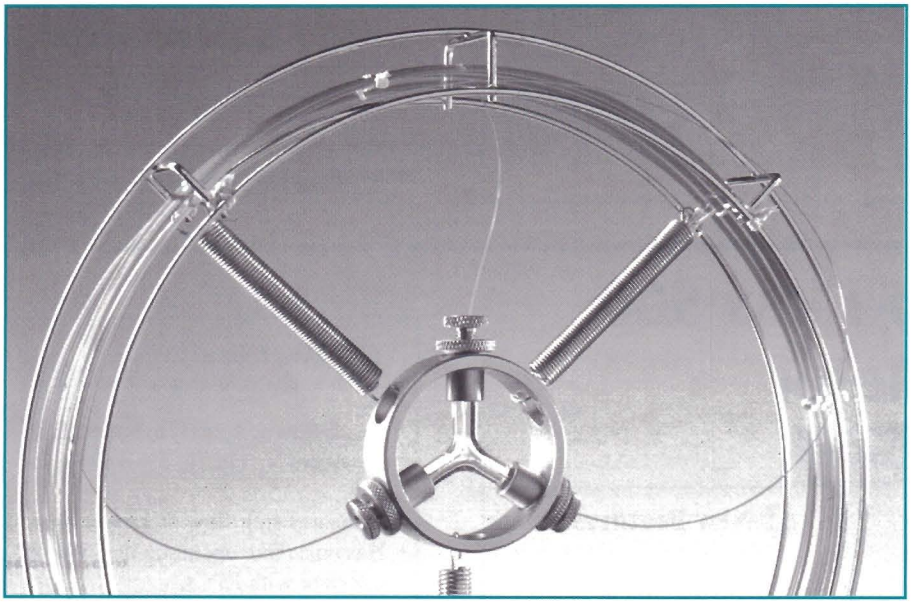
110 V, cat.# 21643
220 V, cat.# 21645

Improved Pesticide Column Kits

Restek developed pesticide column kits in the early 90's to meet the requirements of the EPA's CLP chlorinated pesticide protocol. By connecting two capillary columns with different stationary phases to a "Y" connector and 5 meter guard column, analysts gained the advantage of running both primary and confirmational analyses simultaneously. However, the awkward angle at which the columns were inserted into the connector caused some columns to leak, disconnect, or even break. Restek solved this problem by introducing the Angled "Y" Press-Tight® connector and the "Y" Vu-Union® connector. The "Y" Vu-Union® incorporates a secondary ferrule seal that makes it almost impossible for the connection to come apart, making it ideal for higher temperature analyses. With these solutions in hand, analysts still found getting a leak free connection required "some special magic" Now analysts can rely on Restek's chemists to make leak-free dual column connections for them.

Features

- **Pre-assembled kits offer convenience over purchasing parts separately.**
- **All pesticide column kits are tested for flow and leaks prior to shipping.**
- **Custom column combinations are available with either an Angled "Y" Press-Tight® connector or a "Y" Vu-Union® connector. Call your local distributor for more information.**



"Y" VU-UNION® CONNECTOR

Restek's "Y" Vu-Union® connector is the latest advancement in "Y" connectors. It has quickly gained the reputation as the most reliable connector for pesticide column kits. The primary seal is visible, to assure the connection is made properly without dead volume or leakage. The secondary seal utilizes standard graphite ferrules to physically hold the columns in place and assure a "leak free" connection remains "leak free." Not only are the columns (two 30 meter analytical columns and one 5m x 0.53mm ID guard column) protected in Restek's unique capillary column cage, but the Vu-Union® connector is securely suspended inside the column cage with springs. This configuration reduces stress on the fused silica tubing and acts as a "shock absorber" from the turbulence created inside the GC oven.

ANGLED "Y" PRESS-TIGHT® CONNECTOR

Restek's Angled "Y" Press-Tight® connector has the correct angle to assure easy, long lasting connections. Each one is carefully tested to insure a leak free seal. The guard column is carefully tucked inside the cage and is ready to use when you receive the column.

Simply decide which columns you want to connect and the type of connector you wish to use and let us make the connection for you.

Pesticide Column Kits

0.32mm ID & other phase combinations are available.

Column Combo's (30m, 0.53mm ID, 0.50µm)	Cat.#	"Y" Vu-Union® connector suffix #	Angled "Y" Press-Tight® connector suffix #
Rtx-5/-1701	10950	-495	-496
Rtx-5/-35	10951	-495	-496
Rtx-35/-1701	10952	-495	-496

"Sample Ready" Packed Columns...

...Require Less Than 20 Minutes Stabilization Time!

- **Fully conditioned and "sample ready" for maximum productivity.**
- **Silcosteel® tubing inert as glass.**
- **Deactivated with same process used for capillary columns.**
- **Rugged and flexible.**
- **Universal and versatile, can be bent to any instrument or detector configuration.**
- **Tighter tubing tolerances result in improved efficiency and reproducibility over glass.**

Since the inception of glass deactivation technologies in the 1970's, packed column technology has remained virtually unchanged. In 1994, Restek introduced a breakthrough in packed columns by offering a metal packed column with efficiency and inertness exceeding the best glass packed columns. Restek's Silcosteel® process and deactivation chemistries have produced the most advanced surface for the production of packed columns on the market today.

Because the tubing material is stainless steel and the fused silica layer is flexible, Silcosteel® packed columns are truly universal. Columns can be easily moved from one instrument to another. Silcosteel® packed columns offer analysts the flexibility and versatility previously seen only by capillary column users.

All packings offered by Restek are fully conditioned and "sample ready". With only 15-20 minutes of stabilization time, your GC is ready to run. This improves throughput and lab efficiency. Restek offers a wide range of packings and liquid phases including Silcoport™, the most inert support available for packed columns. In fact, our porous polymer and Molecular sieve columns are the best we've ever seen!

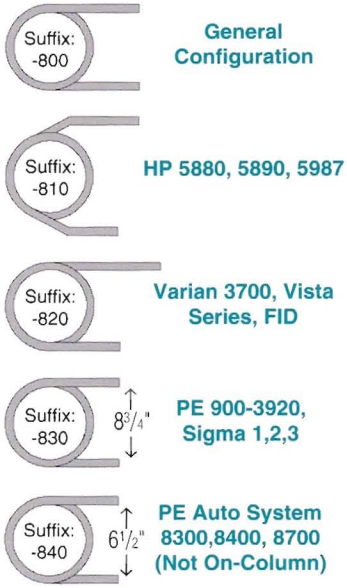
New Silcosteel® Packed Columns:*

2m x 1/8" OD x 2mm ID; Preconditioned
Chromosorb 101 80/100: cat.# 80435
Chromosorb 102 80/100: cat.# 80434
Hayesep Q 80/100: cat.# 80433
Porapak Q 80/100: cat.# 80427
Porapak QS 80/100: cat.# 80426
Porapak R 80/100: cat.# 80425
Tenax TA 60/80: cat.# 80431
Tenax TA 80/100: cat.# 80432
Molesieve 5A 60/80: cat.# 80428
Molesieve 5A 80/100: cat.# 80429

3.05m x 1/8" OD x 2mm ID; Fully activated
Molesieve 5A 80/100: cat.# 80430

2m x 3/16" OD x 4mm ID
1.5% Rt 2250 / 1.95% Rt 2401
on 100/120 SilcoPort: cat.# 80254

Instrument Configurations



** Please specify catalog number and instrument configuration suffix when ordering.*

Call your local distributor for more information on Silcosteel® packed columns or to receive Restek's Packed Column Catalog.

SILCOSTEEL®

Combining the durability of stainless steel with the inertness of glass.

Restek Advances PLOT columns – Again!

Porous polymers have been used for nearly three decades for separating light hydrocarbons and solvents. The columns traditionally used have been 2mm ID columns packed with 80/100 mesh material. The wizards at Restek have now made capillary PLOT columns available that utilize the same porous polymer materials. The Rt-S and Rt-Q PLOT columns offer a choice to the analyst who desires the selectivity of the divinylbenzene porous polymers but the efficiency of capillary columns. Figure 1 illustrates light hydrocarbons analyzed on an Rt-S and Rt-Q PLOT column. The incorporation of different vinyl monomers into the divinylbenzene porous polymer matrix produces noticeable selectivity variations. The Rt-S column provides baseline separation of ethylene and acetylene. PLOT columns offer higher efficiency when compared to packed columns, resulting in faster analyses.

Because of the large amount of porous polymer in packed columns, long conditioning times are necessary. However, with PLOT columns the actual amount of porous polymer is significantly lower. Not only does this shorten conditioning times and increase throughput, it also reduces the overall bleed level (critical with high temperature applications where low background levels are desired).

TRUE SELECTIVITY OF TRADITIONAL POROUS POLYMER PACKINGS

Restek starts with high purity monomers for production of porous polymer PLOT columns. This ensures the columns will have a selectivity identical to that of the Porapak® and HayeSep® packings. When switching from packed columns to capillary columns, very little method development is required because the elution patterns remain the same. The Rt-Q PLOT column is a divinylbenzene homopolymer, whereas the Rt-S PLOT column is a divinylbenzene and 4-vinylpyridine copolymer.

PLOT Columns

- **PLOT columns offer an efficient alternative to packed columns.**
- **Selectivity identical to HayeSep® and Porapak® packings.**
- **All PLOT columns immobilized to eliminate particle generation.**
- **Unbreakable MXT-Q and MXT-Msieve 13X.**
- **Each column individually tested for reproducibility.**

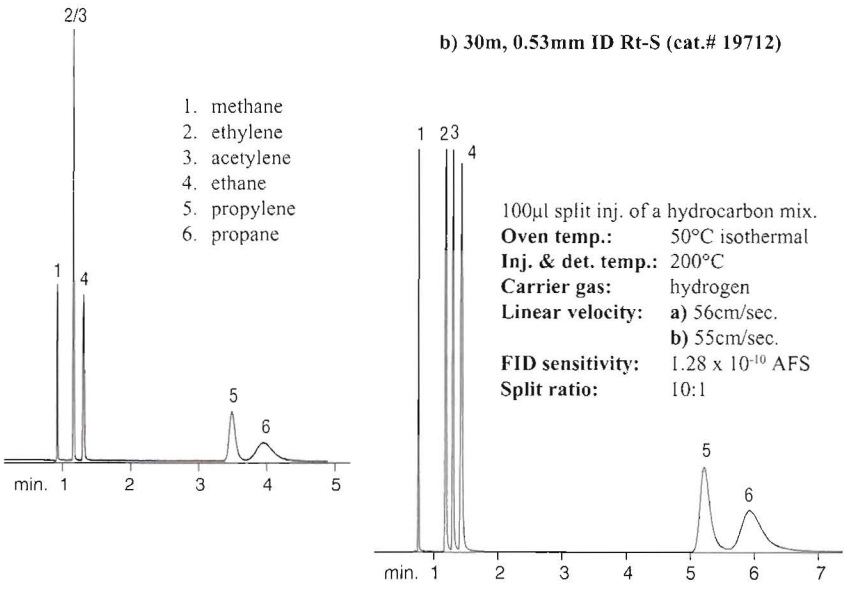
NO PARTICLE TRAPS NECESSARY

Because PLOT columns are made with submicron particles attached to the column wall, finding a good binder is critical. Restek has discovered a binding material that eliminates particle generation from PLOT columns. This process eliminates detector spiking and disruption of the valving system.

NEW LINE OF UNBREAKABLE MXT PLOT COLUMNS

The wizards at Restek have coated PLOT columns utilizing Silcosteel® fused silica lined tubing. The Silcosteel® PLOT columns offer the analyst an unbreakable option to standard fused silica tubing with no compromise in performance. MXT® PLOT columns must meet or exceed the same rigorous specifications as fused silica PLOT columns. Because the tubing is stainless steel, field and process analyzers are no longer at the mercy of brittle fused silica tubing. Current metal PLOT columns are

Figure 1 - Selectivity changes with the incorporation of different monomers in the porous polymer.



available with the Q porous polymer or Molecular sieve 13X zeolite. Figure 2 shows the analysis of light hydrocarbons on an MXT®-Q PLOT column and the analysis of permanent gases on an MXT®-Msieve 13X PLOT column.

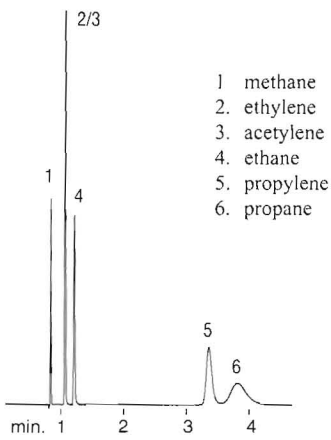
COLUMN TO COLUMN REPRODUCIBILITY GUARANTEED

Each porous polymer PLOT column is tested with a hydrocarbon test mix to insure proper phase thickness and selectivity. Propane is used to calculate the partition ratio which is monitored to insure a reproducible film thickness. The number of plates per meter is calculated and used to evaluate column efficiency. Selectivity is ensured by calculating the retention indices of acetylene and methyl acetylene.

Molecular sieve PLOT columns are all tested with a mixture of permanent gases. In this test the peak height ratio of carbon monoxide is used to determine a uniform film and to calculate the number of plates per meter.

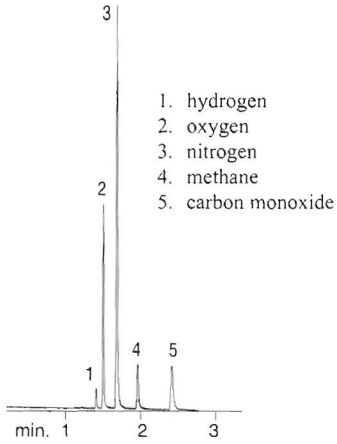
Restek's expanding line of PLOT columns continue to bridge the gap from packed to capillary columns. Through continuing research Restek now offers unbreakable and particle free *P*orous *L*ayer *O*pen *T*ubular capillary columns for the analysis of permanent gases, light hydrocarbons and volatile chemicals.

Figure 2 - MXT® PLOT columns give fast, efficient separation of light hydrocarbons & permanent gases.



30m, 0.53mm ID MXT-Q (cat.# 79716)
100µl split inj. of a hydrocarbon mix.

Oven temp.:	50°C isothermal
Inj. & det. temp.:	200°C
Carrier gas:	hydrogen
Linear velocity:	62.5cm/sec.
FID sensitivity:	1.28 x 10^{-10} AFS
Split ratio:	10:1



30m, 0.53mm ID MXT-Msieve 13X (cat.# 79706)
20µl split inj. of permanent gases.

Oven temp.:	40°C isothermal
Inj. & det. temp.:	200°C
Carrier gas:	helium
Linear velocity:	37cm/sec.
Detector:	TCD (212 mA)
Split ratio:	15:1

PLOT Columns

Rt-S
(fused silica)

30m, 0.53mm ID	cat.# 19712
15m, 0.53mm ID	cat.# 19713
30m, 0.32mm ID	cat.# 19710
15m, 0.32mm ID	cat.# 19711

Rt-Q
(fused silica)

30m, 0.53mm ID	cat.# 19716
15m, 0.53mm ID	cat.# 19715
30m, 0.32mm ID	cat.# 19718
15m, 0.32mm ID	cat.# 19717

MXT®-Q
(Silcosteel®)

30m, 0.53mm ID	cat.# 79716
15m, 0.53mm ID	cat.# 79715

Rt-Msieve 13X
(fused silica)

30m, 0.53mm ID	cat.# 19706
15m, 0.53mm ID	cat.# 19708
30m, 0.32mm ID	cat.# 19705
15m, 0.32mm ID	cat.# 19707

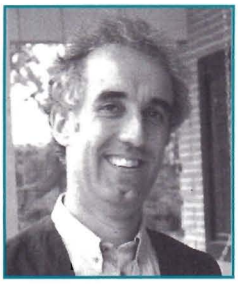
MXT®-Msieve 13X
(Silcosteel®)

30m, 0.53mm ID	cat.# 79706
15m, 0.53mm ID	cat.# 79708

Rt-Alumina
(fused silica)

30m, 0.53mm ID	cat.# 19700
50m, 0.53mm ID	cat.# 19701
30m, 0.32mm ID	cat.# 19702
60m, 0.32mm ID	cat.# 19703

Why 5 cm syringe needles for capillary GC?



Dr. Konrad Grob

GC is a complex technique. All too often the analyst stands in front of his instrument, surprised about a result, maybe annoyed about a problem, and at a loss for an explanation for what he observes. Often, not even his colleague is able to explain. Another of these GC mysteries? Probably he would have the knack of it if he knew the many details involved in the analytical process. We make numerous choices without being aware of them, overlook variables clinging to the illusion that they had been thoroughly investigated in the past and that an international committee has decided that this or that is the correct choice. The length of the syringe needle is one such frequently neglected detail and is an example of a parameter which has never received proper attention.

Many years ago, the manufacturers of GC syringes looked upon their customers and noticed that there was no agreement on how long syringe needles should be for conventional vaporizing (split or splitless) injection. Some said 1.5 inch (the needle protruding 37 mm from the glass barrel), others 3 inch (71 mm), or even longer. So, father syringe producer decided to compromise and have it in between: 2 inch (51 mm). Whether or not he died in the mean time, that's how it still is. Some disagreed, but since it seems to be more important that GC is simple than that it is well optimized, the subject was commonly neglected. The subject of needle length seems not to be of sufficient scientific status to justify closer investigation.

As you can check by a few experiments, the length of the syringe needle and the depth by which a long needle is inserted into the injector often have an important impact on quantitative analysis. The reasons are explained below. It is concluded that they need to be adjusted to the situation. The length of the syringe needle determines from which point inside the liner the sample expands during the evaporation process. It may, however, also influence vaporization itself.

HEADSPACE ANALYSIS:

We start by looking at gas or headspace analysis, because the situation is particularly simple since no vaporization interferes. However, the same principals will also apply to liquid samples. We refer to (manual or automated) injection with a gastight syringe of 0.5-1 ml capacity.

Usually an amount of gas phase is injected that approaches the internal volume of the vaporizing chamber. For instance, a 4 mm ID liner of 80 mm length has an internal volume of 1 ml. A 500 µl sample mixes with carrier gas to form a vapor cloud of close to this volume (inlet pressure compresses the cloud, but increased temperature causes it to expand). Care must be taken to release the sample from the syringe needle in such a way that it ends being positioned inside the chamber.

Gas and headspace samples are usually injected in the split mode in order to achieve sharp initial bands. Depression of the plunger at normal speed introduces the sample at around 0.5-1 ml/s, i.e. 30-60 ml/min. If the sum of the split and the (comparably small) column flow rate corresponds to the rate of injection, expansion of the sample downwards replaces the gas flow from the rear. Gas supply is stopped; the gas phase running off originates from the syringe (assumption of a pressure-regulation/needle valve system, Fig. 1). At higher split flow rates, the sample is diluted with additional carrier gas from the rear. Under these conditions, basically unlimited volumes of sample can be injected without overloading the injector. A short syringe needle merely entering the vaporizing chamber (2-3 cm) serves the purpose, but longer needles are no drawback.

Since headspace analysis is mostly trace analysis, the split flow rate is usually substantially below the 30-60 ml/min mentioned above. This leaves the choice of injecting at a correspondingly reduced rate or temporarily storing the vapor cloud inside the vaporizing chamber. The latter corresponds to common practice. If more sample is injected than gas runs off at the same time, carrier gas must be displaced within the injection system. Appropriately designed injectors with a pressure regulator at the rear and a needle valve in the split outlet have a relatively large internal volume in the gas supply and a small one in the split outlet, causing the sample to expand backwards (Fig. 2). Long syringe needles are required such that the sample expands from a point near the column entrance towards the rear. If the liner is 80 mm long, the column enters by 5 mm, and the injector head is some 12 mm high, the syringe needle should be around 80 mm

Figure 1 - Injection at a rate equal to the flow rate of the gas passing through the liner: the flow from the carrier gas supply is substituted by that leaving the syringe needle.

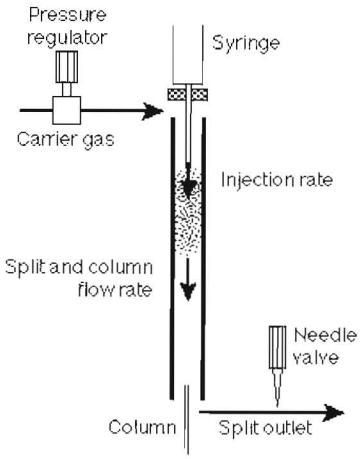
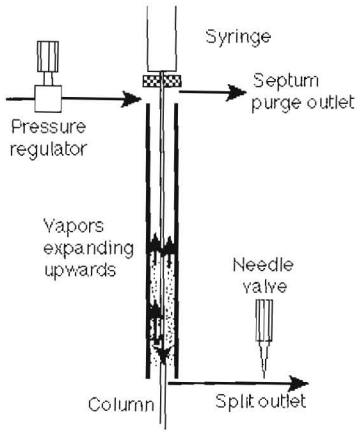


Figure 2 - Head-space injection at a low split flow rate, using gas supply by the pressure regulator/needle valve system: the sample should expand from the column entrance backwards.



long. The commonly used 5 cm needles enter the liner by less than 4 cm and merely exploit the upper half of the chamber. 500 µl thus injected already overfill the injector liner, i.e. cause sample material to be expelled through the septum purge outlet or to penetrate the carrier gas supply line.

Systems with flow-regulated carrier gas supply and a back pressure regulator in the split outlet (e.g. Hewlett Packard) behave differently. Pressure increase by injection causes the back pressure regulator to open widely and increase the split flow rate. The sample cloud expands downwards (Fig. 3). As the volume of the injector can only be exploited by releasing the sample at the top of the chamber, the syringe needle should be no longer than 2-3 cm (or a longer needle should be introduced only partially).

A drawback of this type of gas supply is the split flow rate during the splitting process is rather ill defined.

SPLIT INJECTION OF LIQUID SAMPLES

Split injection of liquids resembles gas/headspace injection except that the rate of vapor formation cannot be controlled. Injection must occur rapidly in order to avoid excessive evaporation inside the syringe needle. 2 µl of a solution in a volatile solvent, such as dichloromethane, creates some 0.9 ml of vapor in maybe 0.5, i.e. vapors are formed at 1.8 ml/s (108 ml/min). With a split (and column) flow rate of 108 ml/min at least, the situation of Fig. 1 applies, i.e. the syringe needle should merely enter the vaporizing chamber. It leaves maximum room between the needle exit and the column entrance for sample evaporation and mixing across the vaporizing chamber. If the split flow rate is lower, i.e. vapors are formed more rapidly than gas is discharged, a long or a short needle is best suited, depending on the carrier gas supply system involved (Fig. 2 or 3).

Samples with high boiling matrices, such as many undiluted liquids, evaporate slowly; discharge of the vapors is a problem only if the split flow rate is extremely low. Such liquids are easily transferred to the wall of the liner (no repulsion by vapors). If an empty liner is used (preferably of narrow bore, e.g. 2 mm), short syringe needles render such transfer more reliable as the risk of shooting the sample liquid by the column entrance becomes small.

SPLITLESS INJECTION

In splitless injection, the sample vapors must be stored in the vaporizing chamber until they are transferred into the column, which may take over a minute. Before being diluted with carrier gas, 2 µl of a solution in hexane produce around 500 µl of vapor, in dichloromethane as much as 900 µl, which shows that the internal volume of an 80 mm x 4 mm ID liner must be fully exploited.

As the split outlet is closed, there is only one way of filling the vaporizing chamber: from the bottom to the top, displacing the carrier gas backwards. The syringe needle must be adjusted to situate the center of sample evaporation slightly above the column entrance. The distance between the needle exit and the column entrance must account for the distance the droplets travel before evaporating, i.e. 1-2 cm. For the usual geometry of the injector this means using 3 inch (71 mm) needles (or rather the vaporizing chamber was designed such that standard 3 inch needles would fit). There is a second reason for depositing the sample close to the column entrance. As shown in Fig. 4 (on the following page), a 5 cm syringe needle leaves a distance of some 40 mm to the column entrance, representing a plug of some 400 µl of carrier gas. Before substantial amounts of sample vapor reach the column, this gas must be discharged into the column, i.e. during 10-20s primarily carrier gas is "injected".

Figure 3 - Sample expanding downwards in the instance of a system with flow regulation/back pressure regulation.

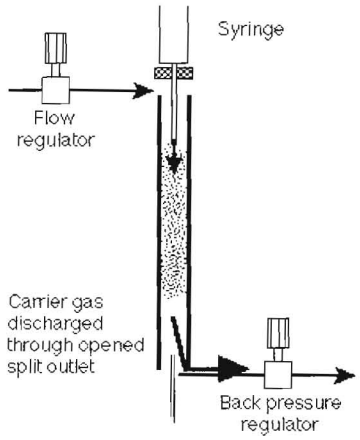
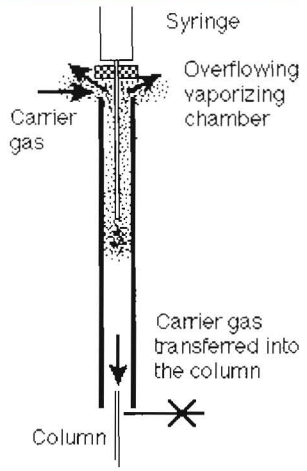


Figure 4 - 5cm syringe needles are too short for splitless injection as the chamber is overfilled even with small sample volumes & some 400µl of carrier gas must be transferred into the column before sample vapors get there.



Knowing how difficult it is to achieve complete sample transfer in splitless injection, this is certainly not the kind of problem we need.

SAMPLE EVAPORATION INSIDE THE NEEDLE

As if the subject were not of sufficient complexity yet - the length of the syringe needle also influences sample evaporation. Parts of the sample may be vaporized inside the needle during injection or when the needle content is eluted after the plunger is fully depressed. On the one hand, this often causes problems as more is injected than measured and preferential vaporization of volatile components discriminates against high boilers. On the other hand, it helps nebulizing the sample liquid at the needle exit, which is the prerequisite for sample evaporation in the gas phase of the injector (the most gentle vaporization process, since there are no contacts with packing materials adsorbing or degrading solutes). There is more vaporization inside long needles accentuating these advantages and disadvantages.

CONCLUSIONS

The 5 cm needle for vaporizing GC injectors is a typical compromise: it is between the desirable long and the desirable short needle, but is hardly ever desirable as such. The following table suggests optimum needle lengths.

Optimum Needle Lengths

Injection Technique	Gas Supply System: Pressure reg./ needle valve	Gas Supply System: Flow reg./ back pres. reg.
Splitless	71 mm	71 mm
Split (flow rate >100 ml/min.)	25 mm	25 mm
Split (flow rate <100 ml/min.)	71 mm	25 mm
Split, high boiling matrix	25 mm	25 mm

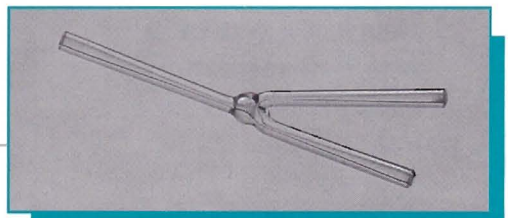
The length of the syringe needle is more critical than usually recognized. Try and see! Although this has been known for more than 15 years, only a few autosamplers give you the choice of varying the injection point. Presumably this is because too many customers ask more about the software for data handling than about the gas chromatograph when they buy a new instrument. Today much emphasis is given to quality assurance. Large amounts of time are invested into general QA procedures, the usefulness of which is not always obvious. Upon such efforts, easily more important optimization of technical aspects is neglected.

PERFORMERS

- Alleviates column end connection strain.
- Inlet and outlet ends conform to the column radius.
- Perform confirmational analysis with a single injection.

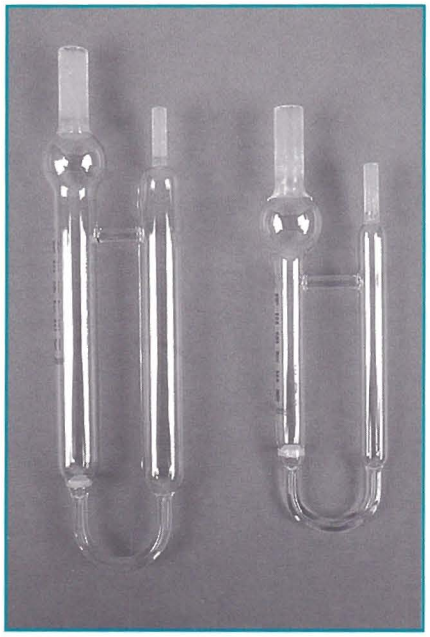
Universal Angled "Y" Press-Tight® Connector

cat.# 20403, each
cat.# 20404, 3-pk.



Purge & Trap Spargers

- For Tekmar 2000, 3000 and ALS 2016/2032
- Available in 5 and 25ml sizes
- Uniform frits to ensure maximum purge efficiency



Restek now offers purge and trap fritted spargers for Tekmar concentrators. These spargers provide maximum purge efficiency for water samples. Each sparger is manufactured with tight tolerances to ensure a leak free seal. These spargers are not recommended for wastewater samples since the frit may become plugged.

Spargers

5ml Fritted Sparger, ½" mount
cat.# 21150, each

25ml Fritted Sparger, ½" mount
cat.# 21151, each

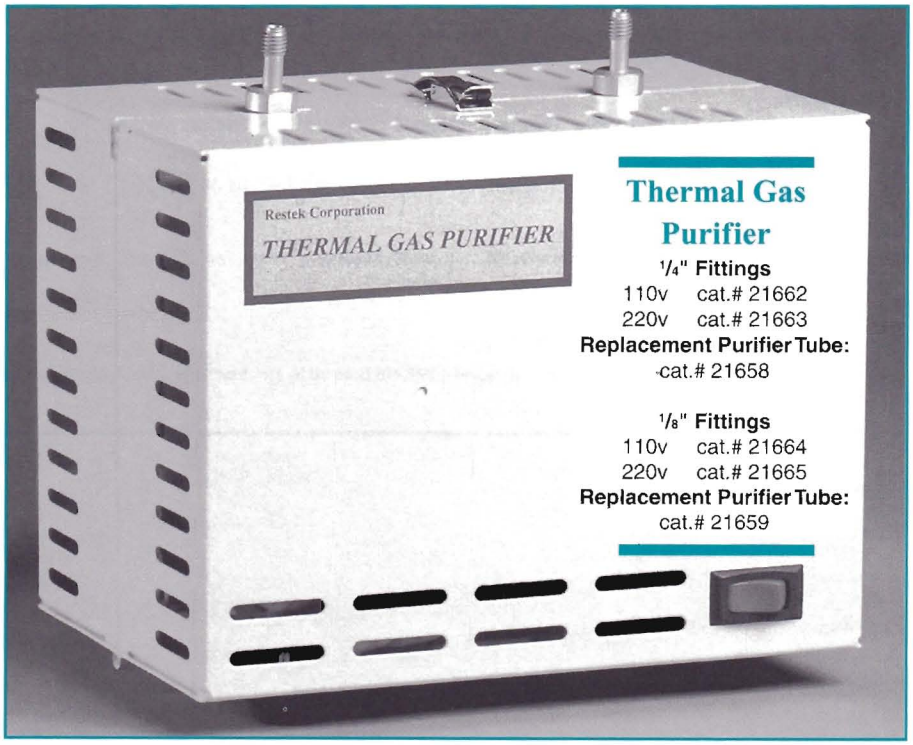
Restek's Thermal Gas Purifier

GC columns and detectors require pure carrier gases to operate at maximum sensitivity. With only 1% of trace contaminants in the carrier gas, column lifetime will be reduced. Restek has developed a new thermal gas purifier that removes trace contaminants from the carrier gas stream. This purifier removes oxygen, water, carbon monoxide and carbon dioxide by a high temperature reaction with purifier granules, compared to conventional adsorption traps. The constant high temperature reaction with the *getter* granules prevents contaminants from returning to the gas stream.

- Top mounted fittings ease installation & save bench space.
- Save money by scrubbing lower grade gases clean.
- Removes oxygen and water.
- Welded end fittings eliminate leaks.
- U-tube configuration eliminates channeling.
- UL certification (pending).

Each of Restek's purifier tubes are thermally cleaned prior to shipment and are filled with N_2 and capped to ensure contamination free start-up. The purifier tubes have welded end fittings that eliminate leaks from occurring. Submicron frits are used in the end of each tube to prevent particles from entering and contaminating the carrier gas lines. One purifier removes 17 liters of oxygen and 42 liters of water vapor, allowing a single unit to service multiple instruments.

Restek's purifier design utilizes less bench space than comparable devices since all gas connections enter and exit the top of the unit. The "U-tube" configuration virtually eliminates channeling of the getter material ensuring maximum purification of the carrier gas and higher flow rates.



Note: Not recommended for use with flammable gases.

Restek (U.S.):
110 Benner Circle, Bellefonte, PA 16823 • **Phone:** (800)356-1688 • **FAX:** (814)353-1309
http://www.restekcorp.com

Restek GmbH:
Sulzbacher Str. 15, D-65812 Bad Soden, Germany •**Telefon:** 06196/65130 • **Telefax:** 06196/62301

Restek France:
1, Rue Montespan, 91024 Evry, France • **Telefon:** 01 60 78 32 10 • **Telefax:** 01 60 78 70 90

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