RESTEK
The Advantage

Innovators of High Resolution Chromatography Products

Rtx®-CLPesticides & Rtx®-CLPesticides2

A complete solution for chlorinated pesticide, herbicide, and PCB analysis.

by Frank Dorman

- **Baseline resolution of the 22 chlorinated pesticides in Methods 8081, CLP, and 608.**
- **Less than 25 minute analysis time.**
- **High thermal stability with low bleed.**
- **Excellent inertness.**
- **Unsurpassed performance for the 508 pesticides and the 8151 herbicides.**

For years, environmental laboratories have struggled with various chlorinated pesticide analytical methods. Keeping track of resolution requirements and breakdown performance criteria while analyzing extracts containing high-boiling contaminants is not easy. With the introduction of the Rtx-CLPesticides and Rtx-CLPesticides2 columns from Restek, it is now possible to analyze the 22 common chlorinated pesticides with baseline resolution on both columns (see **Figures 1 and 2**). In addition, the analysis time is less than 24 minutes on both columns, resulting in higher throughput of samples. Since the columns exhibit baseline resolution, it is possible to combine the calibration standards (Mix A and B) for most methods, again resulting in increased throughput by decreasing the number of injections for calibration runs.

Stationary phase thermal stability and low bleed are also important column features so that sample contamination can be minimized by programming to a high temperature at the end of the analysis. The Rtx-CLPesticides and Rtx-CLPesticides2 columns have 330°C temperature limits, allowing for high temperature "bake out" to minimize the effects of high-boiling contaminants. While these contaminants don't always appear in the GC-ECD chromatogram, they can cause shifts in retention time, elevated baselines, and target compound breakdown.

The inertness of the stationary phase is important to minimize the possibility of on-column breakdown. Restek has published the details of this in previous presentations[1] and issues of *The Restek Advantage*[2]. This is most often observed in cyanopropyl capillary column stationary phases ("-1701") which very often provides the best resolution between target compounds, but suffers from poor inertness and reduced thermal stability. Both the Rtx-CLPesticides and the Rtx-CLPesticides2 columns have excellent inertness and will not be prone to the on-column breakdown problems observed with the "-1701" phases.

While the performance of these columns is unmatched for the 22 chlorinated pesticides as listed in USEPA 8081, many laboratories also use the same GCs for other analyses. It is

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Rtx®-CLPesticides & Rtx®-CLPesticides2

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common for the same instruments to be used for herbicide analysis (EPA Method 8151) and the extended pesticide compound lists (Method 508). The Rtx-CLPesticides and Rtx-CLPesticides2 columns also exhibit excellent performance for these analyses as well, making them the clear choice for any pesticide/PCB/herbicide analysis.

In summary, the combination of the Rtx-CLPesticides and Rtx-CLPesticides2 columns provide unsurpassed performance for the analysis of chlorinated pesticides. They can be conditioned at the end of each analysis to remove high-boiling contaminants without degrading the stationary phase. They also give improved response and reduced breakdown of methoxychlor and DDT. By designing the stationary phase with the requirements of the method in mind, Restek is leading the way into the next era of chromatography. If you are involved with the analysis of chlorinated pesticides and want to improve your resolution capacity to analyze dirty extracts, and increase your throughput, try the Rtx-CLPesticides and Rtx-CLPesticides2 columns.

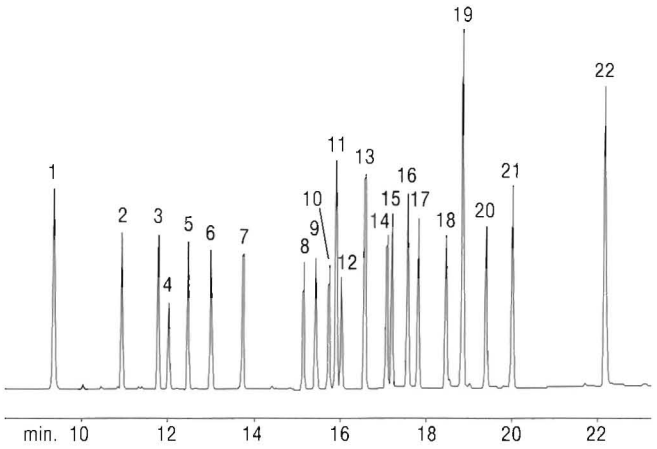
1. *1997 Pittsburgh Conference, Paper 1187.*
2. *The Restek Advantage, Summer 1996.*

Convenient, cost-saving kits are available that include both Rtx-CLPesticides columns, a guard column, and a 5-pack of Press-Tight® connectors.

Call your local distributor for more information!

Figure 1:

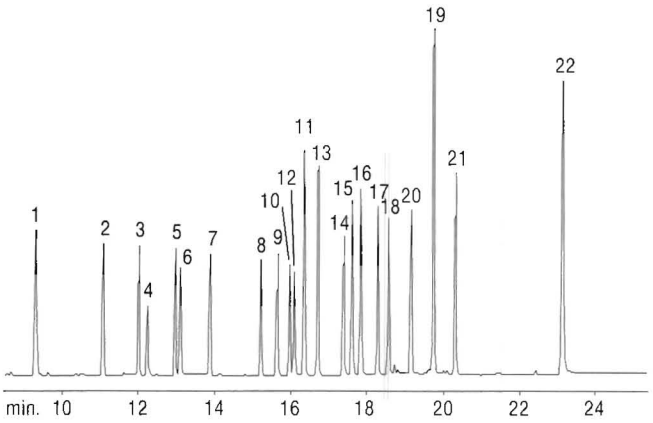
The Rtx-CLPesticides column provides baseline resolution of 22 chlorinated pesticides in EPA 8081 in less than 23 minutes.



30m, 0.32mm ID, 0.50µm Rtx-CLPesticides column (cat # 11139) run in parallel with the Rtx-CLPesticides2 column. **Oven temp.:** 120°C (hold 1min.) to 300°C @ 9°C/min. (hold 10min.); **Dead time:** 1.4 min. **Carrier gas:** He.

Figure 2:

The Rtx-CLPesticides2 column is the ideal confirmation column to the Rtx-CLPesticides column, with 4 elution order changes, baseline resolution of 22 components, and a run time of less than 24 minutes for EPA 8081 pesticides.



30m, 0.32mm ID, 0.25µm Rtx-CLPesticides2 column (cat # 11324) run in parallel with the Rtx-CLPesticides column. **Oven temp.:** 120°C (hold 1min.) to 300°C @ 9°C/min. (hold 10min.); **Dead time:** 1 4 min. **Carrier gas:** He.

Peak List for Figures 1 and 2

1. 2,4,5,6-tetrachloro-m-xylene
2. α-BHC
3. γ-BHC
4. β-BHC
5. δ-BHC
6. heptachlor
7. aldrin
8. heptachlor epoxide
9. γ-chlordane
10. α-chlordane
11. 4,4'-DDE
12. endosulfan I
13. dieldrin
14. endrin
15. 4,4'-DDD
16. endolsulfan II
17. 4,4'-DDT
18. endrin aldehyde
19. methoxychlor
20. endosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl

Product Listing:

New **Rtx-CLPesticides2 Column**

Description	Cat.#
30m, 0.25mm ID, 0.20µm	11323
30m, 0.32mm ID, 0.25µm	11324
30m, 0.53mm ID, 0.42µm	11340
Original Rtx-CLPesticides Columns	**Cat.#**
30m, 0.25mm ID, 0.25µm	11123
30m, 0.32mm ID, 0.50µm	11139
30m, 0.53mm ID, 0.50µm	11140

Restek Breathes New Life into Gas Analysis with the new Rt-Msieve 5A PLOT Column

by Andy Schuyler

Restek's novel Rt-Msieve 5A porous layer open tube (PLOT) column is now available for the separation of the permanent gases (e.g. hydrogen, argon, oxygen, nitrogen, methane and carbon monoxide) commonly analyzed in natural gas and petrochemical samples. Unlike packed molecular sieve columns, PLOT columns have fast analysis times and can baseline resolve difficult to separate components like argon and oxygen without cryogenic cooling of the column oven. This critical resolution, demonstrated in **Figure 1**, is easily obtained at 30°C in less than 7 minutes.

PLOT columns are an excellent choice for fast separations of permanent gases and light hydrocarbons because they provide the high efficiency and retention required without the high pressures, wide peaks and long analysis times encountered with packed columns. Gas analysis systems often use multiple columns and valve switching, so it is critical that the micron-sized particles coating the inside of the PLOT column are completely immobilized. Restek has developed a series of revolutionary column coating and particle adhesion technologies that produces PLOT columns with no particle generation (which can cause detector noise and permanent damage to delicate column switching valves) and consistently high efficiency and retention time reproducibility.

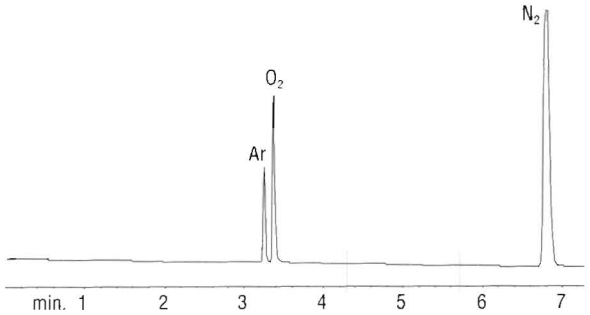
Selection of PLOT columns for the analysis of gases couldn't be easier now that Restek offers a fully re-engineered product line of PLOT columns including the Rt-Alumina, Rt-Msieve 5A and 13X and several porous polymer columns such as Rt-Q PLOT and Rt-S PLOT. The Rt-Msieve 5A is ideal for the separation of Hydrogen/Helium, Ar/O_2, and noble gases. It is also an excellent choice for rapid separation of permanent gases in refinery gas or natural gas as shown in **Figure 2**. Note the excellent resolution, peak symmetry, and the rapid analysis time of less than 4 minutes.

Restek's re-engineered PLOT columns incorporate the latest technological advances in column coating and particle binding to provide affordable, high quality columns without particle generation. These columns are rugged, easy to use, and provide excellent separations in less time than conventional columns.

For a complete listing of PLOT columns, see Restek's *1998 Chromatography Products Catalog* or call your local distributor for technical support.

Figure 1:

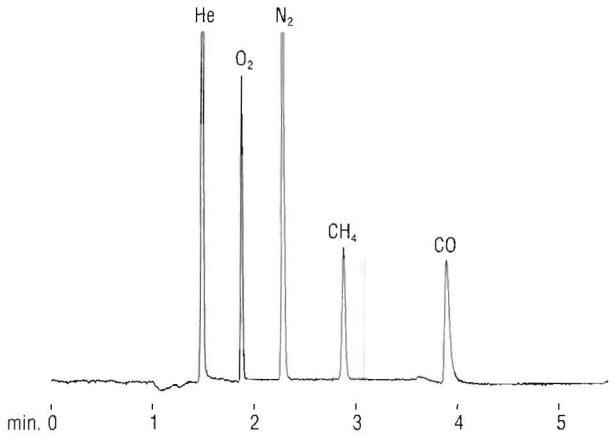
The Rt-Msieve 5A provides baseline resolution of Argon/ Oxygen in Air in under 7 minutes without cryo-cooling.



30m, 0.32mm ID Rt-Msieve 5A PLOT column (cat # 19722). **Column temp.:** 30°C; **Carrier gas:** hydrogen; **Linear velocity:** 30 cm/sec.; **Detector:** MicroTCD.

Figure 2:

The Rt-Msieve 5A resolves permanent gases in refinery gas, natural gas, and transformer oil gas.



30m, 0.53mm ID Rt-Msieve 5A PLOT column (cat # 19723). **Column temp.:** 100°C; **Carrier gas:** hydrogen; **Linear velocity:** 30 cm/sec., **Injector temp.:** 30°C; **Detector:** TCD. 150°C; **Attenuation:** 8; **Range:** 0.05 AFS.

Product Listing:

Rt-Msieve 5A Columns

great value!

Description	Cat.#
15m, 0.32mm ID	19720
15m, 0.53mm ID	19721
30m, 0.32mm ID	19722
30m, 0.53mm ID	19723

Get More Dirt on the Trident™ HPLC Guard Column System

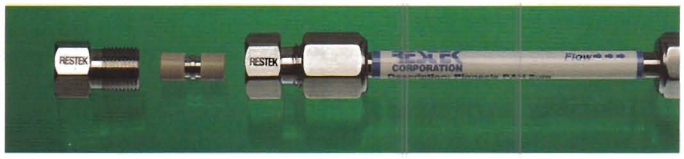
by Randy Romesberg

- **New 20 x 4mm cartridge offers twice the protection for extremely dirty samples.**
- **10 x 2mm design reduces band broadening in LC-MS.**
- **Trident's integral design eliminates troublesome tubing connections.**

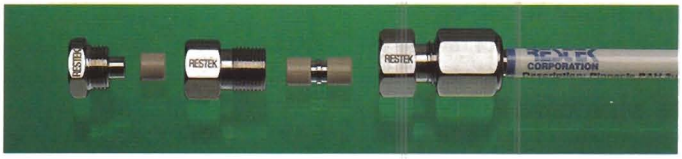
Now you can catch even more dirt on the Trident HPLC guard column system with Restek's new 20 x 4mm guard cartridge. The longer cartridge offers twice the protection as the original 10 x 4mm for extremely dirty samples. For small internal diameter columns a 10 x 2mm cartridge is now available which reduces band broadening in the small internal diameter columns used in LC-MS. The new sizes offer the same versatile configurations and convenient leak-free operation without the need for troublesome connecting tubing just like the original Trident system.

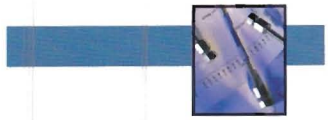
Restek's innovative Guard Column system consists of the analytical column configured with our exclusive Trident™ end fitting and XF filter fitting. This configuration contains the standard internal frit as well as a replaceable external frit, which can be easily changed without disturbing the packed bed. Changing the external frit can reverse the effects of accumulated particles, such as high back pressure or peak distortion. Add a "-700" to any Restek HPLC column catalog number to order this basic configuration.

The system can also be configured to accept an integral guard cartridge for greater protection against sample contaminants. The integral design eliminates the need for a separate holder and connecting tubing, which can cause additional band broadening. To obtain this configuration, order any Restek HPLC column (include the -700 suffix), the XG male fitting, and the appropriate pack of guard cartridges.



For maximum protection against contaminants and particulate matter, the system can be configured with both an integral guard cartridge and a replaceable external frit. To obtain this configuration, order any Restek HPLC column (include the -700 suffix), the XG-XF male fitting, and the appropriate pack of guard cartridges.





Product Listing:

Trident™ HPLC Guard Column Cartridges

3-pack Guard Cartridge (10x4mm)	3µm cat.#	5µm cat.#
Amino	911730210	911750210
Butyl	N/A*	911250210
Cyano	N/A*	911650210
Cyano Amine	918630210	918650210
EcoSep	N/A*	917150210
Methyl	911130210	911150210
Octyl	911330210	911350210
Octyl Amine	918330210	918350210
ODS	911430210	911450210
ODS Amine	918430210	918450210
PAH	917030210	917050210
Phenyl	N/A*	911550210
Phenyl Amine	918530210	918550210
SAX	N/A*	911850210
Silica	911030210	911050210
TO-11	917230210	917250210
Ultra C18	N/A*	917450210
Kromasil C4	N/A*	920250210
Kromasil C8	N/A*	920350210
Kromasil C18	N/A*	920450210
Nucleosil C8	N/A*	930350210
Nucleosil C18	N/A*	930450210
Nucleosil CN	N/A*	930650210
Nucleosil SCX	N/A*	930950210

** A 5µm guard column can be substituted.*

All cartridges listed can be used with the appropriate in-line holder!

Holder for 1cm Guard Cartridge: cat.# 25021, ea.
Holder for 2cm Guard Cartridge: cat.# 25061, ea.
XG Fitting for 1cm Guard Cartidge: cat.# 25025, ea.
XG Fitting for 2cm Guard Cartidge: cat.# 25063, ea.
XG-XF Fitting for 1cm Guard Cartridge: cat.# 25026, ea
XG-XF Fitting for 2cm Guard Cartridge: cat.# 25062, ea
Replacement XF Filter Fitting: cat.# 25024, ea.
Replacement Cap Frits: cat.# 25022, 5-pk.

Informative Technical Presentations at PittCon.

- *Development of a Unique, Stable Hydrophilic Bonded Phase for HPLC.*
  (Paper # 742, Tues., March 3, 4:05p.m.)
- *Method Optimization for the HPLC Analysis of Aldehydes and Ketones*
  (Poster # 1869P, Tues., March 3)
- *Evaluation of a Novel HPLC Cartridge Design and Column Protection System*
  (Poster #1928P, Tues.. March 3)
- *Enhanced Resolution of Parabens and Amines in Oral Solutions*
  (Paper # 1219, Thurs., March 5, 10:45a.m.)

2-pack Guard Cartridge (20x4mm)	5µm cat.#
Amino	911750220
Butyl	911250220
Cyano Amine	918650220
Cyano	911650220
EcoSep	917150220
Methyl	911150220
Octyl Amine	918350220
Octyl	911350220
ODS Amine	918450220
ODS	911450220
PAH	917050220
Phenyl Amine	918550220
Phenyl	911550220
SAX	911850220
Silica	911050220
TO-11	917250220
Ultra C18	917450220
WP Butyl	913250220

3-pack Guard Cartridge (10x2mm)	5µm cat.#
Amino	911750212
Butyl	911250212
Cyano Amine	918650212
Cyano	911650212
EcoSep	917150212
Methyl	911150212
Octyl Amine	918350212
Octyl	911350212
ODS Amine	918450212
ODS	911450212
PAH	917050212
Phenyl Amine	918550212
Phenyl	911550212
SAX	911850212
Silica	911050212
TO-11	917250212
Ultra C18	917450212
WP Butyl	913250212
WP Butyl	913250210

Grape Flavor Analysis using the New Rt-γDEXsa Column

by Sherry Sponsler

- **Gamma-cyclodextrin provides unique chiral separations.**
- **0.25mm and 0.32mm IDs are ideal for use in GC/MS flavor analysis.**
- **Thermally stable to 230°C with low bleed.**

Recent articles have demonstrated the utility of *beta*-cyclodextrin columns for flavor analysis, but sometimes the larger *gamma*-cyclodextrins provide better enantioselectivity of chiral indicating compounds. Restek's new Rt-γDEXsa column provides better separation of specific chiral compounds in grape flavor than most *beta*-cyclodextrin phases.

The enantiomeric ratios of certain chiral compounds found in flavorings can sometimes reveal adulteration. Two chiral indicators in grape flavor are methyl 3-hydroxybutyrate and ethyl 3-hydroxybutyrate. Methyl 3-hydroxybutyrate is racemic and (R)-ethyl 3-hydroxybutyrate is at least 77% predominant in natural grape flavor. Extreme alterations of these ratios can indicate that the flavor is not completely authentic.

Beta-cyclodextrin stationary phases provide enantioselectivity for a variety of chiral compounds, but not for methyl 3-hydroxybutyrate and ethyl 3-hydroxybutyrate. **Figure 1** illustrates analysis of these compounds on an Rt-βDEXsm, a 2,3-di-*O*-methyl-6-*O*-*tert*-butyldimethylsilyl-β-cyclodextrin column. The compounds tail and exhibit poor enantiomer separation.

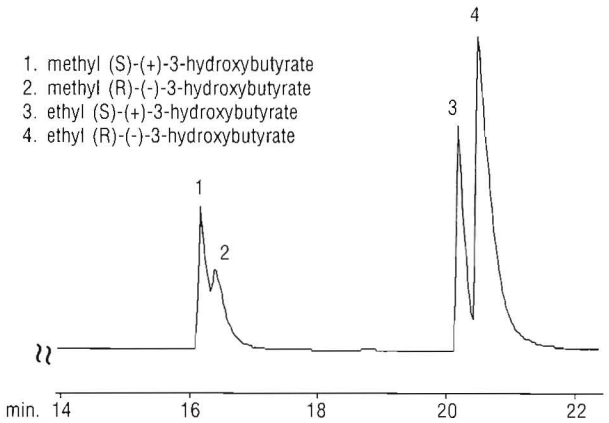
Beta-cyclodextrins with different derivatives can help increase selectivity. **Figure 2** reveals that the Rt-βDEXsa, a 2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl-β-cyclodextrin column, improves enantioselectivity for methyl 3-hydroxybutyrate but not for ethyl 3-hydroxybutyrate.

Although the exact mechanisms of compound-cyclodextrin interaction are not known, it is evident that the size of the cyclodextrin cavity is important. Perhaps some compounds may be too large to effectively interact with the cavity size of *beta*-cyclodextrin, which is composed of 7 glucopyranose units. *Gamma*-cyclodextrins are composed of 8 glucopyranose units and possess a larger cavity, which may be more interactive with larger chiral molecules.

Analysis of methyl 3-hydroxybutyrate and ethyl 3-hydroxybutyrate on the new Rt-γDEXsa column reveals excellent chiral selectivity for both, as shown in **Figure 3**.

Figure 1:

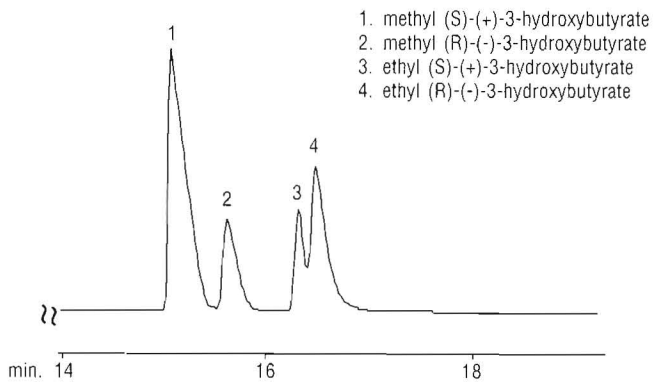
The Rt-βDEXsm column exhibits poor enantiomeric separation of methyl 3-hydroxybutyrate and ethyl 3-hydroxybutyrate in grape flavor extract.



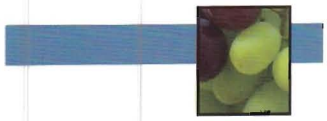
30m, 0.32mm ID, 0.25µm Rt-βDEXsm (cat # 13104). 1µl split injection of methyl and ethyl 3-hydroxybutyrate. On-column concentration: ~150ng/enantiomer. **Oven temp.:** 40°C (hold 1min.) to 200°C @ 2°C/min., **Inj. & Det. temp.:** 200°C & 230°C; **Carrier gas:** hydrogen; **Linear velocity:** 80cm/sec. set @ 40°C; **Split ratio:** 25:1.

Figure 2:

The Rt-βDEXsa column exhibits improved enantiomeric separation of methyl 3-hydroxybutyrate but not for ethyl 3-hydroxybutyrate in grape flavor extract.



30m, 0.32mm ID, 0.25µm Rt-βDEXsa (cat # 13108). 1µl split injection of methyl and ethyl 3-hydroxybutyrate. On-column concentration: ~150ng/enantiomer. **Oven temp.:** 40°C (hold 1min.) to 200°C @ 2°C/min., **Inj. & Det. temp.:** 200°C & 230°C; **Carrier gas:** hydrogen; **Linear velocity:** 80cm/sec. set @ 40°C; **Split ratio:** 25:1



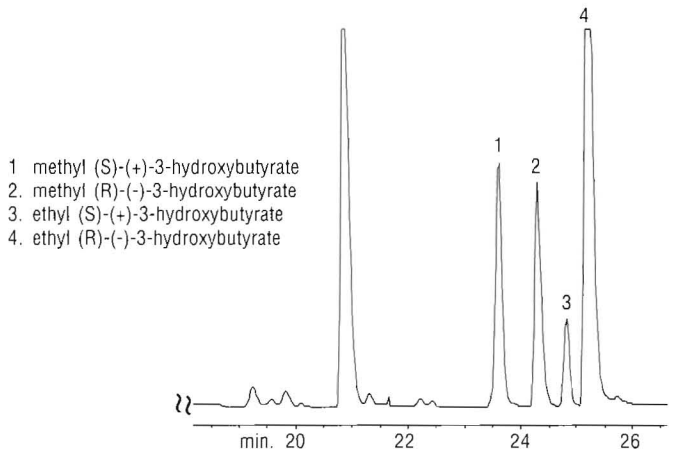
Peak shape and enantiomer separation are improved using the larger cyclodextrin molecules.

The Rt-γDEXsa contains 2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl-γ-cyclodextrins that are dissolved into cyanopropyl phenyl stationary phase (Rtx®-1701). This composition promotes thermal stability to a maximum temperature of 230°C and longevity that is comparable to other capillary columns. This column is available in 0.32 mm ID and also 0.25 mm ID for direct interfacing into a mass spectrometer. Column dimension and stationary phase stability promote utility for GC/MS analysis as well.

Beta-cyclodextrin phases can separate a variety of chiral indicating compounds in flavors, but are not effective in all applications. Using different cyclodextrin derivatives can help chiral selectivity, but going to a larger cyclodextrin sometimes is necessary. Switching from a 2,3-di-*O*-methyl-6-*O*-*tert*-butyldimethylsilyl-β-cyclodextrin to a 2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl-β-cyclodextrin column partially improved enantiomer separation for chiral indicating compounds in grape flavor. However, the 2,3-di-*O*-acetyl-6-*O*-*tert*-butyldimethylsilyl-γ-cyclodextrin column provided the best results. The new Rt-γDEXsa column can offer chiral selectivity for certain separations that are not achievable with *beta*-cyclodextrin columns.

Figure 3:

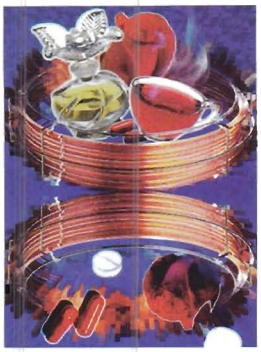
The new Rt-γDEXsa column reveals excellent chiral selectivity for both methyl and ethyl 3-hydroxybutyrate in grape flavor extract.



30m, 0.32mm ID, 0.25µm Rt-γDEXsa (cat # 13113). 1µl split injection of methyl and ethyl 3-hydroxybutyrate. On-column concentration: ~150ng/enantiomer. **Oven temp.:** 40°C (hold 1min.) to 200°C @ 2°C/min., **Inj. & Det. temp.:** 200°C & 230°C; **Carrier gas:** hydrogen; **Linear velocity:** 80cm/sec. set @ 40°C; **Split ratio:** 25:1

Call today for your free copy of Restek's *"A Guide to the Analysis of Chiral Compounds by GC"*

cat.# 59889



Product Listing:

New Rt-γDEXsa Columns

Description	Cat.#
30m, 0.32mm ID, 0.25µm	13112
30m, 0.25mm ID, 0.25µm	13113

Restek offers a wide range of cyclodextrin columns for the analysis of many chiral compounds.

Rt-βDEXsm Columns

30m, 0.25mm ID, 0.25µm	13105
30m, 0.32mm ID, 0.25µm	13104

Rt-βDEXse Columns

30m, 0.25mm ID, 0.25µm	13107
30m, 0.32mm ID, 0.25µm	13106

Rt-βDEXsp Columns

30m, 0.25mm ID, 0.25µm	13111
30m, 0.32mm ID, 0.25µm	13110

Rt-βDEXsa Columns

30m, 0.25mm ID, 0.25µm	13109
30m, 0.32mm ID, 0.25µm	13108

Rt-βDEXcst Columns

30m, 0.25mm ID, 0.25µm	13103
30m, 0.32mm ID, 0.25µm	13102

Rt-βDEXm Columns

30m, 0.25mm ID, 0.25µm	13100
30m, 0.32mm ID, 0.25µm	13101

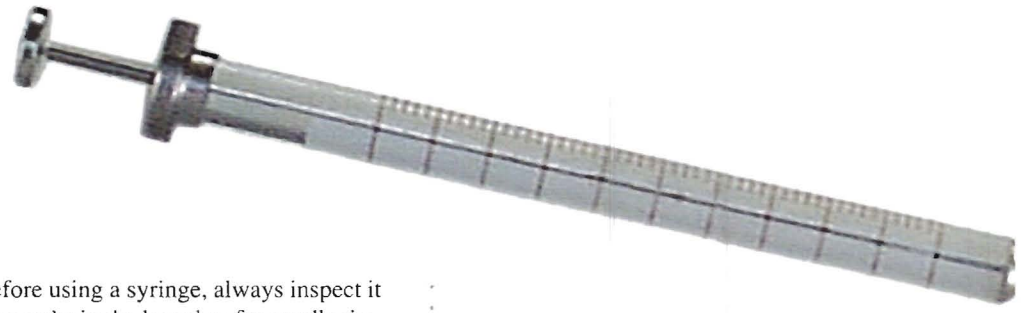
For more information on this analysis, call your local distributor and request the* "Grape Flavor Analysis using the New Rt-γDEXsa Column" *Application Note.

(cat.# 59553)

On The Care and Handli

Compiled by Doug Elliott from the Technical Lite

High quality laboratory syringes are precision fluid measuring and delivery devices. They are not designed to be disposable. Maintenance is the key factor in determining the life-time of a syringe. With proper care and handling, quality laboratory syringes will provide superior performance for many years.



Needle Gauge Size

Gauge	Nominal OD	Nominal ID
26s	.47mm	.13mm
26	.46mm	.26mm
25	.51mm	.26mm
23s	.64mm	.15mm
23	.64mm	.34mm
22s	.72mm	.15mm
22	.72mm	.41mm

Gauge	Nominal OD	Nominal ID
26s	.019 in.	.0050 in.
26	.018 in.	.0102 in.
25	.021 in.	.0102 in.
23s	.025 in.	.0060 in.
23	.025 in.	.0132 in.
22s	.028 in.	.0060 in.
22	.028 in.	.0162 in.

Before using a syringe, always inspect it for cracks in the barrel or for needle tip burrs. A cracked barrel syringe may leak or break during use and should be disposed of properly. Needle tips can be dressed by using fine emery paper, carborundum, or a soap stone to sharpen and remove burrs. Prevent sample carryover by flushing the syringe with solvent 5–20 times. *Caution! The first 2–3 washes should be discarded into a waste container and not returned to the clean solvent vessel.*

Routine Maintenance

To clean the syringe and plunger for storage it is best to use a solvent known to be effective in solvating the sample and preferably be non-alkaline, non-phosphate, and non-detergent based. Remove the plunger from the barrel, rinse with solvent and gently wipe it with a lint free tissue. Always avoid touching the plunger since any abrasions, scratches, or skin oils will often interfere with proper plunger operation. Reinsert the plunger into the barrel and pump deionized water, acetone, or suitable solvent through the needle and syringe. (Although the adhesives used to cement the needles to the syringes are chemically resistant, some solvents and cleaning conditions may deteriorate the bond, resulting in frozen plungers and plugged needles. For this reason, you should avoid immersing the syringe in the cleaning solution for prolonged times.) Allow the syringe to air dry before being placed into storage.

Tech Tip:

When reinserting a Teflon®-tipped plunger into a syringe barrel, lubricate the tip with water or a solvent compatible with the sample. Do not pump the plunger when it is dry to prevent premature wear and possible leakage. To restore the Teflon®-tipped plunger seal or to reduce excess plunger force, take advantage of Teflon's flow properties by heating the assembled syringe in an oven at 60°C for 10-20 minutes.

ıg of Analytical Syringes

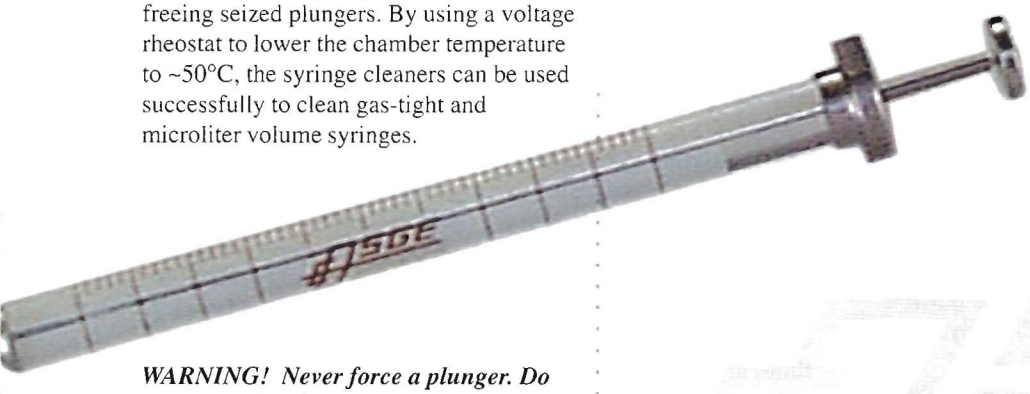
ure of Hamilton Company and SGE, Incorporated

Needle Considerations

When selecting a needle, choose the largest outside diameter effectively possible to minimize the chance of bending. The inside diameter should be kept to a minimum ('s' designation). This minimizes the needle dead volume while increasing the wall thickness to strengthen the needle without compromising the ability of the syringe to draw normal viscosity samples. Medium to high viscosity samples should be diluted prior to use or a larger ID needle should be selected.

Should a needle become clogged, do not attempt to clean it by forcing liquid or compressed air through the syringe. Excessive pressure will split the barrel. Remove the plunger from the syringe and use needle cleaning wires to dislodge any foreign material or residue build-up. Rinse with the appropriate solvent and air dry to complete the process.

Heated syringe cleaners were designed primarily for cleaning the plunger-in-needle microvolume syringes. They use a combination of heat (>350°C) and vacuum to vaporize and extract contaminants from syringe needles. Because they require an open flow to work effectively, they cannot work in cleaning plugged needles and freeing seized plungers. By using a voltage rheostat to lower the chamber temperature to ~50°C, the syringe cleaners can be used successfully to clean gas-tight and microliter volume syringes.



WARNING! Never force a plunger. Do not pump the plunger when the needle is blocked as the high pressure generated could crack the barrel. Always avoid any unnecessary movement of plungers when the syringe is dry. Damage to the inner barrel or plunger could result.

Conclusion

Today's high quality laboratory syringes are engineered devices designed for accuracy and precision. With the proper use and care they will provide years of superior performance. This performance is directly related to the cleanliness of the syringes.

You will find the most popular styles of laboratory syringes from Hamilton and SGE in Restek's 1998 Chromatography Products Guide. Special orders can be placed if there is a syringe type or service component you need that is not listed in the catalog. Please call us at 1-800-356-1688, ext. 4.

We would like to hear from you on how you keep your syringes working well for a long time. We will pass this information along to the other readers when we write about recommended sampling and injection techniques in a future article. Contact Doug by e-mail at: doug@restekcorp.com.

Practical Time Savings in Gas Chromatography Method Development

by Chris Linton, Sherry Sponsler, & Jingzhen Xu

The speed of analysis in capillary gas chromatography can be significantly improved by reducing the bore size of the column (<100μm). Researchers have successfully produced chromatograms on the order of milliseconds using this approach. Unfortunately, heavy demands are placed on sample introduction and peak detection which are not within the realm of capabilities of common instrumentation. The following discussion concentrates on practical time reduction in gas chromatographic separations using standard diameter columns and sample introduction with the Hewlett-Packard 5890 GC.

Theory

Selectivity, efficiency and time are interdependent in chromatography. Theoretically we can predict the minimum time required to maximize selectivity and efficiency.

Considering:

Tp = the time to get a solute past 1 plate
Nreq = the number of plates required for resolution
Tr = retention time required for desired resolution
H = height equivalent to a theoretical plate
μ = linear velocity
k' = partition coefficient
α = selectivity ratio
1+k' = the amount of time a solute spends in the stationary phase
R = resolution

$Tr = Nreq*Tp$
$Tp = (1+k'/\mu)H$

so: $Tr = Nreq(1+k')(H/\mu)$

substituting:

$Nreq = 16R^2(\alpha/\alpha-1)^2(1+k'/k')^2$ Purnell equation

so: $Tr = 16R^2(\alpha/\alpha-1)^2[(1+k')^3/(k')^2](H\mu)$
solve for the first derivative holding all values constant except k'

$Tr = C(k'^3 +3k'^2+ 3k'+1)/k'^2)$

$dTr/dk' = C - 3Ck'^{-2} - 2Ck'^{-3}$

in order to find Tr minimum, dTr/dk' must be set to 0

$k'^3 - 3k' - 2 = 0$ $k' = 2$ or -1

Theoretically, the optimum time for a solute to exit a column is at k'=2. At this time, selectivity and efficiency are optimized. Unfortunately, this model breaks down if a multicomponent sample is considered (all analytes cannot have k'=2). In reality, column length, initial starting temperature and temperture programming can be utilized to adjust retention times close to the theoretical optimum k'=2. **The stationary phase must provide adequate retention (film thickness) and selectivity for the resolution requirements of the separation at hand.**

Temperature Programming

Sample mixtures may contain analytes that vary in volatility to the extent where temperature programming becomes essential in the analysis. A temperature program can be selected such that high volatility compounds elute at low column temperature and low volatility compounds elute at higher column temperature ensuring the minimization of k' values. Program rates may be as high as 25 degrees per minute or greater in order to achieve near optimum k' values.

Another important aspect of temperature programming and column selection is the requirement that all analytes elute during the temperature program. This general rule helps to define column length and limits analysis time and band broadening by preventing isothermal elution of low volatility analytes. Since the temperature program was not optimized for the 45-meter column in **Figure 1**, the last five components were forced to elute under isothermal conditions.

The Limitation of Length

Longer columns generally provide greater challenge for the generation of time savings in gas chromatography. Stationary phases have inherent maximum operating temperatures which in turn limits the the extent of a temperature program. **Therefore, a minimum length of column should be chosen to provide not only the minimum required plates but the shortest retention times around the optimum k' value of 2. The stationary phase must provide adequate retention(film thickness) and selectivity for the resolution requirements of the separation at hand.** The following set of chlorinated pesticide chromatograms illustrates the differences between a near optimized separation on a 15-meter column and the same chromatographic conditions using a 45-meter column. Notice that the retention times are nearly half on the 15-meter column and all components are fully resolved.

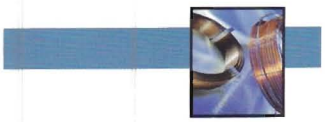
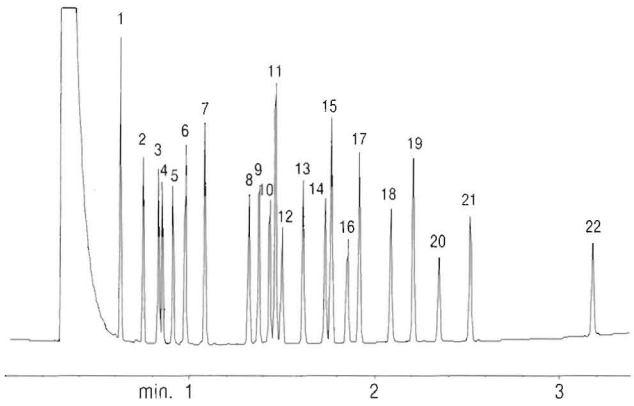
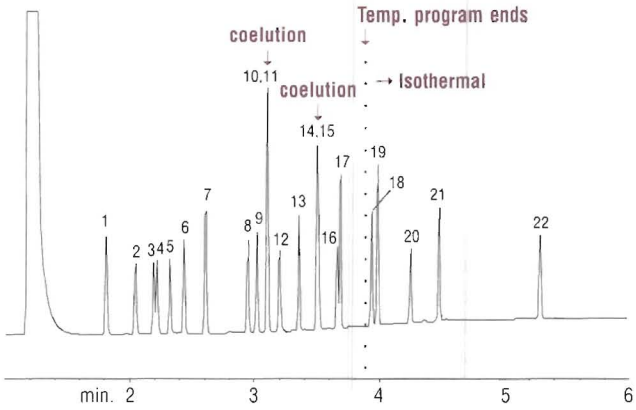


Figure 1:

Under optimized conditions, a shorter column can provide improved resolution and decreased analysis time compared to a longer column.



15m, 0.25mm ID, 0.25µm Rtx-CLPesticides column.
Oven temp.: 200°C to 300°C @ 25°C/min. **Carrier gas:** hydrogen.



45m, 0.25mm ID, 0.25µm Rtx-CLPesticides column.
Oven temp.: 200°C to 300°C @ 25°C/min. **Carrier gas:** hydrogen.

Initial Starting Temperature

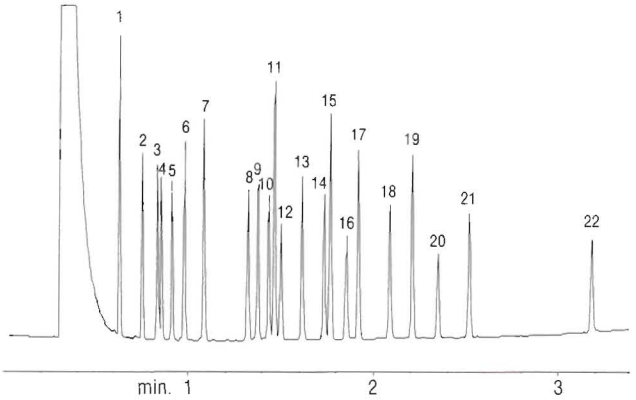
In temperature programming and isothermal gas chromatography, a time savings may be generated by chosing an initial starting temperature which allows for the elution of the first analyte near the solvent front. Starting at initial temperatures which are too low creates an empty gap in time which does not make sense theoretically due to k' elevation. The chromatograms in **Figure 2** illustrate the retention time differences generated by starting temperatures of 40°C and 200°C in the chlorinated pesticide analysis.

Peak List for Figures 1 & 2

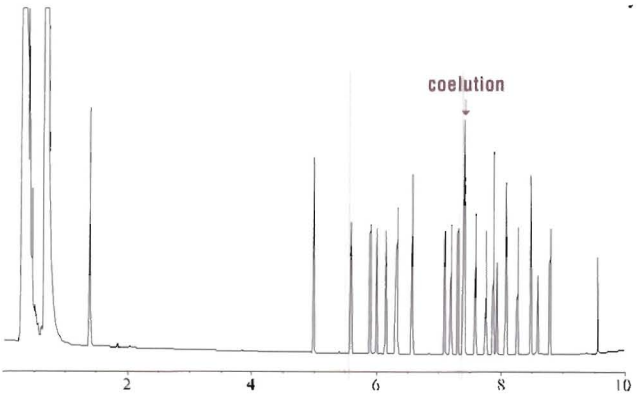
1. 2,4,5,6-tetrachloro-m-xylene
2. α-BHC
3. γ-BHC
4. β-BHC
5. δ-BHC
6. heptachlor
7. aldrin
8. heptachlor epoxide
9. γ-chlordane
10. α-chlordane
11. 4,4'-DDE
12. endosulfan I
13. dieldrin
14. endrin
15. 4,4'-DDD
16. endolsulfan II
17. 4,4'-DDT
18. endrin aldehyde
19. methoxychlor
20. endosulfan sulfate
21. endrin ketone
22. decachlorobiphenyl

Figure 2:

At higher initial temperatures, analysis times can be decreased without sacrificing resolution.



15m, 0.25mm ID, 0.25µm Rtx-CLPesticides column.
Oven temp.: 200°C to 300°C @ 25°C/min. **Carrier gas:** hydrogen.



15m, 0.25mm ID, 0.25µm Rtx-CLPesticides column.
Oven temp.: 40°C to 300°C @ 25°C/min. **Carrier gas:** hydrogen.

Practical Time Savings in Gas Chromatography Method Development

Continued from page 11.

Choosing a Carrier Gas

Figure 3 illustrates the importance of using hydrogen carrier gas to create time savings in gas chromatography. Notice the slopes of the van Deemter plot curves for each of the represented gases. Hydrogen can be used at much higher speeds without significant increases in HETP. Therefore, hydrogen carrier can generate more plates per time than nitrogen or helium making it the time saving carrier gas of choice. As an example, the chromatogram to the left in Figure 1 was generated at approximately 80cm\sec., which is twice the theoretical optimum for hydrogen carrier.

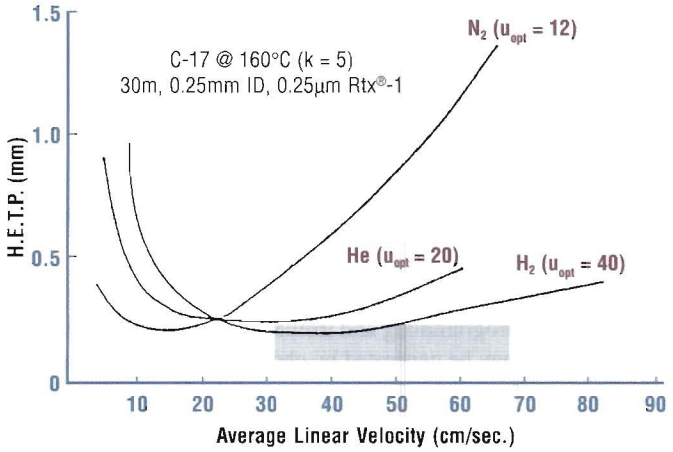
Conclusion

The preceeding discussion was presented in order to provide some practical guidelines in gas chromatography method development in a fashion that may help the analyst save time—our most precious resource. The relationship of selectivity, efficiency and time must be realized by the analyst allowing one to choose the appropriate column dimensions, stationary phase, and carrier gas.

In theory, it is fairly easy to derive optimum retention in gas chromatography. Unfortunately, real world samples provide many challenges resulting in deviation from theoretical calculations. The separation of multicomponent mixtures will always require a selective stationary phase and thoughtful selection of chromatographic parameters such as initial starting temperature and temperature programming. The culmination of these ideas should be useful to the analyst concerning practical time reduction in capillary gas chromatography.

Figure 3:

The van Deemter plots demonstrate the benefits of using hydrogen carrier gas at increased speeds and low HETP values.



Chromatography Reference Books

Modern Practice of Gas Chromatography, 3rd Edition

(Edited by Robert L. Grob, Villanova University)

A book for both beginners and specialists, this work covers principles, instrumentation techniques, and applications of GC.

John Wiley & Sons, Inc., 1995 • 800pp.

cat.# 20464, ea.

Sampling and Analysis of Airborne Pollutants

(Eric D. Winegar and Lawrence H. Keith)

This book provides you with the tools, techniques, and procedures you need to understand and conduct successful sampling and analysis projects. From electro-optical remote sensing to new directions in sampling techniques, this is your guide!

1993 • 384 pp.

cat.# 20468, ea.

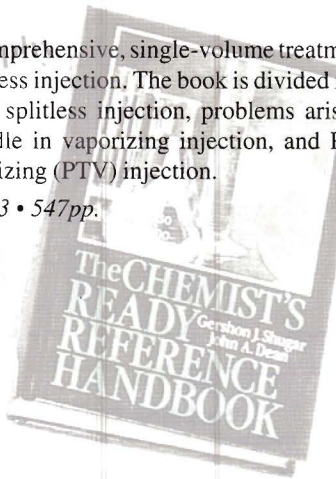
Split and Splitless Injection in Capillary GC, 3rd Edition

(Konrad Grob)

Represents one of the most comprehensive, single-volume treatment of all aspects of split and splitless injection. The book is divided into four sections: split injection, splitless injection, problems arising from the heated syringe needle in vaporizing injection, and Programmed Temperature Vaporizing (PTV) injection.

Huethig Publishing, Ltd., 1993 • 547pp.

cat.# 20451, ea.



Silcosteel® Sample Cylinders

For the accurate collection & analysis of sulfur compounds in refinery & natural gas.

Stainless steel sample cylinders are commonly used in the collection and analysis of refinery and natural gas samples. These samples often contain trace amounts of sulfur containing compounds such as hydrogen sulfide, mercaptans and disulfides. Sulfur compounds in refinery gas can interfere with reactions or damage catalysts in many petrochemical processes. Since sulfur compounds quickly react with stainless steel surfaces, accurate determination of these active compounds is impossible when using normal sample cylinders.

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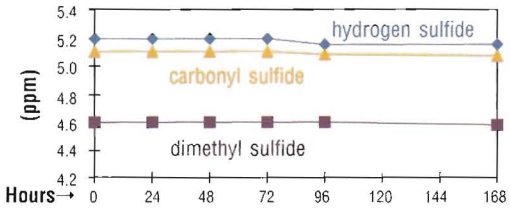
Silcosteel®-treated sample cylinders combine the inertness of glass with the strength of stainless steel and are ideal for the analysis of sulfur compounds in refinery and natural gas samples.

Figure 1:

Results indicate no significant loss of sulfur compounds when stored in a Silcosteel® cylinder for up to 7 days.



New

Sample Cylinders

- 1/4" NPT double-ended cylinders.
- Rated to 1800 psig D.O.T.

Cylinder Size	Cat.#
75cc	24271
150cc	24272
300cc	24273
500cc	24274
1000cc	24275

Silcosteel® Sample Loops

- Available in sizes from 5µl to 5mls.
- For GC and HPLC use.
- Fittings supplied for easy connection to valve.

Prevent your analytes from being adsorbed in the sample loop of your chromatographic system by using Silcosteel®-treated sample loops from Restek.

New

Silcosteel® Sample Loops for:

GC		HPLC	
Size	Cat.#	Size	Cat.#
250µl	22803	5µl	22805
500µl	22804	10µl	22806
1ml	22801	20µl	22807
5ml	22802		

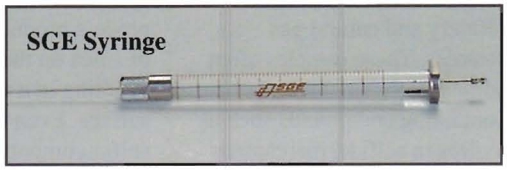
Peak Performers

Syringes for the Hewlett-Packard 7673 Autosampler

Restek now carries a full line of HP 7673 Autosampler syringes made by Hamilton and SGE. Whether it's the standard fitted metal plunger Microliter® style or the Teflon®-tipped Gastight® design, or a 0.5µl Limited Volume to 250µl Large Volume, we have the HP Autosampler syringe to meet your analytical requirements.

- Hamilton and SGE syringes are designed to meet critical autosampler specifications.
- The cone-shaped needle point style is developed to withstand multiple and fast septum injections.
- The 23s single gauge needle is the most popular size for the HP 7673 and the one that Restek stocks for same day shipment. For 26 and 23-26 gauge needle syringes please call for availability.
- 23 gauge needle fits the Merlin Microseal® Septum as well as standard septa on split/splitless and packed column injection ports.

HP 7673 10µl, Autosampler Cemented Needle (ASN)



HP 7673 Fixed Needle (F)

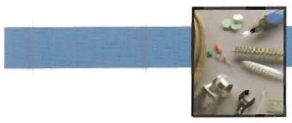
Single Syringes			HAMILTON			SGE		
	Volume (µl)	Needle Termination	Hamilton Model	Hamilton cat.#	Restek cat.#	SGE Model	SGE cat.#	Restek cat.#
Standard Microliter® Syringes								
New	0.5	R	7000-5	86276	24895	0.5BR-HP-.63	000410	24791
	5	ASN/F	75	87987	20168	5F-HP-.63	001810	24781
	5	ASRN/R	75	87957	20172	5R-HP-.63	001815	24793
	10	ASN/F	701	80387	20167	10F-HP-.63	002810	24785
	10	ASRN/R	701	80357	20171	10R-HP-.63	002815	24795
Gastight® Syringes								
	5	ASN	175	80074	24893	—	—	—
	10	ASN/F	1701	80080	24894	10F-HP-GT-.63	002812	24789
	10	ASRN/R	1701	80087	24896	10R-HP-GT-.63	002818	24797
New	25	R	—	—	—	25R-HP-.63	003665	24798
New	50	R	—	—	—	50R-HP-.63	004665	24799
New	100	R	—	—	—	100R-HP-.63	005665	24800
New	250	R	—	—	—	250R-HP-.63	006665	24801
Six-Packs								
	5	ASN/F	75	87990	20170	SK-5F-HP-.63	001814	24783
	10	ASN/F	701	80390	20169	SK-10F-HP-.63	002814	24787
	10*	ASN/F	701*	80391	24600	—	—	—
	10**	ASN	701**	80389	24599	—	—	—

** 23-26 gauge needle; ** 26 gauge needle.*

For large volume injections!

SYRINGE SPECIFICATIONS:	
Needle Length:	1.71"/44mm
Needle Gauge:	23s
Point Style:	HP/Cone
ASN/F:	Fixed, cemented needle
ASRN/R:	Removable needle

See pages 289-302 in Restek's 1998 Chromatography Products Catalog for our complete line of laboratory syringes.



The Ultimate Autosampler Vial

- Larger opening eliminates syringe needle damage.
- Step insert profile precisely aligns any insert.
- Graduated marking spot makes labeling easy.
- Lower cost than most standard vials.
- Includes patented Snap Seal™ closure design.

2.0mL, 11mm Crimp Top Vial

Are you confused about autosampler vials? With so many features available, it's often difficult to choose the feature you need most. Restek has simplified the selection by offering the ultimate autosampler vial.

Compare the popular 2 ml, 11mm Crimp Top Autosampler vial (shown) to the one you are currently using. You get all the benefits of these special features at no additional cost. Your ordering will be easier, faster, and more cost effective.

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Crimp Top Vial, Snap Seal™ Style (12 x 32mm, 11mm Crimp)

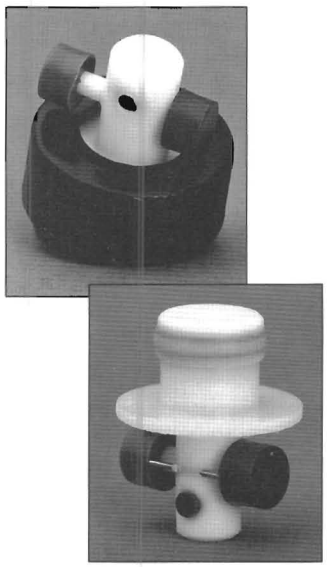
Description	100-pk.	1000-pk.
2.0mL Clear Vial w/ White Graduated Marking Spot*	24383	24384
2.0mL Amber Vial w/ White Graduated Marking Spot*	24385	24386

** Blue, green, rust, or yellow colored marking spots are available on request.*

See pages 279-288 in Restek's 1998 Chromatography Products Catalog for our complete listing of sample vials.

VICI Precision Sampling Mininert Valves for Vials

- Convenient for repetitive sampling.
- Push/push on/off.
- Valve limits sample exposure to the silicon & septum.



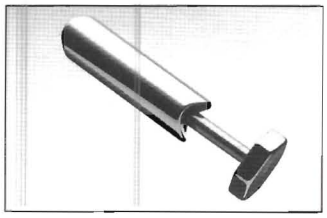
Models are available for screw-cap and crimp-top vials. The screw-cap Mininert is available in 13, 15, 18, 20, and 24 mm cap sizes. The crimp-top valve for 20 mm ID glassware slides into the neck of the vial and features a threaded flange which is turned to secure a tight fit.

VICI Precision Sampling Mininert Valves for Vials

Cap Size	Thread Size	12-pk.
13mm Screw Cap	13 mm-425	24900
15mm Screw Cap	15 mm-425	24901
18mm Screw Cap	18 mm-400	24902
20mm Screw Cap	20 mm-400	24903
24mm Screw Cap	24 mm-400	24904
20mm Crimp Top	—	24905

Replacement Septa and Septum Installation Tool

These silicone septa fit all Mininert valves. The installation tool is a handy device for quickly removing and replacing needle seal septa.



Replacement Septa (50-pack): 24906

Septum Insertion Tool: 24907

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