THE

RESTEK

ADVANTAGE

A Thin Film (0.25µm) 0.53mm ID Rtx®-1701 Provides the Best Overall Resolution of Pesticides in the Least Amount of Time

In March of 1990, the EPA's Contract Lab Program (CLP) Statement of Work was revised to allow the use of 0.53mm ID capillary columns for the analysis of pesticides and Aroclors®. All subsequent statements of work have included this option including the 12/90 and 2/91 revisions. This method makes recommendations for the use of 1701 (cyanopropylphenyl polysiloxanes) and 608 (methyl phenyl polysiloxane) capillary columns, and has a provision for allowing equivalent columns if they meet the resolution and calibration requirements. The method also makes recommendations for run conditions to be used with both columns. However, many labs following these recommendations find it difficult to meet the resolution criteria specified in the method. To reduce analysis time, many labs prefer to run both columns simultaneously using the same run conditions. This is difficult because the optimum run conditions for one column may not be the optimum for the other column.

Selecting a single set of run conditions that will be appropriate for both columns can be a time consuming task. Using $ezGC^{m}$, a new methods development software program, can simplify the job of selecting the optimum run conditions that will work

in	this	issu	ıe
-	imizing C		ide
Ana	lyses with	ezGC"	1
DRO	O Analysis		4
Stan	dards Sp	otlight	7
Clin	ical Corn	er	8
Imp	roved Rtx	®-2330	10
Hint	ts for the (Capillary	
	omatogra		12
Peak	« Perform	ers	14
New	s from Re	stek	16

for both capillary columns. In addition, $ezGC^m$ will insure that the analysis can be achieved in the fastest possible time, thereby reducing analysis costs even further.

EPA CLP pesticide method requirements

The EPA's CLP Statement of Work for pesticides and Aroclors® specifies very strict calibration, resolution, and analytical sequence requirements for the method.

Resolution requirements state

Table I - Resolution C	heck Mixtures, must l	be ≥ 60% resolved
γ-chlordane methoxychlor dieldrin	endrin ketone p,p'-DDE decachlorobiphenyl	endosulfan I tetrachloro-m-xylene endosulfan sulfate
	rformance Evaluation ist be 100% resolved	Mixture,
γ-BHC methoxychlor β-BHC	endrin p,p'-DDT decachlorobiphenyl	α-BHC tetrachloro-m-xylene
	idual Standard Mix st be ≥ 90% resolved	
Mixture A	Mixture	В
α-BHC heptachlor γ-BHC endosulfan I dieldrin endrin p,p'-DDD p,p'-DDT methoxychlor tetrachloro-m-xylene decachlorobiphenyl	β-BHC δ-BHC aldrin heptachlor epoxide α-chlordane γ-chlordane decachlorobiphenyl	p,p'-DDE endosulfan sulfate endrin aldehyde endrin ketone endosulfan II tetrachloro-m-xylene

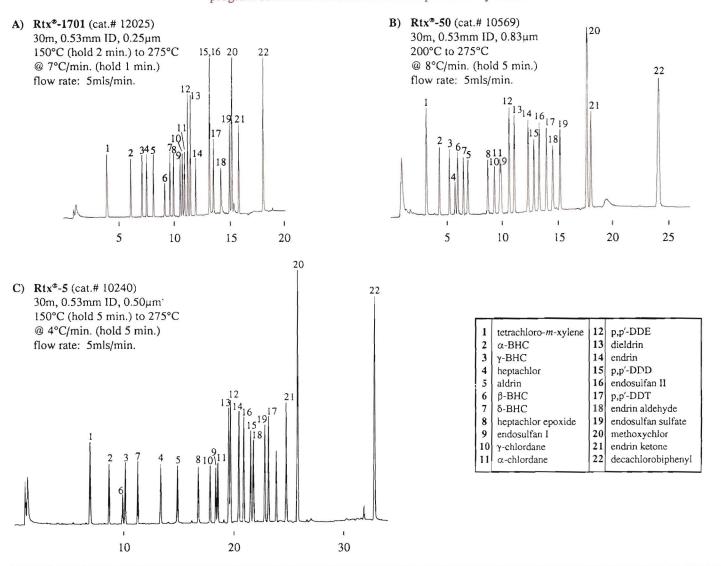
that all components in the Resolution Check Mixture must be greater than or equal to 60% resolved, all components in the Performance Evaluation Mixture must be 100% resolved, and components in the individual pesticide mixtures A or B must be greater than or equal to 90% resolved. Tables I, II, and III list the components in the resolution check mixture, performance evaluation mixture, and the individual pesticide mixtures, respectively.

The resolution criteria specified in the method must be met by both columns used in the analysis. The method recommends the use of 0.53mm ID capillary columns with 1701 (cyanopropyl phenyl polysiloxane) and 608 (phenyl methyl polysiloxane) stationary phases, but equivalent columns can be used if they meet the requirements for resolution and initial calibration. In order to meet the resolution criteria, $ezGC^m$ software

Australian Distributors

www.chromtech.net.au

Figure 1 - Resolution criteria of CLP pesticides are exceeded using optimized temperature program conditions for each column as predicted by $ezGC^{m}$.



was used to optimize column selection and run conditions for the analysis of chlorinated pesticides specified in the EPA's CLP.

How does ezGC™ work?

The first step in optimizing the run conditions is to determine the retention of CLP pesticides using both a slow (4°C/min.) and a fast (12°C/min.) temperature program ramp on each column. The retention data for the two analyses on each column type is entered into $ezGC^{m}$ along with the peak identifications. The software will then optimize the operating parameters such as initial temperature, initial hold time, program rate, final temperature, and final hold time. The software will list the 50 best run parameters in order of shortest analysis time. The list generated for each column type can be compared to determine what set of run parameters, with the shortest analysis time, will work for both columns. Other parameters such as flow rate, column length, ID, and film thickness can also be varied to determine if changing these

parameters will improve resolution or reduce analysis time. A listing of elution order, retention time, and resolution for the components can be obtained for any of the 50 conditions. In addition, a simulated chromatogram can also be printed.

The results of the $ezGC^{TM}$ optimization of the CLP pesticide analysis shows that the three columns and optimized temperature program conditions in Figure 1 exceed the resolution requirements of the method.

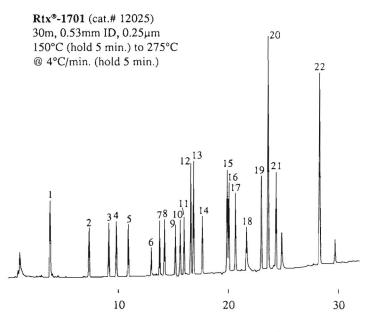
Of the three column choices, the Rtx®-1701 gives the fastest analysis time of the pesticides in 18 minutes, but yields a complete coelution of p,p'-DDD/endosulfan II (Figure 1A). Originally a 0.5µm Rtx®-1701 was used but a computer analysis using $ezGC^{\text{TM}}$ demonstrated that the 0.25µm Rtx[®]-1701 column would provide a faster analysis time and better resolution than the 0.5µm film. In addition, a 0.25µm column will exhibit better thermal stability and less bleed than the 0.5µm Rtx®-1701, making it clearly the column of choice. The

Rtx*-50 also meets and exceeds the resolution criteria with analysis times of 24 minutes, but gives only 20% resolution of α -chlordane/endosulfan I (Figure 1B). The Rtx*-5 gives the best resolution of the CLP pesticides, but has an analysis time of 30 minutes (Figure 1C).

Column choice and recommended run conditions for the simultaneous analysis of CLP pesticides

The CLP Statement of Work also makes recommendations for simultaneous dual column analysis. In dual column confirmational analysis, a sample is run on two columns of differing polarity, yielding two separate chromatograms from one injection. Simultaneous dual column analysis both improves qualitative analysis and sample throughput in the lab. However, finding the optimum temperature program to use on both columns is a difficult, time consuming task. Since the optimum temperature program conditions for the Rtx[®]-1701, -50, and -5 were all different as seen in Figure 1, we used ezGC[™] to predict a simultaneous optimum temperature program for a dual column configuration. Figure 2 shows the results of the simultaneous optimization. The optimized dual column system is made up of the Rtx®-5 (30m, 0.53mm ID, 0.50µm) and the Rtx®-1701 (30m, 0.53mm ID, 0.25µm) and can be run simultaneously at 150°C (initial hold 5 min.) to 275°C @ 4°C/min. (hold 5 min.). All resolution criteria are maintained in the simultaneous dual column confirmational system and analysis is complete in 30 minutes. In addition, neither column shows coelutions under the simultaneous optimum temperature program chosen.

Figure 2 - Simultaneous dual column confirmation of CLP pesticides can be obtained using an Rtx®-5/Rtx®-1701 column combination.



Please refer to Figure 1C for the Rtx®-5 chromatogram and peak identifications.

Problems associated with pesticide analyses

The analytical sequence outlined in the CLP protocol specifies that an injection of the instrument blank and individual standard mixtures A & B be run after every 12 hours to maintain GC calibration. After 24 hours, an instrument blank and performance evaluation mixture must be run to verify calibration. These two steps are repeated at 12 and 24 hour intervals. Additionally, the breakdown of DDT and endrin in the performance evaluation mixtures must be less than 20% and the combined breakdown of DDT and endrin must be less than 30% to maintain system inertness.

Typically, system calibration and inertness are not met when the GC system and column become contaminated from dirty samples. In the $ezGC^{rM}$ optimization of CLP pesticides, the resolution criteria within the software was set to exceed the method requirements (100 vs 60%) to permit longer analytical sequences before contamination affects resolution and inertness and GC maintenance is required. An Rtx®-1701 (30m, 0.53mm ID, 0.25μm), Rtx[®]-50 (30m, 0.53mm ID,0.83μm), or Rtx®-5 (30m, 0.53mm ID, 0.50µm) column can be used to analyze CLP pesticides. The resolution criteria specified in the method is met on any one of these three columns. However, we found that the Rtx®-1701/Rtx®-5 combination offers the best resolution in the least amount of time for dual column simultaneous analysis. (Note - for further information about dual column simultaneous analysis or using 0.53mm ID columns in the direct injection mode, request Restek's Guide to Direct/Oncolumn Flash Vaporization Injection.)

Product Listing

Rtx*-5 30m, 0.53mm ID, 0.50μm, cat.# 10240, \$445 Rtx*-1701 30m, 0.53mm ID, 0.25μm, cat.# 12025, \$445 Rtx*-50 30m, 0.53mm ID, 0.83μm, cat.# 10569, \$495

Resolution Check Mix,

cat.# 32001, \$30 each 32001-500, \$60 ea. w/data pack 32101, \$270 l0pk. w/data pack

Performance Evaluation Mix,

cat.# 32002, \$30 each 32002-500, \$60 ea. w/data pack 32102, \$270 10pk. w/data pack

Pesticide Mix A,

cat.# 32003, \$30 each 32003-500, \$60 ea. w/data pack 32103, \$270 10pk. w/data pack

Pesticide Mix B,

cat.# 32004, \$30 each 32004-500, \$70 ea. w/data pack 32104, \$270 10pk. w/data pack

Pesticide Surrogate Mix,

cat.# 32000, \$25 each 32000-500, \$45 ea. w/data pack 32100, \$225 10pk. w/data pack

For more information on ezGC™ Method Development Software, please call technical service at 1-800-356-1688, ext. 4.

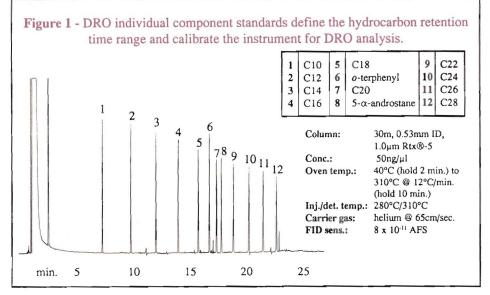
Analysis of Diesel Range Organics in Soil and Water

In the March 1993 issue of *The Restek Advantage*, we published an article on the analysis of Gasoline Range Organics (GRO). This is a follow up article that focuses specifically on the analysis of Diesel Range Organics (DRO) and discusses how to avoid the common problems associated with this analysis.

As in GRO analysis, many states have adopted their own testing methodology for measuring DRO. In general, DRO methods measure the concentration of diesel fuel organics ranging from C10 - C28 in water and soil. DRO methods are designed to measure mid-range petroleum products such as diesel fuel, home heating oil, gas oil, jet fuel and kerosene. The typical DRO analytical method involves a liquid/liquid

extraction with methylene chloride and Kuderna-Danish concentration of the extract. One microliter of the extract is then injected onto a megabore capillary column using a flame ionization detector (FID)¹. Water samples are extracted as specified in SW-846 Method 3510 (separatory funnel liquid/liquid extraction) or Method 3520 (continuous liquid/liquid extraction). Soil samples are extracted using Method 3550 (sonication). The FID responds to all hydrocarbon species and is used to detect the total petroleum hydrocarbons (TPH). A wide range of columns can be used for DRO analysis, depending upon the requirements specified in each state's analytical procedure. In general, the column, operating under the conditions of the method, must meet some minimum requirements for retention and resolution.

To determine TPH, the sum of the area under a range of hydrocarbons is used. States may differ on which hydrocarbons are used to define the beginning and end of the diesel compounds to be measured, but typically begin with C10 or C12 and end with C24 or C28. Integration begins 0.1 minutes before the retention time of the first hydrocarbon marker and ends 0.1 minutes after the last hydrocarbon marker in the component standard. Most methods have specific performance requirements. Examples of method performance requirements include: 1) the beginning hydrocarbon must be completely resolved from the solvent peak or, 2) internal and surrogate standards must be resolved from the hydrocarbons. Most methods also employ a discrimination check to test for molecular weight discrimination. When analyzing such a wide range of hydrocarbons, molecular weight discrimination of the later eluting hydrocarbons is a common problem. Discrimination can occur from poor volatility in the injection port and/or syringe needle discrimination. Due to the higher hydrocarbon range and GC oven temperatures required for DRO analysis, a 30 meter, 0.53mm ID, 1.0µm Rtx®-5 is an excellent column to



meet the resolution and performance requirements of the methods.

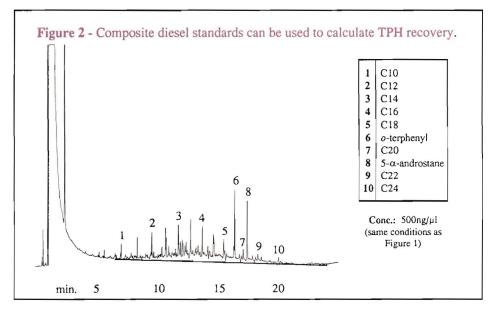
Determining diesel retention range and calibrating response

Since reporting TPH requires the summation of all chromatographic peaks eluting between the first and last component specified in the method, the calibration standard must contain the components defining the retention time range. Individual states differ on which compounds to use in defining the retention time range for diesel. Figure 1 shows a chromatogram of Restek's DRO individual component standard. This standard is used to determine the beginning and ending retention times of the diesel range. This standard is also used to calculate an average response factor by dividing the total area of all components by the total amount injected.

Confirmation of TPH response is achieved by analyzing a quantitative standard containing one or more diesel fuels. This composite standard was designed to be representative of diesels present in a wide range of samples to be analyzed. An example of a chromatogram generated from Restek's DRO composite diesel standard is shown in Figure 2. Once the beginning and ending retention times and response factors from the individual components have been defined, the total area of the composite standard is used to calculate the recovery for a spiked standard.

Improper integration can cause low DRO recoveries

Low recoveries, one common problem associated with DRO analysis, is usually due to errors in integration. The chromatographic profile of diesel oils does not give baseline resolution of all components, which results in a characteristic rising baseline underneath the resolved hydrocarbon components as shown in Figure 2. When calculating the recovery of DRO samples, integration should be accomplished using the baseline hold method. Baseline to baseline integration is performed by



obtaining the baseline at the beginning and end diesel retention markers and applying a horizontal hold between these two points. If valley to valley integration is used, low recoveries will result since a large portion of the total diesel response will be excluded from the integrated area.

High DRO recoveries can be caused by many factors

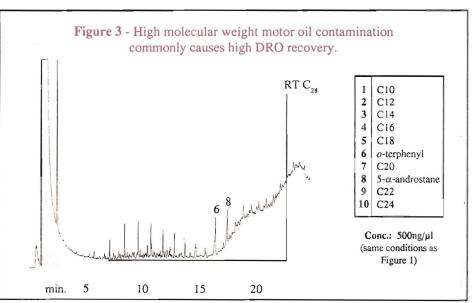
Another problem commonly seen in DRO analysis is erroneously high recoveries (>100%). One cause of high recovery is mass discrimination of high molecular weight hydrocarbons causing a low average response factor value. High recoveries can also be caused by changes in the baseline due to column bleed. Column bleed creates high DRO recovery since the background area under the diesel is increased when programming the GC oven to the higher temperatures required for DRO analysis. To minimize high recoveries from column bleed, perform a blank run prior to analysis to determine the true baseline. This true baseline can be subtracted from the DRO analysis to compensate for the additional area caused from normal column bleed. defined retention time marker of C10 to C28, an erroneously high DRO value would result. In the case of motor oil contamination, the analyst is required to make a judgment call, recognize the patterns of diesel fuel and motor oil, and determine a "cut point" for the DRO range.

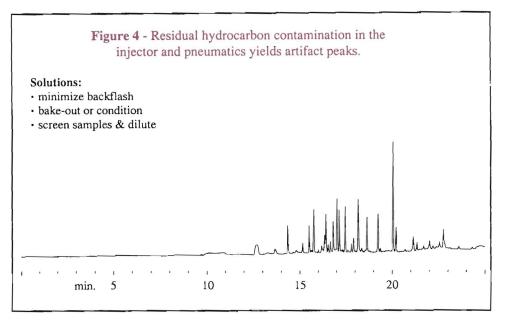
Another common problem creating high DRO recovery or false positives is memory effect. Memory effects can be caused by poor sample volatility, backflash, or carryover. Poor sample volatility is caused when high molecular weight compounds are not completely vaporized and condense in the injection port. Figure 4 (page 6) shows an example of memory effect from a previous injection of high molecular weight hydrocarbons. To increase sample volatil-

ity, an injection port temperature of 280°C to 300°C is recommended. Backflash is created when the sample vaporization cloud exceeds the sample expansion volume of the inlet sleeve. Backflash can be minimized by increasing the inlet pressure, decreasing sample size, or diluting the sample. (Refer to our Guide to Direct/On-column Flash Vaporization Injection for more information on backflash.) Carryover is typically caused when sample residue is left behind after injecting a highly concentrated sample or from high molecular weight contaminants extracted during sample preparation. When running samples, always establish a sequence that injects from low to high concentration. If a sample has an unknown concentration, make a blank run after the sample to bake-out any potential contaminants. To minimize memory effects, prescreen extracts on a separate GC using a 15-meter Rtx®-5 column, prior to final sample preparation. If there is a high concentration of contaminants, the sample should be diluted before injecting it onto the column used for final quantitation.

High molecular weight interferences can create high DRO recoveries

Another source for reported high DRO recovery is from contamination. Contamination can occur from the presence of organic or high molecular weight contaminants, such as phenols, phthalates, lubricating oils or motor oils that were also extracted during the liquid/liquid extraction step. Figure 3 shows a chromatogram of a DRO sample that contains motor oil contamination. Most diesel range organics range from C10 to C28. In Figure 3, it appears that motor oil contamination is present with a hydrocarbon range beginning near C20 and overlapping beyond C28. If an analyst was to sum the area from the





Although DRO methods may differ between states, the basic analytical procedures are similar. The recommended column for DRO analysis is a 30 meter, 0.53mm ID, 1.0μm Rtx®-5. The Rtx®-5 meets resolution requirements of DRO methods and minimizes baseline interferences from column bleed and mass discrimination. System calibration can be accomplished with mixtures of individual hydrocarbons or composite diesel standards. Baseline hold integration should be applied to yield accurate DRO recoveries and prevent low recovery. Baseline subtraction, sample prescreening, and column bake-out should be performed to minimize the potential of high recoveries.

 EPA UST Workgroup American Petroleum Institute, February 1992.

Product Listing

Rtx®-5 30m, 0.53mm ID, 1.0µm, cat.# 10255, \$445

Underground Storage Tank Monitoring Standards DRO Mix (EPA/WISC)

2000µg/ml each in 1ml methylene chloride.

 decane (C10)
 dodecane (C12)

 tetradecane (C14)
 hexadecane (C16)

 octadecane (C18)
 eicosane (C20)

 docosane (C22)
 tetracosane (C24)

 hexacosane (C26)
 octacosane (C28)

Cat.# 31064 \$25 ea.

31064-500 \$55 ea. w/data pack 31164 \$225 10 pk. w/data pack

Recommended internal standard: $5-\alpha$ -androstane Standard 2000µg/ml in 1ml methylene chloride.

Cat.# 31065 \$25 ea.

31065-500 \$35 ea. w/data pack 31165 \$225 10 pk. w/data pack

Recommended surrogate: o-terphenyl Standard

2000µg/ml in 1ml acetone.

Cat.# 31066 \$25 ea.

31066-500 \$35 ea. w/data pack 31166 \$225 10 pk. w/data pack

Diesel Surrogate Standards

Individual mixtures at 10,000µg/ml in 1ml methylene chloride.

		each	10pk.
compound	each	w/data pack	w/data pack
p-terphenyl	31095/\$25	31095-500/\$35	31195/\$225
2-fluorobiphenyl	31096/\$25	31096-500/\$35	31196/\$225
o-terphenyl	31097/\$25	31097-500/\$35	31197/\$225
1-chlorooctadecane	31098/\$25	31098-500/\$35	31198/\$225

Diesel Fuel #2 Composite Standard

5000µg/ml in 1ml methylene chloride.

Cat.# 31093 \$25 ea.

31093-500 \$35 ea. w/data pack 31193 \$225 10 pk. w/data pack

Kerosene Fuel Composite Standard

5000µg/ml in 1ml methylene chloride.

Cat.# 31094 \$25 ea.

31094-500 \$35 ea. w/data pack 31194 \$225 10 pk. w/data pack

Jet Fuel A Standard

Commercial Jet Fuel A prepared at 5000µg/ml in 1ml methylene chloride.

Cat.# 31215 \$25 ea.

31215-500 \$35 ea. w/data pack 31315 \$225 10 pk. w/data pack

Fuel Oil #4 Standard

Fuel Oil #4 is typically used in limited applications where preheating of the fuel prior to burning cannot be utilized. The fuel is a blend of distillate (Fuel Oil #2) and residual (Fuel Oil #6) to meet ASTM viscosity specifications. The Fuel Oil #4 standard offered here has a kinematic viscosity of 21.9 at 38°C (100°F). Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.# 31216 \$25 ea.

31216-500 \$35 ea. w/data pack 31316 \$225 10 pk. w/data pack

Fuel Oil #5 Standard

Fuel Oil #5 is also typically used in applications where little or no preheating of the fuel prior to burning is utilized. As with Fuel Oil #4, Fuel Oil #5 is a blend of distillate and residual fuel to meet viscosity criteria. The Fuel Oil #5 standard offered here has a kinematic viscosity of 106.5 at 38°C (100°F). Kinematic viscosity measurement performed using ASTM D-445. Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.# 31217 \$25 ea.

31217-500 \$35 ea. w/data pack 31317 \$225 10 pk. w/data pack

Fuel Oil #6 Standard

This oil, also called Bunker C or residual, is a black viscous fuel. Applications where it may be used all require the ability to preheat the fuel prior to pumping and burning. Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.# 31218 \$25 ea.

31218-500 \$35 ea. w/data pack 31318 \$225 10 pk. w/data pack

(Product Listing is continued on page 7.)



Australian Distributors Importers & Manufacurers www.chromtech.net.au

Standards Spotlight



Product Update - USP 467 Calibration Mixture

In September 1991 Restek introduced a calibration mixture for USP Method 467, Organic Volatile Impurities (cat.# 36000). Since the initial introduction, the method has been modified several times to its current approved form (1). There are however, more recent modifications being proposed at this time (2).

In our continuing effort to provide quality chemical standards and meet the ever changing method requirements, we are now discontinuing the original USP 467 Calibration Mix (#36000) and introducing two new calibration mixtures for this protocol.

In the Fifth Supplement, USP-NF (1) the requirement to analyze for trace levels of ethylene oxide was removed from

Method 467 and relegated to individual drug monographs. To meet the currently approved version of Method 467, we are offering a Revised USP 467 Mixture, cat.# 36001. Listed below are the components and concentrations of each analyte.

Revised USP 467 Mixture

Prepared in dimethyl sulfoxide, 1ml per ampul.

benzene 100µg/ml
chloroform 50
1,4-dioxane 100
methylene chloride 100
trichloroethene 100

Cat.# 36001 \$20 ea.

36101 \$180 per pack of 10 ampuls

In the most recent edition of Pharmacopeial Forum (2), additional method modifications have been proposed. While the changes have not been formally adopted, laboratories may wish to begin evaluation of the new method. These proposed changes impact the calibration mixture required. The concentration of methylene chloride under consideration is five times higher than the original level in the current protocol (500ppm vs. 100ppm). In addition, the solvent used to prepare the stock calibration standard is now methanol instead of dimethyl sulfoxide. The stock calibration standard required to evaluate the proposed method revisions is listed.

Proposed USP 467 Mixture

Prepared in methanol, 1ml per ampul.

benzene 100µg/ml
chloroform 50
1,4-dioxane 100
methylene chloride 500
trichloroethene 100

Cat.# 36002 \$20 ea.

36102 \$180 per pack of 10 ampuls

- 1) Fifth Supplement, USP-NF, Organic Volatile Impurities <467>, page 2706-2708, Nov. 15, 1991.
- 2) Pharmacopcial Forum, Volume 19, Number 3, May-June 1993, Organic Volatile Impurities <467>, page 5335-5337

(Product Listing continued from page 6.)

JP-4 Military Fuel Standard

Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.# 31219 \$25 ea.

31219-500 \$35 ea. w/data pack 31319 \$225 10 pk. w/data pack

JP-5 Military Fuel Standard

Prepared at 5000µg/ml in 1ml methylene chloride.

Cat.# 31220 \$25 ea.

31220-500 \$35 ea. w/data pack 31320 \$225 10 pk, w/data pack

DRO Mix (Tenn/Miss)

Contains the following compounds at $1000\mu g/ml$ each in 1ml methylene chloride.

decane (C10) undecane (C11) dodecane (C12) tridecane (C13) tetradecane (C14) pentadecane (C15) hexadecane (C16) heptadecane (C17) octadecane (C18) nonadecane (C19) cicosane (C20) heneicosane (C21) docosane (C22) tricosane (C23) tetracosane (C24) pentacosane (C25)

Cat.# 31214 \$30 ea.

31214-500 \$70 ea. w/data pack 31314 \$270 10 pk. w/data pack



Clinical Corner

Quantitative Drug Standards

In an effort to continue meeting the varied demands of our customers, Restek introduces the availability of DEA exempt, quantitative drug standards. These standards can be used as qualitative retention time markers, reference material for spectral identification or for spiking samples for quantitative analysis. Each standard is prepared to the same exacting criteria that is used for our environmental standards.

All raw materials and final products are subject to exhaustive QA testing using DSC, GC/FID, GC/NPD, GC/ MS, & FT-IR.

Comprehensive purity determinations

Our manufacturing process starts with extensive purity determinations for each raw material that goes into our standards. In order for raw materials to pass our quality assurance specifications, they must have a minimum purity of 98%. Drug raw materials are checked by two separate processes to verify purity. Differential Scanning Calorimetry (DSC) is used to assay the purity of all our crystalline materials. Determinations are accurate within 0.1 mole percent. Purity is also checked by performing GC-FID and GC-NPD analysis on all starting materials. These additional checks ensure that extraneous chromatographic peaks will not be present in the final product.

Final product testing

Identity confirmation is also an important part of our manufacturing process. All raw materials are checked by two separate methods to confirm their identity. Mass spectral identification

is accomplished by using GC-MS and infrared spectra are taken using FT-IR. All spectra are compared to reference spectra published in either computer libraries or reference texts.

User friendly packaging

All of Restek's drug standards are provided as quantitative methanolic solutions. Every standard is prepared using precise gravimetric techniques after the raw materials have been approved for use. Subsequently, each lot of standard is analyzed by GC-FID using an internal standard and then compared to a reference lot of the same material in order to maintain reproducibility of concentration from lot to lot.

Restek's standards are packaged in flame sealed, silanized amber glass ampuls under nitrogen to enhance product stability prior to opening. Each ampul is supplied with an ampul breaker to safely open the ampule and a silanized amber screw top vial with label for storage of the unused portion of the standard.

Certificate of analysis

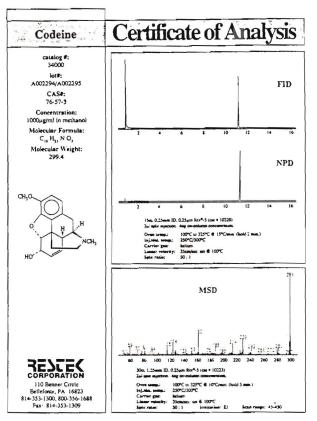
A Certificate of Analysis is supplied with each standard. This certificate includes compound structure, molecular weight and formula, DSC purity, GC-FID purity, GC-NPD purity, GC-MS and FTIR identification. The concentration as stated on the certificate is calculated from the gravimetric data. Concentrations reported are based on the weight of the free acid or free base form of the compound, with corrections being made for the salt form of the raw material. A sample of the Certificate of Analysis is shown in Figure 1.

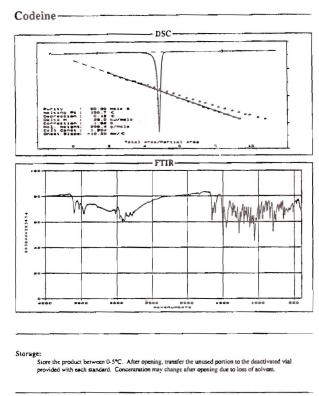
DEA exempt

These standards have been exempted by the Drug Enforcement Administration from the normal requirements for purchasing controlled substances. They can be obtained without a DEA license, phone orders can be accepted, and no additional paperwork is required with your order.

To order any Restek product, call 800-356-1688 (ext. 3). For direct technical service, call 800-356-1688 (ext. 4).







Restek Corporation - 110 Benner Circle - Bellefonte, PA 16823-8812 (814) 353-1300, (800) 356-1688, Fax: (814) 353-1309

All standards are diluted in methanol

Description	Concentration	Cat.#	Price	Description	Concentration	Cat.#	Price
alprazolam	1000µg/ml	34042	\$25	lorazepam	1000µg/ml	34051	\$25
amobarbital	1000µg/ml	34028	\$25	medazepam	1000µg/ml	34052	\$25
dextro-amphetamine	1000µg/ml	34020	\$25	meperidine	1000µg/ml	34004	\$25
aprobarbital	1000µg/ml	34029	\$25	mephobarbital	1000µg/ml	34034	\$25
barbital	1000µg/ml	34030	\$25	meprobamate	1000µg/ml	34059	\$25
benzoylecgonine	1000µg/ml	34016	\$30	methadone	1000µg/ml	34005	\$25
benzphetamine	1000µg/ml	34022	\$25	methamphetamine	1000µg/ml	34021	\$25
bromazepam	1000µg/ml	34043	\$25	methaqualone	1000µg/ml	34064	\$25
butabarbital	1000µg/ml	34031	\$25	methohexital	1000µg/ml	34035	\$25
butalbital	1000µg/ml	34032	\$25	methyprylon	1000µg/ml	34060	\$25
cannabidiol	1000μg/ml	34011	\$25	morphine	1000µg/ml	34006	\$25
cannabinol	1000µg/ml	34010	\$30	nitrazepam	1000µg/ml	34053	\$25
chlordiazepoxide	1000µg/ml	34044	\$25	oxazepam	1000µg/ml	34054	\$25
clobazam	1000µg/ml	34045	\$25	oxycodone	1000µg/ml	34007	\$25
clonazepam	1000µg/ml	34046	\$25	oxymorphone	1000µg/ml	34065	\$25
cocaethylene	1000µg/ml	34019	\$35	pentazocine	1000µg/ml	34062	\$25
cocaine	1000µg/ml	34015	\$25	pentobarbital	1000µg/ml	34036	\$25
codeine	1000µg/ml	34000	\$25	phencyclidine	1000µg/ml	34027	\$30
desmethyl diazepam	1000µg/ml	34048	\$30	phendimetrazine	1000µg/ml	34025	\$25
diazepam	1000µg/ml	34047	\$25	phenmetrazine	1000µg/ml	34026	\$25
ecgonine	1000µg/ml	34017	\$30	phenobarbital	1000µg/ml	34037	\$25
ecgonine methyl ester	1000µg/ml	34018	\$35	phentermine	1000µg/ml	34024	\$25
fenfluramine	1000µg/ml	34023	\$25	prazepam	1000µg/ml	34055	\$25
flunitrazepam	1000µg/ml	34049	\$25	dextro-propoxyphene	1000µg/ml	34008	\$25
flurazepam	1000µg/ml	34050	\$25	secobarbital	1000µg/ml	34038	\$25
d,l-glutethimide	1000µg/ml	34058	\$25	talbutal	1000µg/ml	34039	\$25
hexobarbital	1000µg/ml	34033	\$25	temazepam	1000µg/ml	34056	\$25
hydrocodone	1000µg/ml	34002	\$25	thebaine	1000µg/ml	34009	\$25
hydromorphone	1000µg/ml	34063	\$25	thiamylal	1000µg/ml	34040	\$25
levorphanol	1000µg/ml	34003	\$25	thiopental	1000µg/ml	34041	\$25
				triazolam	1000µg/ml	34057	\$25

Improved Rtx®-2330 Capillary Columns

New Surface Treatment and Advanced Polymer Technology Improve the Quality and Longevity of Rtx®-2330 Capillary Columns

One of the most polar capillary column stationary phases is a 90% biscyanopropyl/ 10% phenylcyanopropyl polysiloxane or Rtx®-2330. Because of the presence of the cyano groups in the polymer backbone, the Rtx®-2330 stationary phase possesses a strong dipole moment and is highly selective for cis/trans compounds or compounds with conjugated double bonds. The Rtx®-2330 is widely used for analyzing fatty acid methyl esters (FAMEs), cis/trans isomers, rosin acids, and TCDD isomers.

Despite the high utility of biscyanopropyl/ phenylcyanopropyl phases, they typically suffer from poor

column efficiencies, higher bleed, and short column lifetimes when thermally cycled. These problems result from difficulties in getting the high cyano content stationary phases to coat uniformly on a smooth fused silica surface. To overcome these limitations, Restek's chemists have developed a new surface treatment that is more compatible with the Rtx®-2330 phase. In addition, they synthesized an advanced polymer that is more compatible with this surface to produce columns with improved column efficiency and lower bleed. The combination of the new surface treatment and advanced polymer technology results in enhanced column lifetimes.

Common problems associated with biscyanopropyl/phenylcyanopropyl columns

The Rtx®-2330 stationary phase is extremely polar because of the high percentage of cyano groups in the polymer backbone. Figure 1 shows the structure and composition of the Rtx®-2330 phase. The high concentration of cyano groups hinders crosslinking and prevents complete crossbonding of the stationary phase to the fused silica surface. Therefore, Rtx®-2330 columns cannot be solvent rinsed. Because of incomplete crossbonding, high cyano content stationary phases have been known to thermally rearrange (puddle) and lose efficiency rapidly while the oven is being cycled up and down in temperature. At high GC oven temperatures (> 275°C), cyano polymers become very fluid and lose critical viscosity leading to high bleed levels, retention time shifts, and loss of resolution for critical pairs.

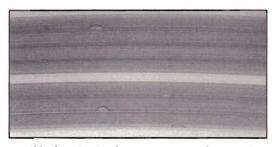
Surface chemistry and polymer composition greatly affect column quality and lifetime

Restek's chemists have developed a surface layer that properly matches the critical surface tension of the 2330 phase. If the

surface layer is not completely compatible in polarity with the stationary phase, the phase will "puddle" or bead up after repeated thermal cycling. If puddling occurs, a column will exhibit poor efficiency and high bleed. The photo below shows phase puddling due to thermal rearrangement. The dark spot on the tubing indicates where stationary phase has beaded up on the column surface.

Finding the optimum surface layer for a stationary phase is one of the most important steps in capillary column manufacturing. The compatibility of the surface layer with the

stationary phase dramatically affects the column's efficiency, thermal stability, and longevity.



An incompatible deactivation layer prevents uniform stationary phase coating and can create phase puddling on the column's surface.

In combination with the new surface layer, we have synthesized an advanced polymer that resists rearrangement and puddling. The new polymer has the same polarity as the original polymer, but crosslinking mechanisms have been enhanced to increase column longevity. The end result is an improved Rtx*-2330 capillary column that exhibits enhanced column efficiency, thermal stability, and column lifetime.

Unique selectivity of the Rtx®-2330 is ideal for a wide variety of applications

Because of the high content of cyano groups in the Rtx®-2330 polymer backbone, it is very selective for a wide variety of applications. In the food industry, many laboratories are involved in the analysis of fatty acids to comply with the new labeling regulations.

Partially hydrogenated oils contain geometric and positional isomers of unsaturated fatty acids. Food chemists who analyze these complex mixtures of FAME cis/trans isomers often have

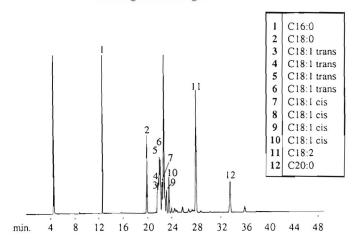
difficulty obtaining the resolution needed for this analysis. Figure 2 shows the analysis of commercial margarine on a 105m, 0.25mm ID, 0.20μm Rtx®-2330 and the resolution obtained of the C18:1 cis/trans isomers on this phase.

EPA Methods 513 and 613 are capillary GC/ECD and GC/MS methods developed for monitoring the highly toxic 2,3,7,8 isomer of tetrachlorodibenzo-p-dioxin (TCDD). The Rtx®-2330 phase is the primary column recommended by the EPA for Methods 513 and 613 since it can resolve 2,3,7,8-TCDD from

the other isomers present in TCDD. Figure 3 shows the analysis of TCDD on a 60m, 0.25mm ID,0.20µm Rtx®-2330 by GC/ECD. Baseline resolution of the 2,3,7,8-TCDD isomer and low column bleed is achieved on this column.

Improvements in the Rtx®-2330 surface layer and polymer technology yield capillary columns with enhanced column efficiencies and increased longevity. Analysts should expect better peak resolution, lower bleed, and longer column lifetimes with the improved Rtx®-2330 columns.

Figure 2 - Resolve C18:1 cis/trans isomers in margarine using the Rtx®-2330.



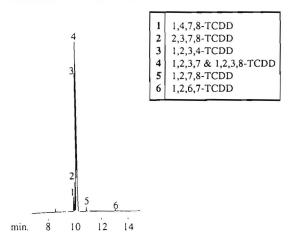
105m, 0.25mm ID, 0.20µm Rtx®-2330 (cat.# 10729) 1.0µl split injection of a commercial margarine mixture. Concentration approximately 5µg.

Oven temp.: 165°C isothermal

Inj. & det. temp.: 275°C Carrier gas: hydrogen Linear velocity: 40cm/sec. FID sensitivity: 4 x 10-11 AFS

Split ratio: 50:1

Figure 3 - Achieve TCDD isomer separation on a 60m, 0.25mm ID, 0.20µm Rtx®-2330.



60m, 0.25mm ID, 0.20µm Rtx®-2330 (cat.# 10726) 2.0µl splitless injection of TCDD isomers. Concentration approximately 0.5µg of each isomer.

Splitless hold: 0.45 min.

Oven temp.: 200°C (hold 1 min.) to 250°C @ 8°C/min. (hold

15 min.) to 275°C @ 15°C/min. (hold 5 min.)

Inj. & det. temp.: 275°C Carrier gas: hydrogen

Linear velocity: 40cm/sec. ECD sensitivity: 21kHz full scale

Product Listing

Rtx®-2330* (90% biscyanopropyl - 10% phenylcyanopropyl polysiloxane) Polymer stable to 275°C

	df (µm)	tem	p. range	15-M	leter	30-M	leter	60-M	leter	105-N	1eter_
0.25mm	0.10	0	275°C	10705	\$225	10708	\$380	10711	\$665	10714	\$870
ID	0.20	0	275°C	10720	\$215	10723	\$370	10726	\$655	10729	\$850
0.32mm	0.10	0	275°C	10706	\$245	10709	\$410	10712	\$725	10715	\$945
ID	0.20	0	275°C	10721	\$235	10724	\$400	10727	\$715	10730	\$925
0.53mm	0.10	0	275°C	10707	\$250	10710	\$455	10713	\$820		
ID	0.20	0	275°C	10722	\$240	10725	\$445	10728	\$795		
0.18mm	df (µm)	tem	p. range	10-M	leter	20-M	leter	40-M	leter		
ID	0.10	0	275°C	40701	\$200	40702	\$320	40703	\$595		-

Caution: As with all high cyano content stationary phases, the analysis of acids, alcohols, and diols may show poor reproducibility due to an interaction of the cyano groups & column surface with these compounds.

*Not solvent rinsable.

Hints for the Capillary Chromatographer



The Importance of Inlet Sleeve Deactivation

Chromatographers frequently call with questions about sleeve deactivation. Due to the popularity of this topic, we have decided to rerun the article on "The Importance of Inlet Sleeve Deactivation" from September 1990's issue of The Restek Advantage.

Your entire sample pathway must be inert

Capillary columns are inherently more inert than packed columns. However, capillary chromatographers still experience breakdown and adsorption of active compounds. Inertness of the sample pathway is particularly important with certain pesticides, herbicides, drugs, amines, and acids. If the column itself is inert, then what is the source of the problem? One major area that most chromatographers overlook is the inlet. The inertness of the inlet sleeve is very important since it is part of the sample pathway and can often be the source of adsorption or breakdown. If the sleeve is not deactivated properly, adsorption can take place in the sleeve and alter the composition of the sample reaching the column.' Also, when analyzing compounds at low levels, adsorption or decomposition of some components in the sleeve results in poor quantitation and even misidentification.

Is it important to deactivate the sleeve?

For most samples, the answer to this question is a resounding yes. We tested the adsorptive characteristics of an undeactivated versus a deactivated sleeve (Figure 1). Endrin, an active pesticide, was injected into an untreated and a

properly deactivated inlet sleeve. The undeactivated sleeve showed 98% breakdown of a 50pg injection of endrin into its respective degradation products, endrin aldehyde and endrin ketone. The deactivated sleeve showed only 6% breakdown.

In addition to breakdown, an improperly deactivated sleeve can cause irreversible adsorption. To demonstrate this effect, we compared the response factors of three active compounds, 2,4dinitrophenol, pentachlorophenol, and benzidine on both undeactivated and properly deactivated sleeves. Table I shows that the response factors for all three compounds are lower on the undeactivated sleeve than on the deactivated sleeve.

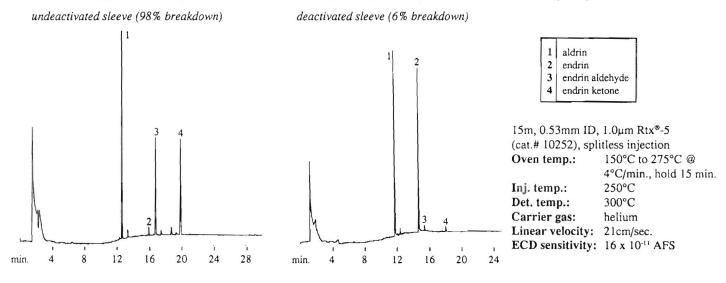
Table I Average response factors relative to naphthalene of 3 injections.

0.248	0.240	0.327
0.185	0.188	0.234

Why do response factors change for active compounds?

A priming effect occurs when improperly deactivated sleeves are used for adsorptive samples. We injected 1.0µl of an endrin standard onto a column and calculated the percentage of breakdown. This first injection primed the sleeve with endrin. When we injected the endrin standard onto the column again, the result was less endrin breakdown. This procedure can be

Figure 1 - Comparison of endrin breakdown on an undeactivated and a deactivated Cyclosplitter®.



repeated until the percentage breakdown remains constant for three injections. If you are running calibration curves and your numbers are jumping around in this manner, your inlet sleeve may not be adequately deactivated.

How do you know that your sleeve has been properly deactivated?

The only true test of a sleeve's inertness is in its actual performance with active compounds. A visual inspection of the sleeve will not tell you that the sleeve has been deactivated. In fact, most instrument manufacturers do not deactivate their inlet sleeves.

Are you using a home-brewed sleeve deactivation recipe?

Many chromatographers are aware that their sleeves are not deactivated and develop many different recipes to treat their sleeves. The most common treatment is with dimethyldichlorosilane (DMDCS). The use of this material presents several problems. It reacts with moisture to produce HCl vapors, so treatments with DMDCS must be done in a wellventilated area. Also, DMDCS, if not properly end capped, can react with humidity in air, resulting in weakened effectiveness.

We examined the quality of in-house deactivation procedures by comparing several home-brew recipes commonly used. We looked at acid treatment, DMDCS treatment, acid and DMDCS treatment, and finally our own proprietary deactivation procedure. We evaluated each of these procedures in duplicate using a Cyclosplitter. The resulting endrin breakdown from these five treatments is shown in Table II. The results clearly show that all deactivation techniques are not equal and a highly inert surface requires a procedure combining both acid and DMDCS treatments.

Table II % endrin breakdown with various deactivations

Treatment	Run #1	Run #2
Acid only	36%	45%
DMDCS only	20.8%	18.3%
Acid and DMDCS	19.8%	18.5%
Restek's Old Deactivation*	10.0%	15%
Restek's New Deactivation	2.2%	1.2%

% endrin breakdown calculation: Σ areas = endrin area + endrin aldehyde area + endrin ketone area

> aldehyde area + ketone area % total breakdown = 100 × Σ area

Will glass wool or beads in your sleeve cause adsorption and breakdown?

Yes. Many chromatographers who analyze dirty samples use a plug of glass wool or beads in their sleeve to act as a filter. However, both wool and beads greatly increase the surface area that the sample contacts and can be a source of adsorption or breakdown. It is critical that the wool or beads be properly deactivated. Even if your wool is deactivated, active sites can be created as the fibers break when inserting the wool into the sleeve. If you plan to use wool, be sure that it is thoroughly deactivated and use care when inserting it into the sleeve.

Can sleeves be cleaned without redeactivation?

Sleeves only need to be redeactivated if the deactivation layer is removed during cleaning. Daily GC use deposits septa particles, sample residue, and pyrolizates on the sleeve surface, contaminating your inlet. One way to prolong the lifetime of your deactivated sleeve is to wash out the sleeve with methanol (since methanol does not swell septa or make them sticky). Nylon tube brushes and pipe cleaners are also helpful for removing small septa fragments from dirty sleeves. However, be careful to avoid scratching the liner with the inner metal core of the brush. You can then use stronger solvents such as methylene chloride, hexane, or the solvent your sample is diluted in, to remove additional contamination. These mild procedures will not require redeactivation.

If the contamination has been pyrolized onto the sleeve, solvent rinsing or other simple cleaning procedures may still leave a residue. The only effective procedures to remove this residue are strong acids, bases, detergents, or baking in a muffle furnace. If you use these harsh procedures, you will remove the deactivation layer, requiring redeactivation.

Analyzing active compounds is a difficult assignment. If you properly deactivate your inlet sleeve you will find many of your analysis problems solved. For more information, please call our technical service group at 800-356-1688, ext. 4. ■

Suggestions? —

Is there a topic you would like to see covered in "Hints for the Capillary Chromatographer"? If so, please call our technical service department toll-free at 800-356-1688 (ext. 4).

^{*} Prior to April 1991.

Peak Performers

Now available from Restek...

Hoke Toggle, Ball, and Plug Valves

Restek now offers Hoke toggle, ball, and plug valves. These high quality, precision machined valves are available in ¹/s" and ¹/4" sizes in both brass and stainless steel. The toggle valves are ideal for applications when instant on/off control is necessary. Hoke toggle valves are rated to 200psig at 21°C and have a maximum operating temperature of 149°C. The forged body ball valves are ideal for high pressure applications. Hoke ball valves have a floating ball to assist in sealing and reduce operating torque and dual seats provide leak-tight bidirectional sealing. They are rated to 6000psig at 21°C and have a maximum operating temperature of 249°C. The plug valves are ideal for applications where throttling or on/off operations are required. Hoke plug valves have dual retaining rings to prevent accidental plug removal. They are rated to 3000psig at 21°C and have a maximum operating temperature of 205°C.

	Bi	rass	Stainless	s Steel
Valve type	1/8"	1/4"	1/8"	1/4"
	21885	21886	21985	21986
Toggle	\$36	\$33	\$85	\$80
Ball	21887	21888	21987	21988
	\$50	\$50	\$90	\$88
Plug	21889	21890	21989	21990
	\$48	\$44	\$70	\$68

Miniature Leak Detector

Leaks in a gas chromatographic system can cause problems ranging from increased detector noise, baseline instabilities, and short column lifetime, to wasting expensive carrier gases. GL Sciences' new portable Leak Detector LD-223 allows analysts to detect minute gas leaks undetected by liquid leak detectors. This leak detector's compact size is designed for easy transport and hand-held usage. Simple, push button operation allows one-touch zero adjustment, while the low dead volume sampling line provides quick sample response. Trace leaks of both helium and hydrogen* can be detected using the

high sensitivity range. Four AA alkaline batteries (not included) provide up to 12 hours of continuous operation.

- · Portable size
- Quick response
- High sensitivity
- Simple operation
- Contamination-free leak detection



GL Sciences Leak Detector: cat.# 21605, \$950 each

Don't miss this new wizard dollar item...

Wizard Pitcher & Glasses Set

We have added another premium gift to our Wizard Dollar promotional program. Now available is a wizard pitcher and glass set. This set contains one 55 ounce glass pitcher and eight 13 ounce beverage glasses for just 25 Wizard Dollars.

If you are not saving your Restek Wizard Dollars, you are missing out on valuable FREE gifts. For more information on this program, call 800-356-1688, ext. 3 to request a copy of the Wizard Dollar Promotional Flyer.

Wizard Pitcher & Glasses set, cat.# 60028, \$25 wizard dollars

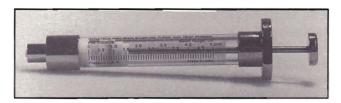


To order any Restek product, call 800-356-1688 (ext. 3).

^{*} not designed for use in explosive atmospheres.

Heavy Duty Purge and Trap Syringe

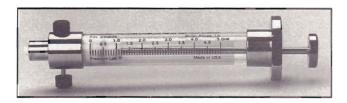
Designed by Dynatech Precision Sampling Corporation for filling and emptying sparge tubes on purge and trap concentrators, these new purge and trap syringes offer the analyst special features for purge and trap.



Without Sample Lok

Size	2ml	5ml	10ml
Catalog number	21205	21206	21209
Price	\$60	\$60	\$60

- · Teflon® Luer-lock tip
- Heavy duty glass barrel and metal front and rear flanges reduce likelihood of breakage if dropped.
- · Pressure-Lok plunger tip



With Sample Lok

Size	5ml	10ml
Catalog number	21208	21207
Price	\$75	\$75

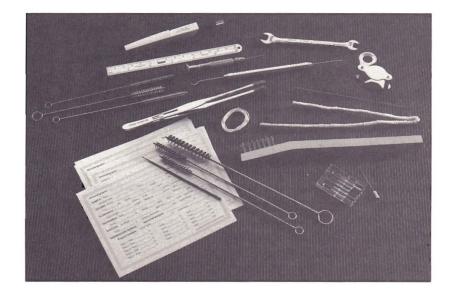
The MLE Tool Kit Contains a Variety of Tools to Make Your Life Easier!

This kit contains all the tools necessary for installing and maintaining your capillary columns.

The MLE Kit includes:

- · Rubber-tipped Slide-Lok tweezers
- · 15cm compact steel ruler
- · Sapphire scribe
- · Pocket magnifier
- Pin vise with three drills (0.41, 0.51, 0.8mm)
- · Four-inch tapered needle file
- Six stainless steel jet reamers (0.25 0.65mm)
- · Five self-adhesive capillary column labels
- · Septum puller
- Three nylon brushes (1/8", 3/16", 1/4")
- · Pipe cleaner (one-foot)
- · 1/4" 5/16" wrench
- One-meter of high temperature string (400°C)
- Three stainless steel brushes (3/16", 1/4", 3/8")
- · Stainless steel toothbrush
- · Glass wool puller/inserter
- · Fused silica column installation guide
- · Capillary reference poster

MLE Tool Kit: cat.# 20118, \$119 each



News from Restek

Restek Celebrates 8th Anniversary



October 1993 marks Restek's eighth anniversary. Since 1985, we have grown to over 80 employees and two locations, Bellefonte and Germany. Another major building expansion is planned for next spring to give us more room for our existing departments.

Special thanks go out to our loyal customers, suppliers, and distributors who have helped to make our rapid success possible. The Restek Wizards are looking forward to future challenges and your continued support.



MasterCard® and VISA® are accepted.

United States:

110 Benner Circle Bellefonte, PA 16823 Orders: (800)356-1688, ext. 3

Tech. service: (800)356-1688, ext. 4

FAX: (814)353-1309

Restek GmbH:

House address: Am Laubach 3 65843 Sulzbach, Germany Mailing address: Postfach 1127 65837 Sulzbach, Germany Telefon: 061 96/26 0 88

© Copyright 1993, Restek Corporation

It's not too late to sign up for Restek's **Capillary Chromatography Seminars!**

Restek's 5th seminar tour begins on September 13 in Sacramento, California and Seattle, Washington. The tour will continue with presentations in 52 cities throughout the United States. This educational course has been updated for 1993 and includes sections on capillary theory & terminology, inlet systems & injection techniques, column selection, column installation, and maintenance & troubleshooting. If you would like more information about our seminar series, call 800-356-1688, ext. 3.

Upcoming Restek Exhibits:

October 5-7 in San Francisco, CA (Booth # 115) Paper Presentations: "Efficient GC Method Development Using Thermodynamic Retention Modeling"

Society of Forensic Toxicologists (SOFT) Meeting

October 13-15 in Pheonix, AZ (Booth #26) Paper Presentations: "Optimization of Drug Analysis Using Gas Chromatography & Thermodynamic Retention Times"

Eastern Analytical Symposium (EAS)

November 15-19 in Somerset, NJ (Booth # 233) Short Course: Advanced Gas Chromatography

The Restek logo, Rtx[®], and Cyclosplitter[®] (U.S. Pat. No. 5,119,669) are trademarks of Restek Corporation. All other trademarks are the property of their respective owners. Restek capillary columns are manufactured under U.S. patent 4,293,415, licensed by Hewlett-Packard Company.

Restek Corporation

110 Benner Circle Bellefonte, PA 16823-8812

FORWARDING & RETURN POSTAGE GUARANTEED ADDRESS CORRECTION REQUESTED

Bulk Rate US Postage PAID Permit No. 1110 Cincinnati, Ohio

Eliminate Wasteful Duplicate Mailings

If you receive duplicates of this newsletter, please call us at 800-356-1688, ext. 102. We will gladly remove names from our files upon request.