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The Advantage

**Innovators of
High Resolution
Chromatography
Products**

Pharmaceutical Researchers, The Future of ChromatographySM Arrives Today!

by David Bliesner

Restek Analytical ServicesSM:

- **cGMP, HPLC, and GC method development and validation**
- **Stationary phase development**
- **Full lines of HPLC and GC columns and accessories**
- **Innovative hardware and accessory designs**
- **Unsurpassed HPLC and GC technical support**
- **HPLC and GC educational services**

Restek Corporation is proud to announce the grand opening of Restek Analytical ServicesSM (RAS). RAS is a unique and innovative, current Good Manufacturing Practices (cGMP)-compliant chromatography laboratory, designed to deliver quality products and services for the pharmaceutical market. No other column manufacturer can provide such complete services to support their products. Along with a full line of both HPLC and GC columns, RAS offers analytical method development and validation, HPLC/GC education and training, custom stationary phase design, and cGMP/regulatory services and support.

RAS has assembled seasoned researchers and analysts who

have extensive experience in cGMP method development and validation. These experts are a perfect complement to our existing manufacturing staff. As a group, we understand the pressures and time constraints associated with pharmaceutical research and have practical experience analyzing many of the common drugs currently under development. The entire RAS staff is dedicated to helping you achieve your goals in a timely and compliant fashion.

In addition to offering a complete selection of quality HPLC and GC columns, we can help design tools to fit your unique requirements. We continue to develop new stationary phases and applications in response to customer

inquiries and needs. We can design a product and service package specially for you.

For further information, please request the RAS fulfillment folder (cat.# 59630). This folder includes details on quality assurance, research and development, analytical development and validation, laboratory and regulatory service, and HPLC columns and accessories.

Or, feel free to contact your local distributor to discuss your specific applications. Call and find out why RAS is truly the *future of chromatography*.



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**Spring/Summer
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Restek Analytical ServicesSM

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RAS has assembled some of the most talented, customer-oriented analysts in the HPLC and pharmaceutical industries. Meet the leaders of RAS:



Dr. David M. Bliesner,
RAS Director

Expertise:

- HPLC method development and validation in a cGMP environment
 - Qualification and operation of cGMP analytical laboratories
 - Thin layer chromatography
 - cGMP auditing and data review
 - 15 years combined R&D and leadership experience
- dbliesner@restekcorp.com**



David S. Bell,
RAS Senior Researcher

Expertise:

- HPLC method development and validation in a cGMP environment
 - Thin layer chromatography
 - 8 years of experience
- dbell@restekcorp.com**



Keith J. Duff,
RAS R&D Group Leader

Expertise:

- HPLC bonded phase synthesis
 - Synthetic chemistry
 - HPLC method development
 - 16 years of experience
- keithd@restekcorp.com**



Larry T. Peters,
RAS Senior HPLC Technician

Expertise:

- HPLC column packing
 - HPLC column bonding
 - 16 years of experience
- larryp@restekcorp.com**



Randy L. Romesberg,
RAS Production Group Leader

Expertise:

- R&D and HPLC packing materials and hardware production
 - 9 years of experience
- rrome@restekcorp.com**

RAS Products & Services:

Development & Validation

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- Ruggedness testing
- Robustness testing
- HPLC column crossover validation
- Method troubleshooting and optimizing

Laboratory & Regulatory Services

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- Assistance in preparation with CMC sections for NDAs
- Laboratory auditing services
- On-site data review and troubleshooting
- Pre-approval inspection (PAI) preparation assistance

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- Developing custom stationary phases
- Designing new hardware and accessories
- Investigating new separation technologies
- Improving existing products

Quality Assurance

- Full compliance with cGMPs
- QA testing is an integrated part of the RAS system

HPLC Columns & Accessories

- Pinnacle[®], Hypersil[®], Inertsil[®], Kromasil[®], and Nucleosil[®] HPLC columns
- Unsurpassed technical support
- Quality HPLC accessories
- 30-day "no questions asked" return policy
- Stationary phase and hardware design

"In the modern chromatography laboratory, you want one thing — reliable solutions to your chromatographic problems. Previously, when a method could not be reproduced, questions such as, 'Is it the column or did something change in the sample workup?' could not be answered by one source. By combining product development and method development under one umbrella, you can be assured that your problem will be solved with full accountability.

Any method validation done by Restek is supported by a team of dedicated scientists with your success in mind. RAS is a fully cGMP compliant laboratory that provides personalized services dependent on your needs. We can provide training, method development and validation, or custom design stationary phases to speed up your analysis or achieve a difficult separation. Please give us a call and let us help you."

David M. Bliesner, Ph.D.
Director, RASSM



Chiral Separation of Underivatized Pharmaceuticals Using Capillary GC

by Sherry Sponsler



- **Fast, simple analysis**
- **Thermal stability ensures low column bleed**
- **Reproducible enantioselectivity**

Many pharmaceutical compounds are chiral, where each enantiomer may exhibit different biological activity and toxicological effects. For this reason, manufacture and isolation of a drug as a pure enantiomer may be desired. Although such separations are typically performed on chiral liquid chromatography (LC) columns, many pharmaceuticals can be analyzed by capillary gas chromatography (GC).

GC analysis often requires derivatization of the sample to reduce reactivity, increase volatility, and enhance enantiomeric selectivity. However, ibuprofen and thalidomide can be analyzed by capillary GC, without derivatization. The Rt- β DEXsm™ and Rt- β DEXcst™ columns provide excellent chiral separation of these underivatized optical isomers within reasonable analysis times.

Ibuprofen is a commonly used pain reliever. Currently it is manufactured in the racemic form. However, researchers determined that R-enantiomer, the enantio-pure form, is much more potent than the racemic mixture. A smaller dosage of the enantio-pure form would be required to provide the same effect or pain relief.¹ The free optical isomers of ibuprofen are quickly resolved on an Rt-

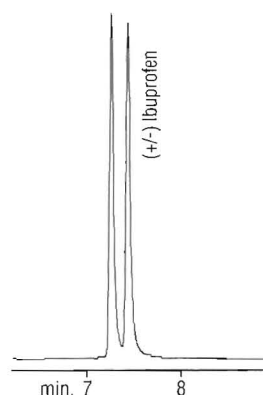
β DEXsm™ column in under 10 minutes (Figure 1).

During the 1950's, thalidomide was prescribed to pregnant women for morning sickness, but was banned in 1962 because of its mutagenic effect on fetuses. It was later discovered that the S-enantiomer was the primarily responsible teratogen. However, both enantio-pure forms racemize *in vitro* and *in vivo*.² Recently, thalidomide has re-emerged as a therapeutic agent for mouth sores in HIV patients, and for problems associated with skin and autoimmune disorders. Thalidomide is resolved into its enantiomers on the Rt- β DEXcst™ column, as shown in Figure 2. The thermal stability of these columns to 230°C allows analysis of larger semi-volatile components, such as thalidomide, without the interference of column bleed.

Chiral separation of many pharmaceutical compounds is possible by capillary GC, and some do not require derivatization. The Rt- β DEX™ columns provide unique enantioselectivity and thermal stability for separating other pharmaceutical compounds.

Figure 1:

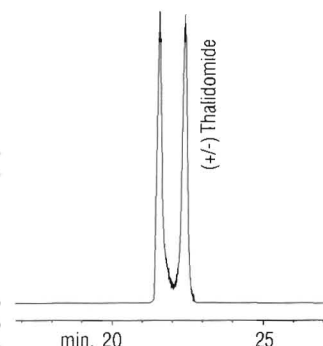
Ibuprofen Analysis in 10 Minutes on an Rt- β DEXsm™ Column.



30m, 0.32mm ID, 0.25 μ m Rt- β DEXsm (cat.# 13104). On-column injection, concentration approximately 125ng each enantiomer.
Oven temp.: 175°C to 200°C @ 2°C/min.,
Inj./det. temp.: 200°C/230°C;
Carrier gas: helium @ 60cm/sec.,
Detector type: GC-FID;
Split ratio: 13:1 using cup splitter sleeve (cat.# 20709).

Figure 2:

Thermal Stability to 230°C for a Better Thalidomide Separation on an Rt- β DEXcst™ Column.



30m, 0.32mm ID, 0.25 μ m Rt- β DEXcst (cat.# 13102). On-column injection, concentration approximately 15ng each enantiomer.
Oven temp.: 200°C to 230°C @ 1°C/min.,
Inj./det. temp.: 200°C/230°C;
Carrier gas: hydrogen @ 80cm/sec.,
Detector type: GC-FID;
Split ratio: 13:1 using cup splitter sleeve (cat.# 20709).

References

- ¹ Lemko, C.H., C. Gilles, and R. Foster. *Stereospecific High-Performance Liquid Chromatographic Assay of Ibuprofen: Improved Sensitivity and Sample Processing Efficiency*. *J. of Chromat.* 1993; 619 pp 330-335.
- ² Schmidt, H.J., H. Nav, D. Neubert. *The Anantiomers of the Teratogenic Thalidamide Analogue EM 12:1. Chiral Inversion and Plasma Pharmacokinetics*, *Arch. Toxicol.* 1998; 62 pp. 2-3.

Product Listing:

Rt- β DEXsm™ 30m, 0.25 μ m df

0.25mm ID	0.32mm ID
13105	13104

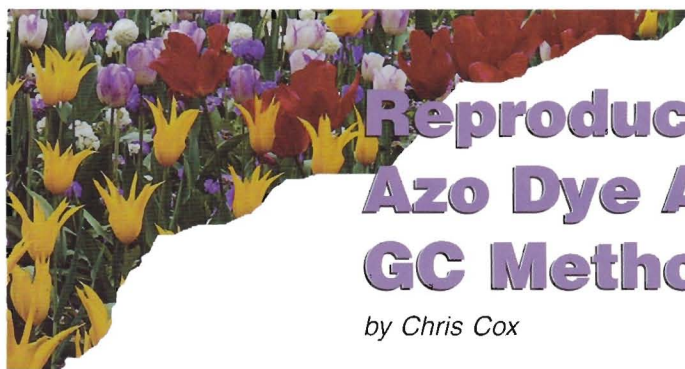
Rt- β DEXcst™ 30m, 0.25 μ m df

0.25mm ID	0.32mm ID
13103	13102

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INTERNATIONAL



Reproducible, Accurate Azo Dye Analysis Using New GC Method

by Chris Cox

- **Base-deactivated products improve reproducibility**
- **Pure, stable calibration standards ensure accurate results**

Some consumer goods that come into contact with the human body, such as clothing, jewelry, and toys, are processed using azo dyes, which can metabolize into carcinogenic aryl amines. Because of this, European countries have shown concern over the presence of azo dyes in their manufactured products. Products containing azo dyes that could form into one or more of a list

of amines cannot be distributed in Germany.¹ In 1996, the German Government Institute BgVV², published a method for the analysis of azo dyes.

A few problems have been encountered with this method. Analysts commonly see "priming effects." This is when the response factors rise for the first several injections before they reach an equilibrium state.

- While standard deactivation of columns and sleeves works well in most situations, they typically do not perform well for amine analyses using splitless injections at low concentrations.

Restek has developed an ideal GC method for this azo dye analysis; it has total system inertness, with no priming effects and excellent reproducibility.

An Rtx®-5 Amine column, combined with an amine-deactivated inlet sleeve and wool, provides a significant advantage in the analysis of amines. The priming effect of the standard deactivation versus the amine deactivation was evalu-

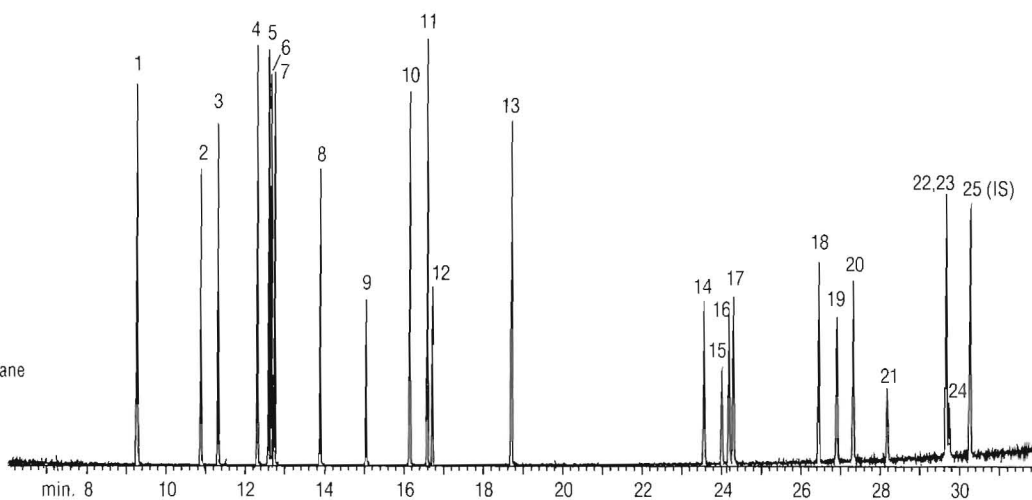
ated by comparing the response factors for each during multiple runs. With the standard deactivation, the response factors for the first injection were as much as 10% lower than the tenth injection. However, with the amine deactivation, there were no priming effects. The elimination of priming effects greatly improves the reproducibility of the analysis.

Another difficulty with the German method is the absence of pure, stable calibration standards. Many of the target compounds often must be tested and re-purified to ensure accurate preparation of the calibration standards. Restek's azo

Figure 1:

Restek's Deactivated System and Azo Dye Standard Ensures Accurate Results.

1. o-toluidine
2. 2-methoxyaniline
3. 4-chloroaniline
4. 2-methoxy-5-methylaniline
5. 2,4,5-trimethylaniline
6. 3-chloro-2-methylaniline
7. 4-chloro-2-methylaniline
8. 2,4-diaminotoluene
9. 2,4-diaminoanisole
10. 2-naphthylamine
11. 2-aminobiphenyl
12. 2-methyl-5-nitroaniline
13. 4-aminobiphenyl
14. 4-phenylazoaniline
15. 4-aminophenylether
16. benzidine
17. bis-(4-aminophenyl)-methane
18. 4'-amino-2,3'-dimethylazobenzene
19. 4,4'-diamino-3,3'-dimethyldiphenyl methane
20. 3,3'-dimethylbenzidine
21. 4-aminophenylthioether
22. 3,3'-dichlorobenzidine
23. 4,4'-methylene-bis(2-chloroaniline)
24. 3,3'-dimethoxybenzidine
25. 3,3',5,5'-tetramethyl benzidine (IS)



30m, 0.25mm ID, 0.25µm Rtx®-5 Amine (cat.# 12323). 1µl splitless injection using a 4mm gooseneck sleeve for HP GCs (cat.# 20798-211.1). 10µg/ml in ethylacetate. **Oven temp.:** 50°C to 200°C @ 10°C/min. to 270°C @ 5°C/min.; **Inj./det. temp.:** 250°/300°; **Carrier gas:** helium @ 1ml/min.; **Detector type:** MSD; **Splitless hold time:** 1 min.

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INTERNATIONAL

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dye standard contains raw materials that are carefully tested and purified to provide the most pure and stable calibration standard available. **Figure 1** shows the analysis of Restek's azo dye standard. We used the procedure as detailed in the German method. By substituting Restek's specially-deacti-

vated column and inlet sleeve, and using Restek's azo dye standard, we achieved reproducible and accurate results. For details on sample preparation, request the *Reproducible, Accurate Azo Dye Analysis Using New GC Method Application Note* (cat.# 59552).

References

- ¹ German consumers goods act of July 15, 1994, Lebensmittel-und Bedarfsgegenständegesetz, §35 LMBG.
- ² Bundesinstitut für gesundheitlichen Verbraucherschutz und Veterinärmedizin.

Product Listing:

Azo Dye Precursors and Metabolites Reference Materials

Carcinogenic Aromatic Amines For European Standard Method

Azo Dye Mix #1

2-aminobiphenyl
4-aminobiphenyl
4'-amino-2,3'-dimethylazobenzene (o-aminoazotoluene)
4-aminophenylether (4,4'-oxydianiline)
4-aminophenylthioether (4,4'-thiodianiline)
benzidine
bis-(4-aminophenyl)-methane (4,4'-diaminodiphenylmethane)
4-chloroaniline (p-chloroaniline)
3-chloro-2-methylaniline (3-chloro-o-toluidine)
4-chloro-2-methylaniline (4-chloro-o-toluidine)
2,4-diaminotoluene
3,3'-dichlorobenzidine
3,3'-dimethoxybenzidine (o-dianisidine)
3,3'-dimethylbenzidine (o-tolidine)
2-methoxyaniline (o-anisidine)
4,4'-diamino-3,3'-dimethyldiphenyl methane
2-methoxy-5-methylaniline (p-cresidine)
4-methoxy-1,3-phenylenediamine sulfate hydrate** (2,4-diaminoanisole)
4,4'-methylene-bis(2-chloroaniline)
2-methyl-5-nitroaniline (2-amino-4-nitrotoluene)
2-naphthylamine
4-phenylazoaniline (p-aminoazobenzene)
o-toluidine
2,4,5-trimethylaniline

**concentration reported as free base

10µg/ml each in ethyl acetate, 1ml/ampul

	Each	5-pk.	10-pk.
	31466	31466-510	
w/data pack	31466-500	31466-520	31566

Internal Standard for Azo Dye Method

3,3',5,5'-tetramethylbenzidine
1000µg/ml in ethyl acetate, 1ml/ampul

	Each	5-pk.	10-pk.
	31467	31467-510	
w/data pack	31467-500	31467-520	31567

Restek Corporation

Rtx®-5 Amine Capillary Column

30m, 0.25mm ID, 0.25µm, cat.# 12323

Base-Deactivated Fused Silica Guard Columns

5m, 0.25mm ID 10000 each 10000-600 6-pk.

Base-Deactivated Inlet Sleeves Packed with Base-Deactivated Wool

	4mm Gooseneck for HP GCs	4mm Gooseneck for Varian GCs	5mm Gooseneck For CE GCs
each	20798-211.1	20904-211.1	20945-211.1
5-pk.	20799-211.5	20905-211.5	20946-211.5
25-pk.	20800-211.25	20906-211.25	20947-211.25

Kováts Professor úr 70. születésnapját ünnepeltük a Balaton Szimpóziumon.

A Hungarian tribute to Dr. Kováts. See page 11 for English version.

Kováts Professor úr 70. születésnapját ünnepeltük a Balaton Szimpóziumon. Magyarországi disztribútorunk, a Lab-Comp Kft. egyik tulajdonosa, Verebélyi Klára kérésére az 1997. Szeptember 3-5 között Siófokon megtartott Balaton Szimpóziumon előadást tartottam MXT kapilláris kolonnáinkról. Habár a majdnem 20 éves kromatográfias eladói tevékenységem után e tárgybeli tudásom elég jónak számít, az én ismereteim mégis elhalványulnak a nagy kromatográfus nevek mellett, akik részt vettek a szimpóziumon: Kováts, Nyiredy, Görög, Szepes, Klebovich, Engelhardt, Davankov és Kaiser, illetve akik hozzájuk kapcsolódnak, csak nem tudtak részt venni a Szimpóziumon, mint Horváth és Ettre. Nagy szerencsém volt, hogy együtt dolgozhattam, és sokat tanulhattam Dr. Ettre-től. Kimerítő kanadai szemináriumi körutak során boldogan válaszoltam kérdéseimre, válaszait gyakran cédulákon, éttermi számlákon vagy

repülőtéren szállókártyákon illusztrálva. A szimpózium fénypontja sz. Kováts Ervin Professor úr 70. születésnapjának megünneplése volt. 1927. szeptember 29-én született Budapesten, és itt is nevelkedett. 1945-ben jelentkezett a Műszaki Egyetem Vegyészmérnöki karára, ahol 1949-ben diplomát kapott. A magyar diploma kézhezvétele után Svájcba emigrált ahol 1951-ben vegyészmérnöki diplomát kapott, majd 2 évvel később doktorált a Zürichi Állami Műszaki Egyetemen. Doktori értekezését professor Hans H. Gunthardt vezetése alatt a Szerves Kémiai Laboratóriumban dolgozta ki. Ezt 10 év intenzív munka követte a Nobel díjas Leopold Ruzicka professzorral. A munka nagy részét a vezető Svájci illóolajgyártó cég a Firmenich és Cie finanszírozta.

Continued on back page...

The complete article and translations in German, French, and Italian are located at www.restekcorp.com.



New Rt-U PLOT Columns

Expanded Selectivity for GC Analysis of Gases and Solvents

by Jingzhen Xu and Andy Schuyler

- A polar, DVB copolymer-based PLOT Column
- A confirmation column for Rt-Q & Rt-S PLOT columns
- Specially designed for gas & solvent analyses

A divinylbenzene (DVB) porous polymer phase has a major advantage over other phases used in PLOT columns—it is less susceptible to the effects of moisture, which can influence column retention characteristics. Restek Rt-S and Rt-Q PLOT columns provide the hydrophobic nature and selectivity of a divinylbenzene porous polymer phase, and the efficiency of a capillary column. Now, Restek has developed a new, more polar column to join the Rt-S and Rt-Q PLOT column family—the Rt-U PLOT column.

The Rt-U PLOT column is manufactured by incorporating ethyleneglycoldimethylacrylate into the polymer chain to provide excellent selectivity for unsaturated compounds. The Rt-Q PLOT column is nonpolar and separates compounds based on volatility. The Rt-S PLOT column provides an intermediate polarity.

As shown in **Figure 1**, these three Restek PLOT columns provide different selectivities for hydrocarbons. On the Rt-U PLOT column, acetylene elutes after ethane, and propylene coelutes with propane. On the Rt-Q PLOT column, acetylene

and ethylene coelute, and propylene elutes before propane. However, on the Rt-S PLOT column, acetylene overlaps with ethane under similar run conditions, and propylene still elutes before propane. For hydrocarbon analyses, the Rt-U PLOT column is the most selective for all unsaturates. The Rt-Q PLOT column is least selective for functional groups. The Rt-S PLOT column is selective for polar compounds having high degrees of unsaturation such as acetylene.

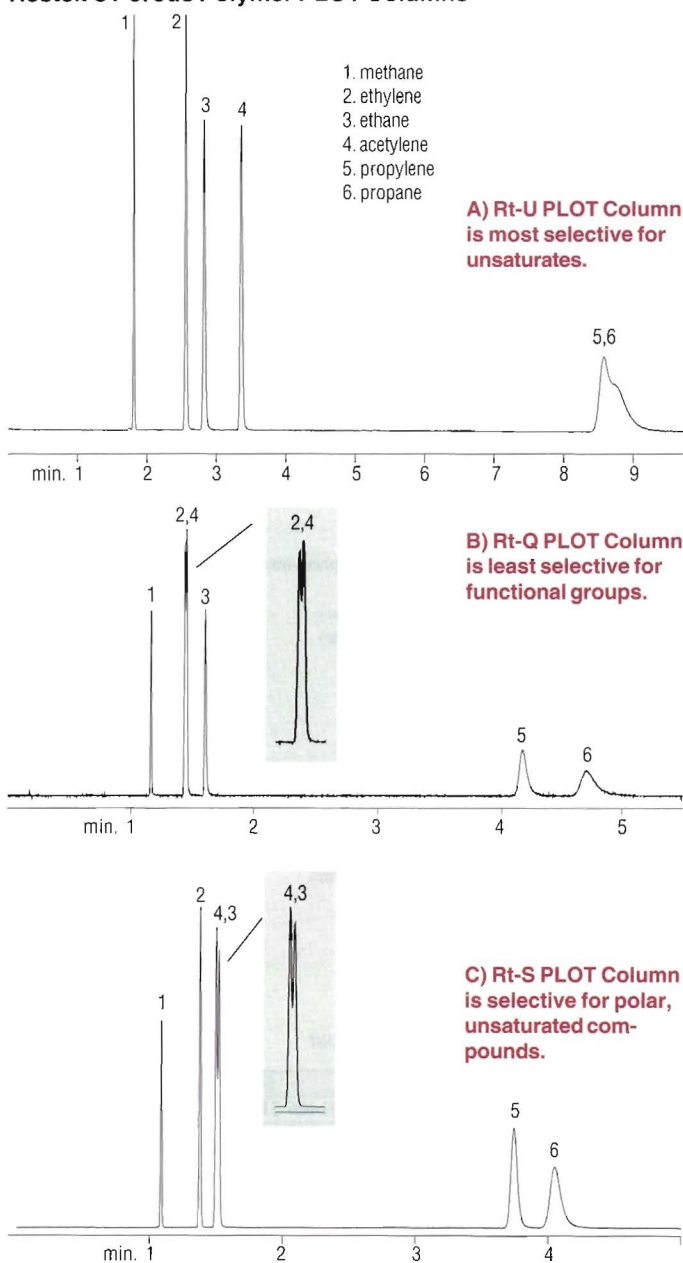
As shown in **Figure 2**, various solvents are separated on the Rt-U and Rt-Q PLOT columns. On the Rt-U PLOT column, polar solvents have much stronger retention. Ethyl acetate elutes after n-hexane, and acetone elutes after n-pentane. On the Rt-Q PLOT column, ethyl acetate elutes before n-hexane, and acetone elutes before n-pentane.

Restek's porous polymer PLOT columns separate a wide variety of chemical functionalities. The line of Rt-Q, Rt-S, and now Rt-U PLOT columns, offers much more versatility for volatile solvent analysis.

Continued top of next page...

Figure 1:

Achieve Different Selectivities for C1-C3 Hydrocarbons on Restek's Porous Polymer PLOT Columns



* 30m, 0.32mm ID, 100µl split injection. 1000ppm (v/v) each in helium with a Cyclosplitter® sleeve (cat.# 20755). Oven temp.: 50°C isothermal; Inj./det. temp.: 200°C; Carrier gas: Hydrogen; Detector: FID; Split ratio: 20/1



Figure 2:

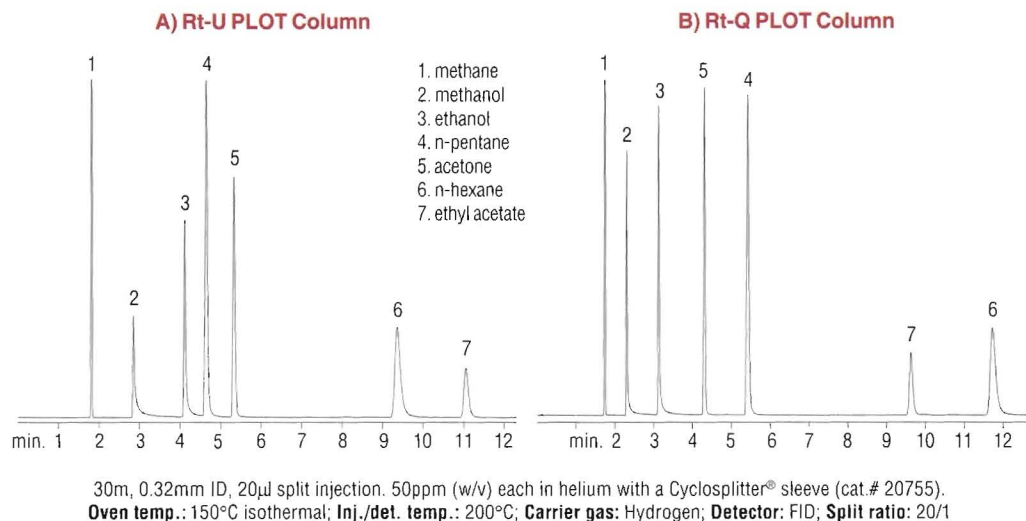
The Rt-U PLOT Column Shows a Stronger Retention for Polar Solvents Compared to the Rt-Q PLOT Column

Product Listing:

Rt-U PLOT Column

Length	0.32mm ID
15m	19725
30m	19724
Length	0.53mm ID
15m	19727
30m	19726

See Restek's 1998
Chromatography Products
Catalog for ordering
information on other
PLOT columns.



New Analytical Reference Materials for GC Analysis of Organo Tins

- Most complete line of organo tin calibration mixes available
- Organo tin analysis technical training offered

Surrogate Mixtures

2000µg/ml in methylene chloride, 1ml/ampul

Tri-n-propyltin Chloride Mixture

	Each	5-pk.	10-pk.
	31476	31476-510	
w/data pack	31476-500	31476-520	31576

Triphenyltin Chloride Mixture

	Each	5-pk.	10-pk.
	31477	31477-510	
w/data pack	31477-500	31477-520	31577

Individual Calibration Compounds for Environmental Analysis and the Electronics Industry

2000µg/ml in methylene chloride, 1ml/ampul

Tributyltin Chloride Mixture

	Each	5-pk.	10-pk.
	31478	31478-510	
w/data pack	31478-500	31478-520	31578

Calibration Mixes for Environmental Analyses

2000µg/ml ea. in methylene chloride, 1ml/ampul

Butyltin Chlorides Calibration Mixture

tetrabutyltin, tributyltin chloride, dibutyltin dichloride, butyltin trichloride.

	Each	5-pk.	10-pk.
	31472	31472-510	
w/data pack	31472-500	31472-520	31572

Phenyltin Chlorides Calibration Mixture

tetraphenyltin, triphenyltin chloride, diphenyltin dichloride, phenyltin trichloride.

	Each	5-pk.	10-pk.
	31473	31473-510	
w/data pack	31473-500	31473-520	31573

Internal Standard Mixtures

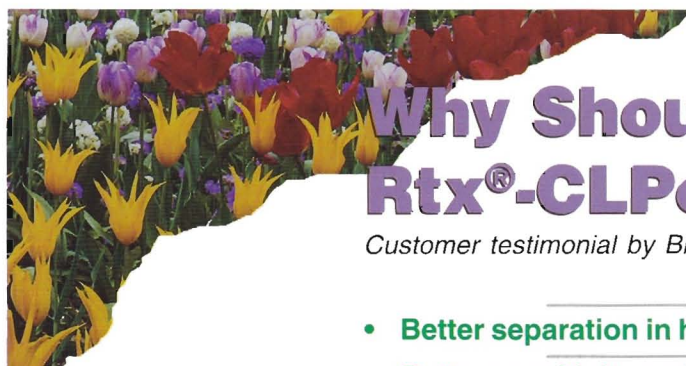
2000µg/ml in methylene chloride, 1ml/ampul

Tetra-n-propyltin Mixture

	Each	5-pk.	10-pk.
	31474	31474-510	
w/data pack	31474-500	31474-520	31574

Tetraphenyltin Mixture

	Each	5-pk.	10-pk.
	31475	31475-510	
w/data pack	31475-500	31475-520	31575



Why Should You Use Rtx®-CLPesticides Columns?

Customer testimonial by Bill DesJardins, ITS-Environmental

If you are involved with the analysis of chlorinated pesticides and want to improve your resolution and increase your throughput, just read what Bill DesJardins has to say about Restek's Rtx®-CLPesticides capillary GC columns:

"The Rtx®-CLPesticides screening column gives me better separation in half the time than a comparable 5% phenyl column. The narrower, taller peaks increased my sensitivity two-fold, and I did not experience any increase in interferences.

The total run time for a pesticide screen used to be 14 minutes with a 15m Rtx®-5 column. Even with the "long" run time, I had several co-elutions, including gamma BHC and beta BHC. With the Rtx®-CLPesticides 15m column, all 22 pesticides are resolved with a run time of only 5.5 minutes (see **Figure 1**). The better separation is not just for pesticides. The column also produces about 50% more peaks for an Aroclor mixture, allowing better identification of compounds in complex matrices.

The Rtx®-CLPesticides screening column gives better sensitivity than columns previously used for pesticide and PCB extract screening. Because of the faster retention times, the peaks on the Rtx®-CLPesticides are sharper and taller. This increases the signal-to-noise ratio, thus

- **Better separation in half the time**
- **Better sensitivity and highly stable**
- **Increased throughput**

increasing sensitivity. For screening, that means I can achieve the required detection limit (i.e. the upper half of the normal analytical calibration range) with a higher extract dilution. This, in turn, produces a cleaner screening system that is able to screen more samples and better withstand big hits from complex samples.

Retention time stability is rock solid — as it has to be. With only a few hundredths of a minute between peaks, I cannot have very wide peak ID windows. So far (several

thousand runs), I have not had peaks drift outside the ID window using the Rtx®-CLPesticides screening column, unless I have done maintenance.

The high thermal stability helps to maintain analytical integrity.

The accumulation of high-boiling compounds can be a bother during normal analysis but, since the thermal stability (TMax) of the Rtx®-CLPesticides column is 330°C, they can be quickly baked-out with a slight increase in run time.

An interesting dilemma: Because I can make many more injections per day on an Rtx®-CLPesticides screening column, I have found that a 100-place autosampler doesn't have enough capacity to hold 14 hours worth of runs. Now my real time-limitation is the size of my autosampler!"

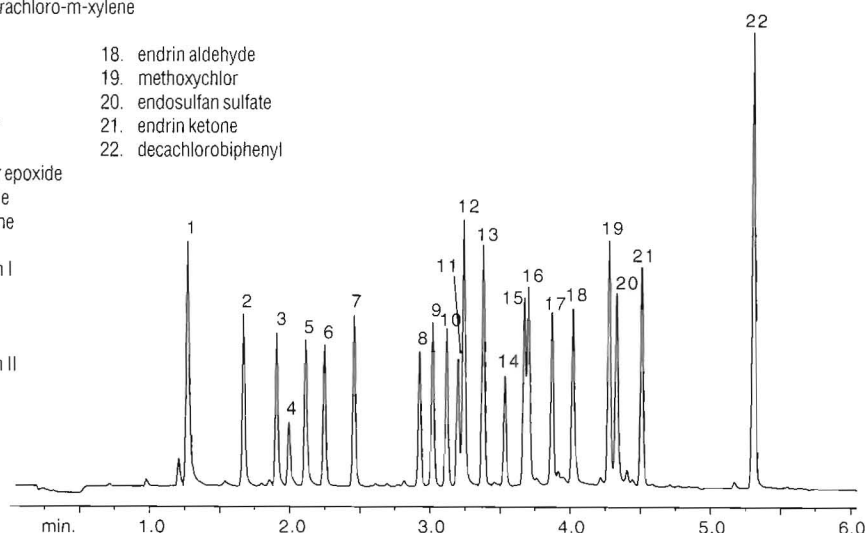
Bill DesJardins, Chemist
ITS - Environmental
55 South Park Drive
Colchester, VT 05446

For more information on these columns designed specially for chlorinated pesticide analyses, call our customer service group and ask for the Rtx®-CLPesticides brochure (cat.# 59699).

Figure 1:

Twenty-two Chlorinated Pesticides Calibration Standard Acquired After the Analysis of 3000 Sample Extracts.

- | | |
|---------------------------------|------------------------|
| 1. 2,4,5,6-tetrachloro-m-xylene | |
| 2. α-BHC | |
| 3. γ-BHC | 18. endrin aldehyde |
| 4. β-BHC | 19. methoxychlor |
| 5. δ-BHC | 20. endosulfan sulfate |
| 6. heptachlor | 21. endrin ketone |
| 7. aldrin | 22. decachlorobiphenyl |
| 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. endosulfan II | |
| 17. 4,4'-DDT | |



15m, 0.25mm ID, 0.25µm Rtx®-CLPesticides column (cat.# 11120). 2µl splitless injection. **Concentration:** 40 to 80ppb. **Oven temp.:** 125°C to 290°C @ 29°C/min. (hold 3 min.); **Inj./det. temp.:** 175°C/300°C; **Inj. port:** Restek split/splitless; **Carrier gas:** hydrogen @ 20psi constant pressure.

Restek Corporation

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INTERNATIONAL

Silcosteel®-Treated GC Injection Ports Prevent Adsorption

by Gary Barone

- Reduces sample breakdown
- Proven inert coating technique

A common problem with splitless, direct, and flash injections in gas chromatography (GC) is containment of a portion of the sample within the volume of the injection port liner. During these injection techniques, large volumes of vapor are formed that exceed the volume of the inlet liner — this is called backflash. Once outside the inlet liner, the solvent and sample vapor may contact the metal surface of the injection port body, causing adsorption or breakdown of the

sample. Restek can coat your injection port body with an inert Silcosteel® treatment that will reduce adsorption or breakdown when backflash occurs.

Restek has been applying Silcosteel® since 1987 to passive transfer lines and components used in analytical systems. The Silcosteel® process permanently bonds a thin, flexible, and inert fused silica coating to the surface of steel and stainless steel components.

This coating acts as a rugged barrier between active components and the adsorptive surface.

Application of the Silcosteel® coating to injection port systems has been used extensively in the process industry and is now available for benchtop instruments. If you have experienced decomposition or adsorption that is not attributable to your column, liner, or detector, try Silcosteel® treatment for your injection port — it may be the answer.

To have your injection port Silcosteel®-coated, simply con-

tact one of our customer service representatives. A catalog number and a Return Authorization (RA) number will be assigned, allowing you to return the injection port to Restek. Send us only the injection port body, without liners or heating block attached. The typical turnaround time is 10 working days from receipt of the port.

Call today for your free brochure on the Silcosteel process (cat.# 59654).

Ultra-Clean XAD®-2 Resin

Amberlite XAD®-2 resin is an excellent adsorbent for trapping PAHs, although it requires extensive clean-up prior to use due to impurities introduced during manufacturing. The TO-13 method specifies a clean-up procedure to prepare the resin for use. However, to meet the minimum contamination level, the XAD®-2 resin may require several cleanings. Many laboratories use their own clean-up procedure, taking a week or more to effectively meet TO-13 method requirements.

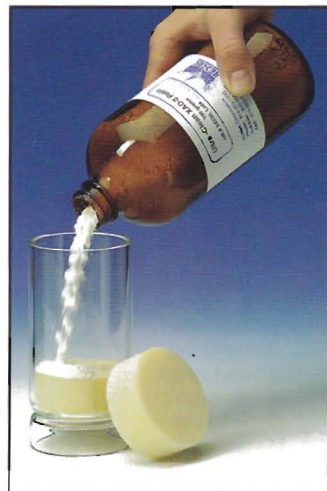
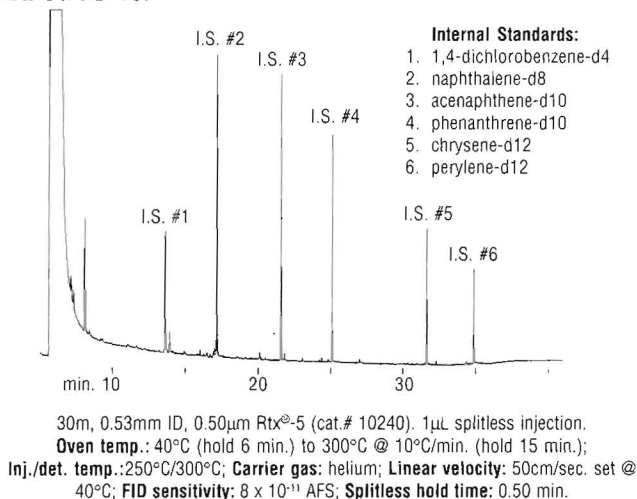
To help laboratories eliminate time-consuming clean-up and still meet TO-13 method requirements, Restek developed Ultra-Clean XAD®-2 resin for semi-volatile air sampling. The clean-up procedure meets the

specified minimum contamination levels and each batch is tested by capillary GC using a flame ionization detector (FID)

to ensure cleanliness. **Figure 1** shows an analysis of a cleaned batch of Amberlite XAD®-2 resin.

Figure 1:

Ultra-Clean XAD®-2 resin meets the requirements of EPA method TO-13.



Product Listing:

Ultra-Clean XAD®-2 Resin

100 grams: cat.# 24230

500 grams: cat.# 24231

Restek Corporation

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INTERNATIONAL



Rtx®-624 for Analysis of Rum and Scotch

by Sherry Sponsler

The maturity and quality of rum and scotch can be determined by the inspection of primary alcohols, acids, ketones, trace esters and fusel oils. The Rtx®-624 is a capillary column that provides excellent resolution and quantitation for these important volatile compounds that characterize aroma and flavor and determine proof value.

Some analysts have traditionally used packed columns. CarboBlack™ packed columns, for example, offer better sample capacity and accommodate larger volume injections. They also provide consistent and adequate separation of isoamyl alcohol and active amyl alcohol (also

known as 3-methyl-1-butanol and 2-methyl-1-butanol), two compounds that influence aroma strength of rum and scotch.

Capillary columns with Carbowax® phases have been used to provide better resolution of the volatile alcohols, acids and esters in rum and scotch, but there are three problems. One problem is consistent baseline resolution between isoamyl alcohol and active amyl alcohol. These compounds are barely resolved on PEG columns, and analysts have complained that this separation is not always consistent nor always achieved from column-to-column. The second problem is that isopropanol and ethanol coelute on Stabilwax® and Rtx®-Wax columns. This affects the

quantitation of ethanol and inhibits the detection of denaturants such as isopropanol and methanol. Third, a thin film is required to achieve the separation of active amyl and isoamyl alcohols, but methanol exhibits a strange asymmetrical peak shape that is only corrected by a thicker-filmed 1.0µm Carbowax® column. So sample capacity is very limited.

The 60m x 0.25mm ID x 1.4µm Rtx®-624 column does not have these problems. Isopropanol does not coelute with ethanol, methanol exhibits symmetrical peak shape, and the separation is much more than just baseline resolution for the active amyl and isoamyl alcohols. Note that these compounds exhibit a reversed elution order than on a CarboBlack™ packed column

or on a Stabilwax® capillary column. This is an excellent column for the analysis of rum (Figure 1). This column also provides a good response to acetic acid, which is quantitated to determine the stage of fermentation or maturity of scotch. (Figure 2).

The Rtx®-624 contains a stationary phase that is 6% cyanopropylphenyl methyl-94% dimethylpolysiloxane. The selectivity of the phase provides excellent resolution and quantitation of the flavor volatiles in rum and scotch, and should be the capillary column of choice for GC.

Product Listing:

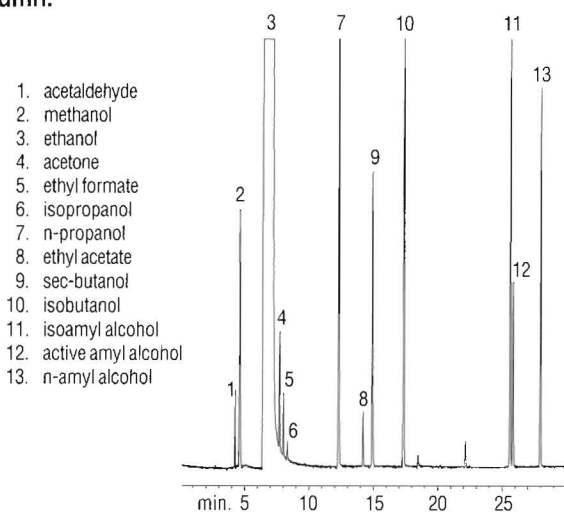
Rtx®-624 Column

60m, 0.25mm ID, 1.4µm

cat.# 10969

Figure 1:

Baseline resolution of rum components on an Rtx®-624 column.

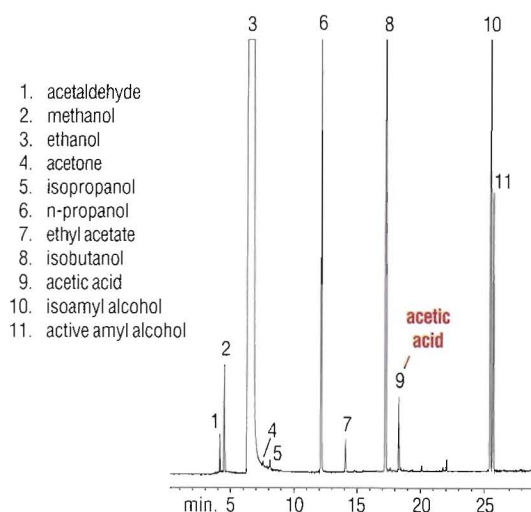


60m, 0.25mm ID, 1.4µm Rtx-624 (cat.# 10969).

1.0µl split injection using a Cyclosplitter® sleeve (cat.# 20706). Concentration: neat; Oven temp.: 35°C (hold 5 min.) to 100°C @ 1°C/min.; Inj./det. temp.: 150°C/200°C; Carrier gas: hydrogen @ 40cm/sec.; Split ratio: 100:1

Figure 2:

Improved analysis of acetic acid on an Rtx®-624 column.



60m, 0.25mm ID, 1.4µm Rtx-624 (cat.# 10969).

1.0µl split injection using a Cyclosplitter® sleeve (cat.# 20706). Concentration: neat; Oven temp.: 35°C (hold 5 min.) to 100°C @ 1°C/min.; Inj./det. temp.: 150°C/200°C; Carrier gas: hydrogen @ 40cm/sec.; Split ratio: 100:1

Professor Kováts Celebrates 70th Birthday at Balaton Symposium

by Dr. David Mannus

I had the pleasure of presenting a paper on our MXT® capillary columns at the Balaton Symposium in Siófok, Hungary during September 3-5, 1997 at the invitation of Klára Verébelyi, owner of LAB-COMP, our distributor in Hungary. After almost twenty years in chromatography sales, my knowledge of the subject would be described as fairly good. However, my knowledge pales into insignificance alongside the great names in chromatography who attended the symposium: Kováts, Nyiredy, Görög, Szepesy, Klebovich, Engelhardt, Davankov and Kaiser as well as those who are associated, but were unable to attend: Horváth and Ettre. It has been my good fortune to work with, and learn a great deal from, Dr. Ettre. During hectic seminar tours of Canada, he was happy to answer all of my questions often illustrating his replies on scraps of paper such as restaurant receipts or airline boarding cards.

The highlight of the symposium was the celebration of the 70th birthday of Professor Ervin sz. Kováts. He was born on September 29, 1927, in Budapest, Hungary, where he grew up. In 1945 he entered the Technical University to study Chemical Engineering. After receiving his Diploma in 1949 he emigrated to Switzerland and, in 1951, received a Chemical Engineering Diploma and two years later his Doctorate from the Federal Technical

University in Zurich. He worked on his Doctoral thesis in the Laboratory of Organic Chemistry under the guidance of Professor Hans H. Gunthardt. This was followed by ten years of intensive work with the Nobel Laureate Professor Leopold Ruzicka on a major project financed by Firmenich & Cie., the leading Swiss manufacturer of essential oils. In 1968 Dr. Kováts was appointed a full professor at the Ecole Polytechnique Fédérale (EPFL) in Lausanne where one year later he became head of the Laboratoire de Chemie Technique. He retired from the EPFL in 1994, after twenty-six years, when he became a research professor at the University of Veszprém, in Hungary, where he continues his work today.

Professor Kováts is best known for the Kováts Retention Index System for substance characterisation in gas chromatography, which he developed during his research into the systematic study of essential oils. Dr. Kováts says "I realized that the logarithmic distance of the peak maxima of the homologues gives marks on the chromatogram similar to those on tape measures used by the tailors. In re-evaluating all my data, the simplest homologous series, that of n-paraffins, was chosen as a base. The fixed points, the peak maxima, were defined as 100 times the carbon number: 500 at the peak of n-pentane, 600 at that of n-hexane, etc. The *retention indices* of other compounds were found by

linear interpolation." It must be remembered that this work was carried out in 1958. Dr. Kováts and his team made their own instrument; the world's first automated preparative gas chromatograph. In the next decade Rohrschneider and also McReynolds used the retention index system for their characterisation of the polarity of stationary phases used in gas chromatography. This characterisation system is still universally used today by Restek and other phase and column manufacturers.

At the gala evening as I enjoyed my coffee and my slice of Dr. Kováts' birthday cake, it occurred to me that, if it

had not been for Dr. Kováts, our president, Paul Silvis, may not have founded Restek Corporation, MXT® columns may never have been invented, and I would not be here enjoying the beauty and ambience of Balaton nor traveling to the many other symposia and trade expositions in Europe. Thank you, Dr. Kováts, from everyone at Restek Corporation.

Dr. Dave Mannus
European Sales Manager

*Visit www.restekcorp.com
for translated versions of
this tribute to Dr. Kováts.*

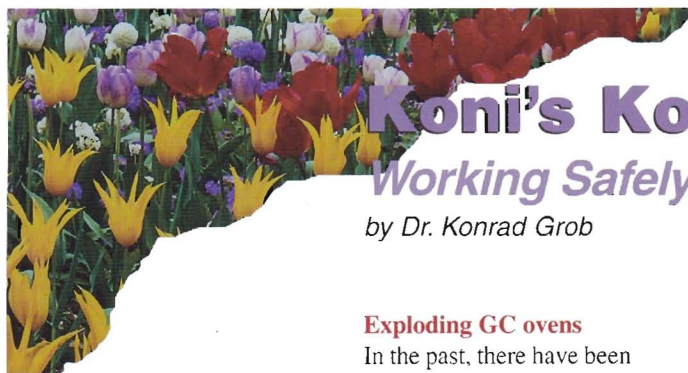


Dr. David Mannus (right), and Klára Verébelyi (left), are proud to honor Dr. Kováts (center) at the Balaton Symposium this past fall.

Restek Corporation

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INTERNATIONAL



Koni's Korner

Working Safely with Hydrogen as a Carrier Gas

by Dr. Konrad Grob

**Broad agreement is
that hydrogen is
the best carrier gas
for capillary GC
applications.**

Nitrogen cannot seriously be considered as a carrier gas option, because diffusion speeds of the solute molecules are roughly four times lower than in hydrogen or helium, rendering the separation process exceedingly slow. Helium is the best alternative if hydrogen cannot be used, but hydrogen enables faster chromatography whenever inlet pressure exceeds roughly 0.7 bar, with a rapidly expanding difference when the required inlet pressure increases. Hydrogen is almost a must for high-temperature work such as triglyceride analysis, and analysis with long columns such as fatty acid methyl ester analysis on 100m columns. Also, hydrogen is available in unlimited amounts (using helium depletes limited natural resources). Hydrogen cannot be used with thermoionic detectors and some mass spectrometers, but the main argument against hydrogen concerns safety because it forms an explosive mixture with air. Can a lab manager take the responsibility for using hydrogen as carrier gas? Yes, if some simple safety measures are taken.

Exploding GC ovens

In the past, there have been many hydrogen explosions in GC ovens. I know of four in Switzerland, the latest one probably being 15 years back. I caused one myself in the seventies during production of glass capillary columns. I prepared about five columns a day using a procedure requiring five heat treatments in a GC oven. I could install a column in hardly more than a second, virtually without turning a screw. I set a high inlet pressure to remove the air and heated the GC at full power to 280°C. One day, a column had enough tension to pull the inlet out of the ferrule. Hydrogen ran into the oven at about 1 bar, without any restriction. The heating filaments were red-hot (2.5 kW, in an old instrument) and ignited the mixture. The explosion was heard through several labs. The instrument seemed to jump 1-2cm from the bench and lost the dust from its top. Opening the door, I was amazed to see the glass capillary column hanging a bit lower than before, but not being broken. The door did not open as smoothly as before, this being the only reminder of the event.

The other explosion in our lab occurred shortly after an on-column injection for triglyceride analysis. We changed the columns almost daily, and the inlet was installed rapidly, with a soft fitting used many times, tightening by fingers. As the syringe needle did not enter the column inlet easily, my coworker pushed a bit harder than usual. The needle went down and he did the injection.

He started the program (with ballistic heating from the injection temperature of 70°C to 250°C) and switched on the recorder. He had just turned his back to the instrument when there was the bang. In fact, he injected into the oven, because the syringe had pushed the column out of the fitting. Again, there was no damage, neither to the instrument, nor to the glass capillary column.

The two other explosions happened in other laboratories, in both instances because it remained unnoticed that the column had been removed from the instrument. Somebody wanted to bake out the column, set high inlet pressure and heated the oven at full power. There was no need for coffee to wake up that morning. The instruments were slightly deformed, but were used for another ten years at least.

4% Hydrogen, 630°C

We had many large leaks with broken columns, poor connections, and experiments more outrageous than can be described here - and there were no explosions. Beside some good luck, this is due to the fact that explosions of hydrogen are much less likely to occur than explosions of solvent vapors. A minimum of 4% hydrogen in air is required for an explosion to occur, and the mixture must be heated to about 630°C to be ignited (in contrast to around 0.1% and hardly over 200°C for vapors of many solvents). In fact, all the explosions I know of occurred with old instruments, characterized by high heating power and

relatively small filaments, which turned red-hot when heating ballistically. Newer instruments with less heating power and larger filaments hardly reach the temperature for ignition. Also, the 4% concentration is not that easily reached; concluding from the experience of many critical situations, it seems that the two most common risks in practice, breakage of the column or a leaking connection to an injector, hardly ever result in an explosion. All explosions I heard of were the result of unhindered flow of hydrogen from the injector into the oven.

MEASURES ENSURING SAFETY

Hydrogen sensors

Risks must be taken seriously, even when it seems unlikely that severe damage may occur. Since the early eighties we gradually equipped all of our instruments with hydrogen sensors, a small device available from various sources. Some air is picked from the zone of the oven ventilator and brought to a sensor detecting hydrogen in the concentration range of 0.1 to 1%. When 1% is reached, the gas chromatograph is switched to cooling, which stops the heating and purges the oven with ambient air. Usually a lamp blinks and an alarm signal calls for attention. A luxury version even replaces the hydrogen in the carrier gas line with nitrogen. The sensor also goes off if concentrations of solvent vapors in the laboratory are high, which eliminates a further risk (independently of whether hydrogen or helium is used as carrier gas).



Flow-regulated carrier gas supply

Classical gas chromatographs regulate the carrier gas by pressure. When there is no column, such systems may deliver many liters of gas per minute into the oven. Hewlett Packard introduced a flow-regulated system, in the worst case limiting the flow into the oven to the rate adjusted for the application, i.e. column plus split plus septum purge flow rate. The maximum flow rate is 500ml/min. Flow regulation strongly reduces risks, but is not considered to be safe. A rough estimation provides some clues. A GC oven has an internal volume of around 40 liters. The explosion limit of 4% is reached when the oven contains 1.6-liter of hydrogen. If the system leaks by 500ml/min, it takes little more than 3 minutes to render the oven atmosphere explosive. If the imposed flow rate is 100ml/min only, however, the critical limit is calculated to be reached in 16 minutes. This assumes a tight oven, which is not realistic; hydrogen diffuses extremely rapidly and the oven atmosphere is exchanged in far less time than 16 minutes. Hence, with a leak of 100ml/min, the hydrogen concentration in the oven cannot reach the explosion limit. Potentially dangerous flow rates exceeding 100ml/min are of interest for split injection with high split ratios. If the high flow rate is turned down 1 minute after the injection, this eliminates this risk and also conserves carrier gas. In splitless injection, the adjusted flow rate will be substantially below 100ml/min anyway. In on-column injection, the flow rate during analysis is far below the critical minimum. It depends on the construction of the injector seal, however,

whether the flow rate can be limited to a few milliliters per minute: during injection, a rotating valve leaks at a far higher flow rate, and pressure would collapse when limiting the flow rate to less than about 100ml/min. Hence, flow regulation or a restrictor limiting flow in a pressure-regulated system can exclude a dangerous hydrogen concentration in the oven.

Intermediate storage in small cylinders

Rather frequently a column is dismantled without switching off the carrier gas supply. Other times a septum leaks to such an extent that the hot carrier gas can burn fingers held many centimeters above the septum. In both these situations, hydrogen may leak into the laboratory at high flow rates. The risk of a laboratory explosion is minimal, however. A small laboratory contains maybe 50,000 liters of air. 4% of hydrogen corresponds to 2000 liters, i.e., to the content of a full 10-liter cylinder at 200 bar pressure. Admittedly, mostly 25- or 50-liter cylinders are used,

but since the laboratory atmosphere is exchanged many times per hour, these cylinders would have to be emptied in minutes to reach the explosion limit. Again, hydrogen is far less dangerous than solvents, for many of which 50-liters of vapor is the limit, corresponding to a spill of around 250ml of liquid solvent. And, since the solvent vapors tend to form a "lake" above the floor, the explosion limit is reached locally even more rapidly. A simple safety measure rules out large scale loss of hydrogen and is also a warning of massive leaks: The main reservoir, maybe a 50-liter cylinder, is not directly connected to the laboratory gas supply, but via a small daily reservoir. The large tank is closed (except when almost empty). The gas is consumed from a 3- to 10-liter cylinder, adjusted to the daily consumption. A 3-liter cylinder filled up to 50 bar contains 150 liters of hydrogen. If, for instance, three gas chromatographs consume 100ml/min each (25ml/min for the FID), the cylinder must be refilled every 25 hours, which

suits when these instruments work day and night. If filled to 20 bar only, it would contain an amount well fitting the consumption of a working day. A manometer or an electronic readout is positioned such that everybody will see it. If the daily reservoir must be refilled on the same day, this is a warning and a leak will be detected long before the large reservoir is emptied. The manometer can be equipped with an alarm indicating low pressure. In Switzerland, this is a standard installation found in many laboratories for more than 20 years.

Hydrogen generators

Hydrogen generators are an alternative to the small daily reservoir. They deliver the gas at limited flow rates and totally avoid the necessity of storing gas. If split injection must be possible at high split flow rate, however, rather large generators and/or one for every few instruments are needed.

Conclusion

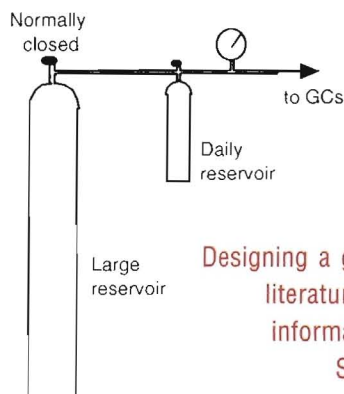
I understand the dilemma of the laboratory manager: he or she may recognize that hydrogen is preferable and cheaper than helium, but does not want to take risks. Can these risks be managed? Hydrogen sensors rule out explosions in the oven, and daily reservoirs or hydrogen generators eliminate risks in the laboratory (as well as costs caused by large losses). Checking tightness of the gas plumbing every 6-12 months is also advisable whether hydrogen or helium is used.

Dr. Konrad Grob can be reached by E-Mail at Koni@grob.org

Figure 1:

Gas supply system ruling out large losses:

Gas is consumed from a daily reservoir and the well-observable manometer provides control over the gas consumption.



Designing a gas system? Request literature packet #59625 for information on Purus™ Gas Systems from Restek.



Genuine Restek Replacement Parts

Making GCs Work Better

“Restek was founded on supplying superior quality, low-cost manufactured parts and accessories for GCs. Restek offers a complete line of GC operating supplies and maintenance parts — from our design innovations to our original manufacturer replacement parts. At Restek, we do whatever it takes to provide you with the best products.”

The GC Accessories Team



Innovation

This is more than making new columns or superior chemical standards. This also means helping you optimize your entire GC system, from inlet to detector. Restek has designed and manufactured GC accessories for 13 years, and we improve upon, or innovate, the existing products. For example, we offer deactivated injection ports to minimize sample breakdown. Whether it's to make your job easier, to improve your chromatography, or to save you time and money, we strive to be innovative.

Quality

Every product we manufacture is carefully evaluated and tested to ensure high quality. We actually use the products we make in our applications and QA labs — before you use it, we use it. However, we think quality goes beyond in-house testing. A lot tracking system lets us trace products to the end user so we know from where your individual product originated. And, we have a customer response team dedicated to ensuring your satisfaction after the sale.

Technical Service

We have chemists on call to answer your chromatography questions. Before joining Restek, our experienced

scientists have used the products and worked in outside labs. Real life answers for your real-world problems.

Price

We offer competitive pricing on all GC accessories. Compare our prices to what you currently pay and you'll see we offer the best value.

Delivery

The final element in meeting your needs is fast delivery. More than 97% of our products are shipped the same day the order is placed. We pride ourselves on getting the job done right and on time. Need a part in an emergency? We can ship via overnight delivery for emergency orders placed as late as 5:30pm EST.

If you have a specific accessory design requirement, please contact your local distributor.

Peak Performers

Replacement Reducing Nut for the HP 5890/6890 GC



Restek now offers the replacement reducing nut for the HP 5890/6890 GCs. Made from high quality stainless steel and meets original equipment specifications.

Genuine Restek Replacement for HP Part No. 18740-20800. **Cat.# 22078, ea.**

Replacement FID/NPD Capillary Adaptor for the HP 5890/6890 GC



Restek now offers the FID/NPD replacement capillary adaptor for the HP 5890/6890 GCs. Made from high quality stainless steel and meets original equipment specifications.

Genuine Restek Replacement for HP Part No. 19244-80610. **Cat.# 22077, ea.**

Capillary FID Jets for the HP 6890 GC



Restek now offers two jets specifically designed for the HP 6890 GC. The Standard version made from high quality stainless steel and the High Performance version which is Silcosteel®-treated for optimum inertness. Both are designed with a fluted tip to guide the capillary column into the jet.

Genuine Restek Replacement for HP Part No. G1531-80560.

Standard:

Cat.# 21621, ea.
Cat.# 21682, 3-pack

High Performance:

Cat.# 21620, ea.
Cat.# 21683, 3-pack

Air Diverters for the HP 5890 GC



Direct the GC exhaust heat away from the lab!

These Air Diverters are made from high-quality aluminum and meet OEM specifications. Installation is easy.

Genuine Restek Replacement for HP Part No. 19247-60510. **Cat. #22076, ea.**

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Other trademarks: Hypersil (Hypersil), Inertsil (GL Sciences), Kromasil (Eka Nobel), Nucleosil (Macherey-Nagel), and Carbowax (Union Carbide Corp.), XAD (Rohm & Haas, Co.).

Updated Toxic Organic (TO) Methods Available on CD-ROM

Restek now offers an easy-to-use, updated version of the *Compendium of Methods for the Determination of Toxic Organic Compounds in Air** on CD-ROM. This version includes the new second edition methods: TO-4a, TO-9a, TO-10a, TO-11a, TO-13a, TO-14a, TO-15, TO-16 and TO-17, and the non-edited first edition methods: TO-1, TO-2, TO-3, TO-5, TO-6, TO-7, TO-8 and TO-12.

A built-in search feature easily tracks down specific information. The key words entered will be searched across the entire compendium and all

- Contains methods TO-1 through TO-17
- Includes the new second edition methods and the non-edited first edition methods
- Includes Adobe® Acrobat Reader® software

individual documents containing the term will appear in a list.

Two tables are also included to help find the appropriate method for your application: Brief Method Description and Applicability, and Method Applicability to Compounds of Primary Interest.

The compendium CD-ROM is easy to use. The entire collection is built around a

familiar and intuitive "homepage" model, and navigating the exhaustive list of TO-Methods is as easy as pointing and clicking. Visit our web site at <http://www.restekcorp.com> for a demonstration, or call us and try it yourself!

** These methods were written by the U.S. Environmental Protection Agency's (EPA) National Risk Management Research Laboratory (NRMRL). The 2nd edition was released in the fall of 1997.*



Product Listing:

Compendium of Methods for the Determination of Toxic Organic Compounds in Air, on CD-ROM

cat.# 24280

Restek Corporation

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INTERNATIONAL