


# the Reporter

FOR EUROPE MAGAZINE

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## Editorial

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Kind regards  
Dr. Ingo Haag



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## HPLC Article

# Assessing Column Comparison Studies: Modified-Euerby Comparison of Discovery Zr-PBD and Discovery C18

Many reports discussing column screening procedures for purposes of stationary phase selection have recently become prominent in analytical journals. This report discusses the use of a modified-Euerby column classification study as a means to gain further understanding of a polybutadiene-coated zirconia stationary phase. Although silica-based stationary phases remain the workhorse for high-performance liquid chromatography (HPLC) analyses, separations based on modified zirconia phases are fast becoming a popular alternative. The interest in zirconia columns stems from their ability to withstand extreme pH and temperature conditions as well as their offering of unique selectivity and retention for various classes of compounds. The merits of the adopted procedure and the information it provided are discussed.

### Introduction

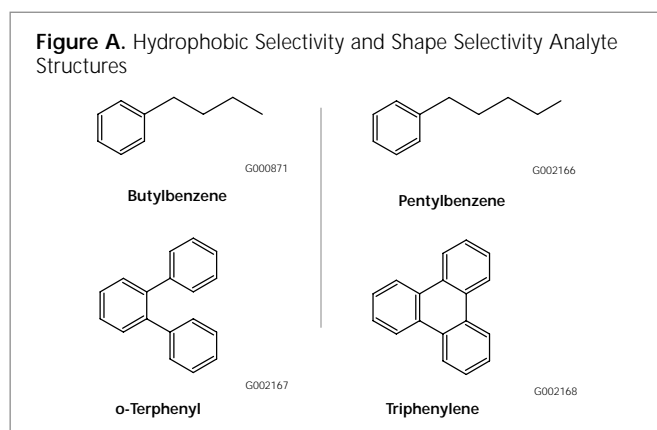
To best utilize the unique offerings of any stationary phase it is paramount that the underlying mechanisms of interaction be understood. Many reports discussing column classification studies can be found in recent literature (1,2,3). These types of studies may:

1. Demonstrate chromatographic orthogonality between stationary phases of differing bonding chemistries and substrate structure (a method development aid)
2. Demonstrate similarity in columns (a method transfer aid)
3. Help elucidate molecular interactions available on a given phase chemistry

There are several test procedures in analytical journals with significant data to assess their applicability. The NIST Standard Reference Material® 870 consists of uracil (to marker), toluene, ethylbenzene (hydrophobic retention markers), quinazoline (activity toward chelators) and amitriptyline (activity toward bases). Sander and Wise have evaluated the test and found it suitable to compare common reversed-phase systems such as C18 and polar embedded phases<sup>(1)</sup>. Euerby, et. al. recently reported the use of the well-known Tanaka test procedure to evaluate 135 stationary phases of differing chemistries<sup>(2)</sup>. The results of this testing are discussed in terms of hydrophobicity, shape selectivity, hydrogen bonding capacity, total ion-exchange capacity and acidic ion-exchange capacity. A third report recently published by Neue, et. al., discusses still another set of test conditions<sup>(3)</sup>. This latter report again describes parameters such as hydrophobicity and shape selectivity. In addition, silanophilic interactions as well as a "phenolic selectivity" parameter are discussed. Each of the test procedures noted have advantages and disadvantages. The fact that good retention data were obtained for the vastly differing phase chemistries using the Euerby procedure suggests that the conditions are universal enough to cover a wide range of chemistries and supports. Both the NIST procedure and the Neue procedure measure silanophilic interactions near neutral pH. Since silanophilic interactions are pH dependent, the additional set of conditions of the Euerby procedure at acidic pH is expected to add significant information regarding these important interactions. One disadvantage of the Euerby procedure is the lack of complete ionization of the benzylamine analyte used to determine ionic interaction at pH 7.6 (4). This incomplete ionization may cause some irreproducibility, therefore the addition of the quaternary amine, berberine, has

been added to eliminate the dependence of hydrophobic interaction on pH. The addition of berberine was expected to better isolate the changes in phase characteristics as a function of pH. The other missing component to the mixture, which may provide significant information, is an acid. Since many analytes contain carboxylic acids, the addition of such a compound should provide essential data for method developers as well as for stationary phase research and development efforts. The carboxylic acid moiety generally exhibits a pKa value of about 4.5. This value is reasonably centered between the 2.7 and 7.6 pH conditions used in the Euerby tests and thus was expected to provide information in both the ionized and neutral states. The term modified-Euerby is used in this report to describe a procedure where the stated analyte additions and a few minor changes in mobile phase to increase analyte retention were made to the literature procedure.

In this study the modified-Euerby procedure was used to examine the retention characteristics of Discovery Zr-PBD as compared to Discovery C18. Hydrophobic selectivity, shape



selectivity, hydrogen bonding capacity, and total ion exchange capacity at both pH 2.7 and 7.6 were determined. The values obtained were evaluated qualitatively against the observed chromatograms. The merits of the procedure and the information it provided are discussed.

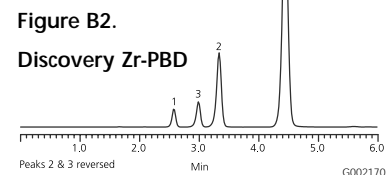
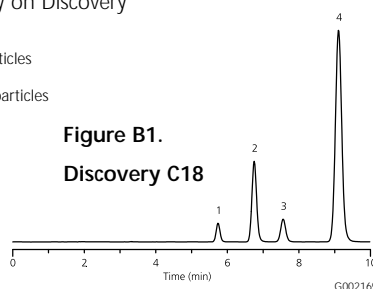
### Results and Discussion

Figure A shows the structures of the hydrophobic selectivity probes, pentylbenzene (PB) and butylbenzene (BB) as well as the shape selectivity probes triphenylene (T) and o-terphenyl (O). The observed chromatograms are shown in Figure B and the numerical results are listed in Table I. Similar hydrophobic selectivity values were obtained for each of the phases, however, much greater resolution of PB and BB is observed in the chromatographic trace. As discussed in the Case Study of this Reporter, low retention, as is observed for these analytes on Zr-PBD, results in irreproducible values. The calculated values for shape selectivity indicate that the Zr-PBD column is more shape selective than the C18. Examination of the chromatogram, however, shows that the C18 exhibits greater resolution of the probe analytes. In this case, the capacity factors for the analytes, T and O, on the Zr-PBD are greater, however, they may still be too low for accurate parameter measurement.

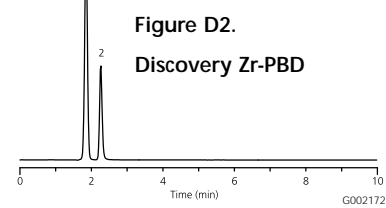
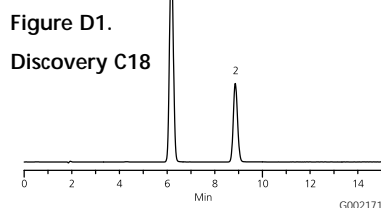
Figure C shows the structures of the hydrogen bonding

**Figure B. Hydrophobic Selectivity and Steric Selectivity on Discovery C18 and Discovery Zr-PBD**

Column 1: Discovery C18, 15cm x 4.6mm ID, 5µm particles  
 Cat. No.: 504955-U  
 Column 2: Discovery Zr-PBD, 15cm x 4.6mm ID, 5µm particles  
 Cat. No.: 65718-U  
 Mobile Phase: 20:80 water:methanol  
 Flow Rate: 1.0ml/min  
 Temp.: 40°C  
 Det.: UV, 254nm  
 Inj.: 5µL  
 Sample:  
 1. Butylbenzene, BB (0.3mg/ml) 2. o-Terphenyl, O (0.02mg/ml)  
 3. Pentylbenzene, PB (0.6mg/ml) 4. Triphenylene, T (0.05mg/ml)

**Figure D. Hydrogen Bonding Selectivity on Discovery C18 and Discovery Zr-PBD**

Column 1: Discovery C18, 15cm x 4.6mm ID, 5µm particles  
 Cat. No.: 504955-U  
 Column 2: Discovery Zr-PBD, 15cm x 4.6mm ID, 5µm particles  
 Cat. No.: 65718-U  
 Mobile Phase: 20:80 water:methanol  
 Flow Rate: 1.0ml/min  
 Temp.: 40°C  
 Det.: UV, 254nm  
 Inj.: 5µL  
 Sample:  
 1. Caffeine, C (0.5mg/ml)  
 2. Phenol, P (1.0mg/ml)

**Table I. Hydrophobic Selectivity and Shape Selectivity Results**

Phase	PB (k')	BB (k')	Hydrophobic Selectivity (k'PB/k'BB)	T (k')	O (k')	Shape Selectivity (k'T/k'O)
C18	3.198	2.189	1.406	4.053	2.750	1.474
Zr-PBD	0.864	0.607	1.423	1.768	1.082	1.634

**Table II. Hydrogen Bonding Capacity Results Selectivity Results**

Phase	C (k')	P (k')	Hydrophobic Capacity (k'c/k'p)
C18	2.246	3.652	0.615
Zr-PBD	0.109	0.355	0.307

capacity probes, caffeine (C) and phenol (P). The obtained chromatograms are given in Figure D and the numerical values obtained are listed in Table II. Low retention for both C and P on the Zr-PBD phase results in low confidence in the calculated values. The chromatograms show little significant difference in selectivity other than that attributable to lower hydrophobic interaction of the Zr-PBD phase.

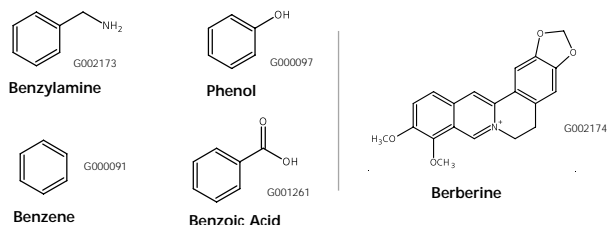
Figure E presents the structures of the ion-exchange probes used for both the pH 2.7 and the pH 7.6 studies. The chromatograms obtained at pH 2.7 are shown in Figure F and the numerical results are given in Table III. The original Euerby method defines the ratio of benzylamine and phenol capacity factors as the total ion-exchange capacity. The data in Table III shows that the retention for benzylamine and phenol are quite low for both phases. As indicated before, this produces questionable results. The retention for berberine and benzene

**Figure C. Hydrogen Bonding Analyte Structures**

under the same conditions appears to be adequate for determining this parameter. In addition, the use of a quaternary amine negates effects due to incomplete ionization. The values obtained using k'BB/k'BZ indicate that the Zr-PBD exhibits greater ion-exchange capacity under these conditions when compared to C18. Examination of the chromatograms supports the obtained values. The Zr-PBD shows greater retention for the basic analytes benzylamine and berberine. The ion-exchange character, combined with the lower hydrophobicity of the phase, results in selectivity differences between the two

columns. Neutral benzoic acid is preferentially retained on the C18 phase, corresponding to the greater hydrophobicity of the C18 stationary phase.

The chromatograms obtained at pH 7.6 are presented in Figure G and the numerical results are listed in Table IV. Once again the retention of benzylamine is observed to be very low. Both berberine and benzene, however, are again well retained. The larger total ion-exchange value obtained for the Zr-PBD phase is supported by the preferential retention of benzylamine and berberine observed in the chromatographic traces. At pH 7.6, benzoic acid is expected to be fully ionized. The benzoic acid analyte appears to be excluded from the pores of the Zr-PBD phase (elutes before the void volume), which further supports that the surface of the Zr-PBD is negatively charged.

**Figure E. Total Ion-Exchange Capacity at Both pH 2.7 and pH 7.6 Analyte Structures**

## Conclusions

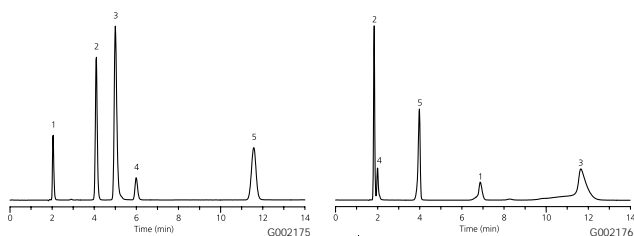
In this study, the suitability of column classification procedures such as the Euerby method for determining phase orthogonality, similarity, and dominant retention mechanisms were investigated. Where low retention was observed, discrepancies between the obtained values and the qualitative examination of the chromatographic traces were evident. This study, coupled with other current investigations, suggests that adequate retention ( $k' > 1.5$ ) is required for the generation of

**Figure F. Total Ion-Exchange Capacity at pH 2.7 on Discovery C18 and Discovery Zr-PBD**

Column 1: Discovery C18, 15cm x 4.6mm ID, 5µm particles  
 Cat. No.: 504955-U  
 Column 2: Discovery Zr-PBD, 15cm x 4.6mm ID, 5µm particles  
 Cat. No.: 65718-U  
 Mobile Phase: 60:40, 20mM KH<sub>2</sub>PO<sub>4</sub>, pH 2.7 with H<sub>3</sub>PO<sub>4</sub>:methanol  
 Flow Rate: 1.0ml/min  
 Temp.: 40°C  
 Det.: UV, 254nm  
 Inj.: 5µL  
 Sample:  
 1. Benzylamine, B (0.5mg/ml) 2. Phenol, P (0.5mg/ml) 3. Berberine, BN (0.1mg/ml)  
 4. Benzoic Acid, BA (0.1mg/ml) 5. Benzene, BZ (1.0mg/ml)

**Figure F1. Discovery C18**

**Figure F2. Discovery Zr-PBD**

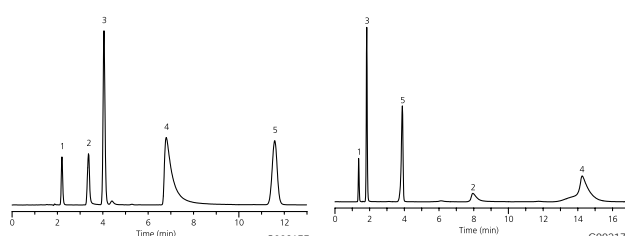


**Figure G. Total Ion-Exchange Capacity at pH 7.6 on Discovery C18 and Discovery Zr-PBD**

Column 1: Discovery C18, 15cm x 4.6mm ID, 5µm particles  
 Cat. No.: 504955-U  
 Column 2: Discovery Zr-PBD, 15cm x 4.6mm ID, 5µm particles  
 Cat. No.: 65718-U  
 Mobile Phase: 60:40, 20mM KH<sub>2</sub>PO<sub>4</sub>, pH 7.6 with H<sub>3</sub>PO<sub>4</sub>:methanol  
 Flow Rate: 1.0ml/min  
 Temp.: 40°C  
 Det.: UV, 254nm  
 Inj.: 5µL  
 Sample:  
 1. Benzoic Acid, BA (0.1mg/ml) 2. Benzylamine, B (0.5mg/ml) 3. Phenol, P (0.5mg/ml)  
 4. Berberine, BN (0.1mg/ml) 5. Benzene, BZ (1.0mg/ml)

**Figure G1. Discovery C18**

**Figure G2. Discovery Zr-PBD**



**Table III. Total Ion-Exchange, pH 2.7 Results Selectivity Results**

Phase	B (k')	P (k')	Total Ion Exchange Capacity (k'B/k'P)	BB (k')	BZ (k')	Total Ion Exchange Capacity (k'BB/k'BZ)	BA (k')
C18	0.077	1.145	0.0672	1.609	5.065	0.318	2.136
Zr-PBD	3.237	0.139	23.288	6.185	1.457	4.245	0.239

**Table IV. Total Ion-Exchange, pH 7.6 Results Selectivity Results**

Phase	B (k')	P (k')	Total Ion Exchange Capacity (k'B/k'P)	BB (k')	BZ (k')	Total Ion Exchange Capacity (k'BB/k'BZ)	BA (k')
C18	0.783	1.145	0.684	2.599	5.113	0.508	0.167
Zr-PBD	4.011	0.165	24.309	8.075	1.456	5.546	-0.128

reproducible values. As columns vary significantly in their retention characteristics, it is difficult or impossible to develop a single set of conditions that can produce adequate retention. It is therefore recommended that qualitative analysis of the chromatographic traces be included in the overall data analysis scheme.

The addition of berberine and benzene proved to be a significant improvement over the literature procedure. At both pH levels, benzylamine was only marginally retained and, as discussed earlier, this low retention reduces confidence in the parameter values. The addition of benzoic acid was only marginally useful in this case.

Combining the quantitative and qualitative data from this study, the Zr-PBD was shown to exhibit less hydrophobic

retention as compared to C18. Shape selectivity and hydrogen bonding capacity were similar between the two phases. Total ion-exchange studies revealed a significant contribution of ionic interactions to retention using the Zr-PBD under these conditions. The data also show that the Zr-PBD is significantly different than the C18 and therefore is not likely to be a direct replacement for a given set of conditions. If a reduction in hydrophobic retention or an increase in basic analyte retention compared to a C18 is desired, the Zr-PBD may be a suitable alternative.

References

- L.C. Sander, S.A. Wise, Journal of Separation Science 26 (2003) 283.
- M.R. Euerby, P. Petersson, Journal of Chromatography A 994 (2003) 13.
- U.D. Neue, K. VanTran, P.C. Iraneta, B.A. Alden, Journal of Separation Science 26 (2003) 174.
- U.D. Neue, E. Serowik, P. Iraneta, B.A. Alden, T.H. Walter, Journal of Chromatography A 849 (1999) 87.

**Ordering information**

**Discovery Zr-PBD**

Prod. No.	Description
<b>3 micron</b>	
65713-U	5cm x 2.1mm
65714-U	7.5cm x 2.1mm
65715-U	15cm x 2.1mm
65716-U	5cm x 4.6mm
65717-U	7.5cm x 4.6mm
65718-U	15cm x 4.6mm
65811-U	1cm x 2.1mm Supelguard Cartridge Kit
65813-U	1cm x 4mm Supelguard Cartridge Kit
65812-U	1cm x 2.1mm Supelguard Cartridges, pk. of 2
65814-U	1cm x 4mm Supelguard Cartridges, pk. of 2
<b>5 micron</b>	
65719-U	5cm x 2.1mm
65720-U	15cm x 2.1mm
65722-U	5cm x 4.6mm
65723-U	15cm x 4.6mm
65724-U	25cm x 4.6mm
65815-U	1cm x 2.1mm Supelguard Cartridge Kit
65817-U	1cm x 4mm Supelguard Cartridge Kit
65816-U	1cm x 2.1mm Supelguard Cartridges, pk. of 2
65818-U	1cm x 4mm Supelguard Cartridges, pk. of 2

**Ordering information**

**Discovery C18 5µm Particles**

Prod. No.	ID (mm)	Length (cm)
50494721	2.1	5.0
569220-U	2.1	10.0
569229-U	2.1	12.5
50495521	2.1	15.0
504947-30	3.0	5.0
569221-U	3.0	10.0
569230-U	3.0	12.5
504955-30	3.0	15.0
504971-30	3.0	25.0
504947-40	4.0	5.0
569222-U	4.0	10.0
569231-U	4.0	12.5
504955-40	4.0	15.0
504971-40	4.0	25.0
504947	4.6	5.0
569223-U	4.6	10.0
569232-U	4.6	12.5
504955	4.6	15.0
504971	4.6	25.0
569224-U	10.0	25.0
569226-U	21.2	25.0

**i** Information Request.....1001

**Your Problem Solving Partner in Chromatography**

# HPLC Case Study

## Reproducibility of Column Classification Study Parameters

There has recently been a renewed interest in HPLC column classification studies as indicated by the numerous publications on the subject over the past couple of years. The studies are of interest to the practitioner as well as column manufacturers as they may provide information on column similarity and stationary phase orthogonality, as well as information regarding the dominant interactions for a given phase. In this study, the quality of parameters that quantitatively describe the various column attributes is assessed in terms of the probing analyte retention. The data suggests that adequate retention of the probing analytes must be obtained on a given phase to generate reproducible values.

### Introduction

As discussed in the Main Article of this Reporter, column classification studies are often used to assess the similarities and differences between phases, as well as insights into the molecular interactions that result in analyte retention. In some cases, low retention times for probe analytes using literature procedures has been observed. The purpose of this study was to assess how retention affects the reproducibility of the column characteristic parameters.

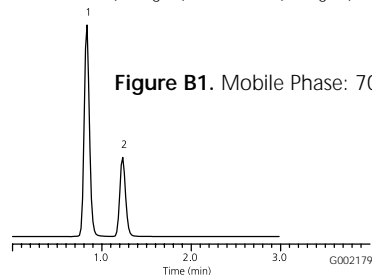
The hydrogen bonding capacity for Discovery C18, according to the procedure of Euerby<sup>(1)</sup>, was evaluated using both a 5cm and 15cm length column. The study was repeated after adjusting the water:methanol ratio in the mobile phase from the literature ratio of 70:30 to 80:20 to increase retention of the probing analytes. The reproducibility of values obtained under both conditions was evaluated.

**Figure A:** Structures of Hydrogen Bonding Capacity Probes



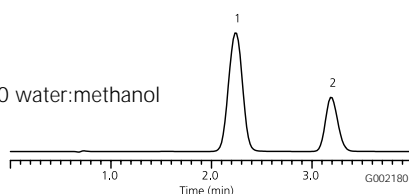
**Figure B.** Chromatograms Obtained on 5cm Discovery C18

Column: Discovery C18, 15cm x 4.6mm ID, 5 $\mu$ m particles  
Cat. No.: 504947-U  
Mobile Phase: 80:20 water:methanol or 70:30 water:methanol  
Flow Rate: 1.0ml/min  
Temperature: 40°C  
Det.: UV, 254nm  
Inj.: 5ml  
Sample: 1. Caffeine, C (0.5mg/ml) 2. Phenol, P (1.0mg/ml)



**Figure B2.**

Mobile Phase: 80:20 water:methanol

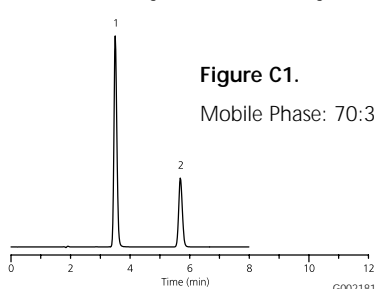


### Results and Discussion

The structures of the analyte probes are presented in Figure A. The chromatographic traces obtained are given in Figures B and C and the retention data are listed in Table I. The characteristic parameters for a given phase should be similar regardless of the geometry of the column used. The calculated hydrogen bonding capacity values using the 70:30 water:methanol mobile phase were 0.210 and 0.427 for the 5cm and 15cm columns, respectively. Using the more retentive 80:20 water:methanol mobile phase, the results were nearly identical. Clearly, the more retentive system provided more reproducible values.

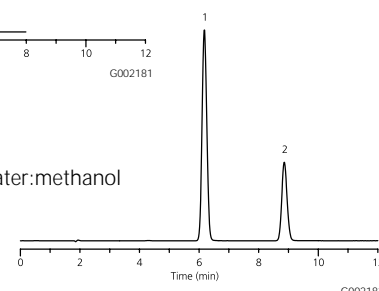
**Figure C.** Chromatograms Obtained on 15cm Discovery C18

Column: Discovery C18, 15cm x 4.6mm ID, 5 $\mu$ m particles  
Cat. No.: 504955-U  
Mobile Phase: 80:20 water:methanol or 70:30 water:methanol  
Flow Rate: 1.0ml/min  
Temperature: 40°C  
Det.: UV, 254nm  
Inj.: 5ml  
Sample: 1. Caffeine, C (0.5mg/ml) 2. Phenol, P (1.0mg/ml)



**Figure C2.**

Mobile Phase: 80:20 water:methanol



**Table I.** Probing Analyte Retention Data

Discovery C18	Mobile Phase(Ratio) (water:methanol)	C (k')	P (k')	Hydrogen Bonding Capacity (k' <sub>c</sub> /k' <sub>p</sub> )
5cm x 4.6mm ID	70:30	0.143	0.682	0.210
5cm x 4.6mm ID	80:20	2.001	3.265	0.616
15cm x 4.6mm ID	70:30	0.840	1.983	0.427
15cm x 4.6mm ID	80:20	2.246	3.652	0.615

### Conclusions

The results of this study indicate that adequate retention is necessary to generate reproducible parameter values. When column characteristic parameter values are used to assess column similarity or orthogonality, care should be taken to ensure their validity. As discussed in the previous Article of this Reporter, qualitative examination of the corresponding chromatographic traces are necessary when absolute retention data are not provided. Based on this and other similar studies, capacity factors of greater than or equal to 1.5 appear to generate reproducible and reliable data.

### References

- M.R. Euerby, P. Petersson, Journal of Chromatography A 994 (2003) 13.

Ordering information (see page 9)

**i** Information Request .....1002

## HPLC Article

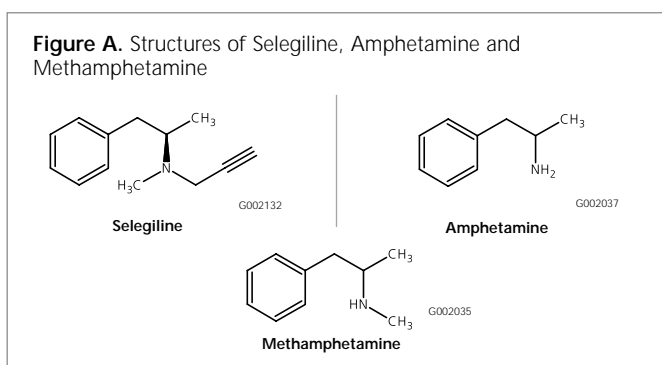
# Effect of Ion-Pairing Modifier Purity on HPLC Quantitation

For developing precise and robust HPLC methods, and to ensure their reproducibility, high purity solvents and reagents are critical. In this study, the effect of ion-pairing reagent purity on the quantitation of impurities in selegiline was assessed. Baseline instability and ghost peaks due to impurities in ion-pairing reagents were shown to produce an increase in method irreproducibility.

### Introduction

The use of low purity reagents in HPLC analyses can adversely affect method precision and robustness. The effects of low purity reagents are often manifested as positive or negative ghost peaks and/or poor baseline stability. These effects are particularly prevalent at low ultra-violet (UV) wavelengths.

In this study, the effect of ion-pairing reagent quality on the reproducibility of a selegiline purity assay was investigated. Two mobile phases were prepared that differed only in the ion-pairing reagent used. In one case, a highly purified ion-pairing reagent was employed. In the other, a reagent known to be of lesser quality was used. The effect was quantitatively assessed based on reproducibility data and qualitatively evaluated based on examination of the resulting chromatographic traces.



### Results

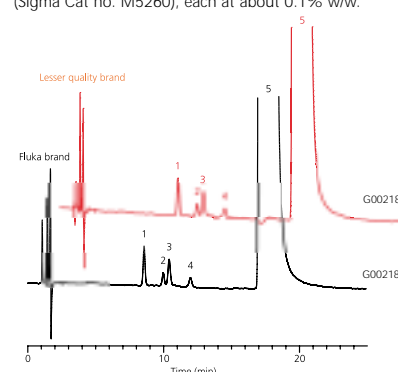
The structures of selegiline, amphetamine and methamphetamine are presented in Figure A. The chromatographic traces obtained using the different ion-pairing reagents are shown in Figure B. Qualitative examination of the chromatographic traces shows that baseline drift is more apparent using the lesser quality reagent. In addition, a negative "ghost" peak is observed around 15 minutes using the lesser quality reagent that is not apparent using the Fluka

**Figure B. Comparison of Chromatograms Obtained Using Fluka and Lesser Quality Reagents**

Column: Discovery HS C18, 15cm x 4.6mm ID, 3µm  
 Cat. No.: 569252-U  
 Mobile Phase: 80:20, 5mM heptanesulfonic acid\* in 10mM KH<sub>2</sub>PO<sub>4</sub>, pH to 3 w/ H<sub>3</sub>PO<sub>4</sub>: CH<sub>3</sub>CN  
 Flow Rate: 1.2ml/min  
 Temperature: 35°C  
 Det.: UV, 205nm  
 Inj.: 10ml  
 \* Mobile Phase 1: Fluka brand heptanesulfonic acid (Cat#: 51832)  
 Mobile Phase 2: lesser quality brand heptanesulfonic acid  
 Sample Preparation: A 1mg/ml solution of selegiline ((-)-Deprenyl, Sigma Cat no. M003) in mobile phase was spiked with amphetamine (Sigma Cat no. A2262) and methamphetamine (Sigma Cat no. M5260), each at about 0.1% w/w.

- Peak Identification:  
 1. Amphetamine  
 2. Unknown 1  
 3. Methamphetamine  
 4. Unknown 2  
 5. Selegiline

The sample solution was analyzed 6 times using each of the mobile phase prepared. Selegiline impurity responses were recorded and compared.



reagent. Any selegiline impurities that might retain in this area would be adversely affected by the baseline inflection.

A quantitative comparison of the two systems is provided in Table I. The absolute selegiline impurity responses were similar, however, the reproducibility of the measurements was superior using the more purified reagent. Three of the four observed selegiline impurities exhibited higher percent coefficients of variation using the lesser quality reagent.

### Conclusions

The purity of reagents and solvents used in HPLC analyses can affect method precision and reproducibility. The high purity Fluka brand heptanesulfonic acid ion-pair reagent was shown to be superior to a lesser quality reagent in terms of baseline stability, lack of ghost peaks and their cumulative effects on reproducibility. Although the differences in this case were not striking, under certain conditions the ramifications of impure reagents can be dramatic.

**i** Information Request .....1002

**Table I. Quantitative Comparison of Fluka and Lesser Quality Ion-Pair Reagents**

Fluka Reagent									
Analyte	Inj 1	Inj 2	Inj 3	Inj 4	Inj 5	Inj 6	Avg	Stdev	%CV
Amphetamine	21.47	21.32	21.11	21.43	21.7	21.63	21.44	0.21	1.00
Unknown 1	7.49	7.63	7.71	7.78	7.49	7.79	7.65	0.14	1.77
Methamphetamine	17.58	17.27	17.98	17.84	17.47	17.85	17.67	0.27	1.53
Unknown 2	7.27	7.22	6.96	7.90	7.46	7.11	7.32	0.33	4.50
Selegiline	15784	15778.80	15777.40	15777.40	15768.70	15772.90	15776.53	5.23	0.03
Lesser Quality Reagent									
Analyte	Inj 1	Inj 2	Inj 3	Inj 4	Inj 5	Inj 6	Avg	Stdev	%CV
Amphetamine	21.81	23.08	21.08	21.21	21.95	20.87	21.67	0.81	3.74
Unknown 1	8.05	7.82	8.47	7.86	7.03	8.02	7.88	0.47	6.02
Methamphetamine	17.66	17.69	18.01	17.76	17.53	17.99	17.77	0.19	1.07
Unknown 2	6.87	7.18	7.65	7.51	7.22	6.65	7.18	0.38	5.24
Selegiline	15809.70	15759.80	15771.90	15774.20	15767.80	15788.80	15778.70	17.92	0.11

# Product profile

## Discovery Silica-Based Phases

### Discovery C18

## Classic Reversed-Phase Retention and Selectivity with Excellent Peak Shape

Use Discovery C18 for any method that specifies a C18. The exceptional peak shape, reproducibility, and stability make it the column of choice for all C18 methods from demanding to routine.

- Classic C18 selectivity and retention
- Excellent peak shape
- Stable, no-bleed LC/MS separations

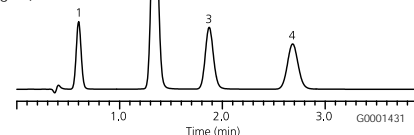
#### Properties of Discovery C18

USP Code	L1
Bonded Phase	Octadecylsilane
Endcap (yes / no)	Yes
Particle Platform	Silica
Particle Shape	Spherical
Particle Purity	<10ppm metals
Particle Sizes (µm)	5
Pore Size (Å)	180
Surface Area (m <sup>2</sup> /g)	200
Packing Density (g/ml)	0.58
%C	12
Coverage (µmoles/m <sup>2</sup> )	3
pH Range	2 to 8
Temperature Range	<70°C

**Figure 2.** Organic Acids

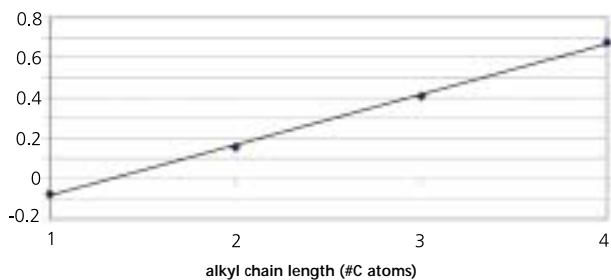
Column: Discovery C18, 5cm x 4.6mm ID, 5µm (504947)  
 Mobile Phase: 60:40, 0.1%TFA in Water:MeOH  
 Flow Rate: 2.0ml/min  
 Temp.: 20°C  
 Det.: UV at 254nm  
 Inj.: 10µL

1. Homovanillic acid (0.0625µg/ml)
2. Sorbic acid (0.00625µg/ml)
3. Salicylic acid (0.0625µg/ml)
4. p-Toluic acid (0.00625µg/ml)



**Figure 1.** Discovery C18 operates via a predictable reversed-phase mechanism. Compounds elute in order of increasing hydrophobicity.

Alkylparabens on Discovery C18  
 Mobile Phase: 60:40 Water:CH<sub>3</sub>CN

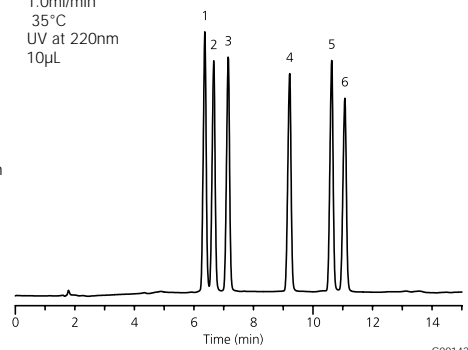


**Figure 3.** Antibiotics (Fluoroquinolones from Tablets)

Column: Discovery C18, 15cm x 4.6mm ID, 5µm (504955)  
 Mobile Phase: (A) 25 mM Potassium Phosphate (pH 3.0) (B) CH<sub>3</sub>CN  
 Flow Rate: 1.0ml/min  
 Temp.: 35°C  
 Det.: UV at 220nm  
 Inj.: 10µL

1. Levofloxacin
2. Ciprofloxacin
3. Lomefloxacin
4. Sparfloxacin
5. Grepafloxacin
6. Trovafloxacin

Gradient:  
 Min %A %B  
 0 90 10  
 15 65 35



## Discovery C18

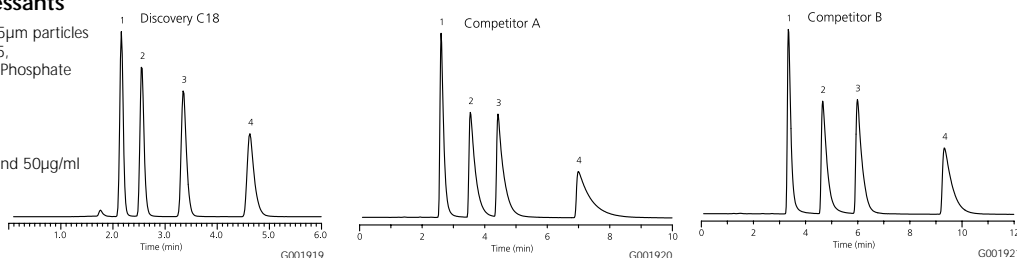
Excellent Peak Shape Compared to Competitive C18 Columns

All Discovery HPLC phases begin with pure, metal-free, high quality silica and employ advanced bonded phase technology. As a result, they give excellent peak shape in simple mobile phases.

**Figure 1. Tricyclic Antidepressants**

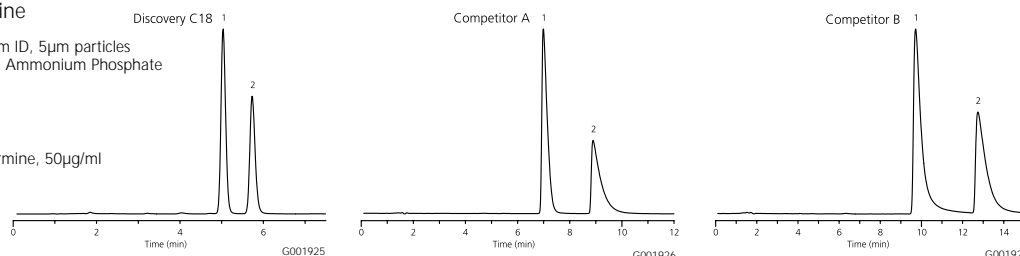
Columns: 15cm x 4.6mm ID, 5µm particles  
 Mobile Phase: 55:45, 25mM Ammonium Phosphate  
 CH<sub>3</sub>CN  
 (pH 7):  
 Flow Rate: 1ml/min  
 Temp.: 30°C  
 Det.: UV at 254nm  
 Sample: 10µL, each compound 50µg/ml

1. Nordoxepin
2. Nortriptyline
3. Doxepin
4. Amitriptyline



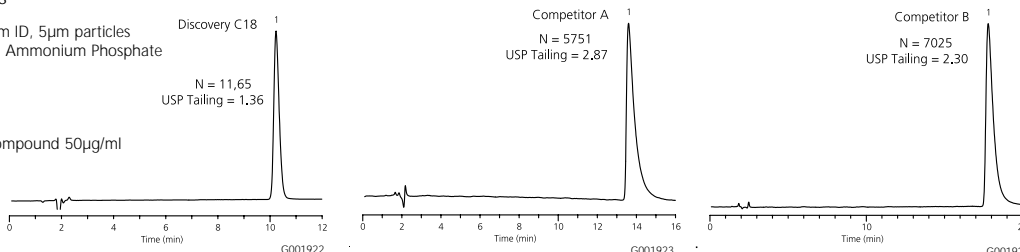
**Figure 2.** Phentermine

Columns: 15cm x 4.6mm ID, 5µm particles  
 Mobile Phase: 90:10, 25mM Ammonium Phosphate  
 (pH 7): CH<sub>3</sub>CN  
 Flow Rate: 1ml/min  
 Temp.: 30°C  
 Det.: UV at 210nm  
 Sample: 10µL, Phentermine, 50µg/ml

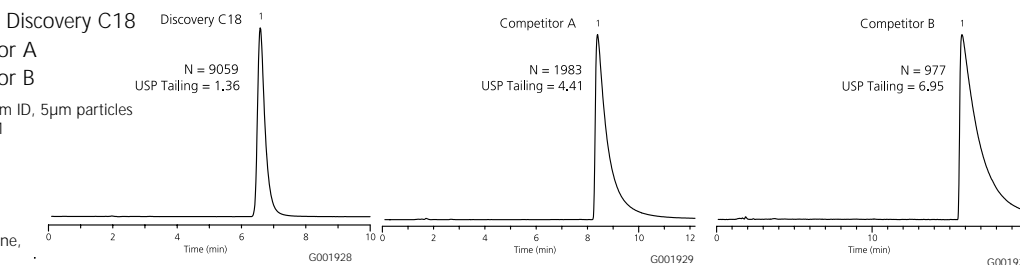
**Figure 3.** Fluoxetine

Columns: 15cm x 4.6mm ID, 5µm particles  
 Mobile Phase: 60:40, 25mM Ammonium Phosphate  
 (pH 7): CH<sub>3</sub>CN  
 Flow Rate: 1ml/min  
 Temp.: 30°C  
 Det.: UV at 227nm  
 Sample: 10µL, each compound 50µg/ml

1. Fluoxetine
2. Norfluoxetine

**Figure 4.** Quinidine Discovery C18  
Competitor A  
Competitor B

Columns: 15cm x 4.6mm ID, 5µm particles  
 Mobile Phase: 75:25, 25mM Ammonium Phosphate (pH 7): CH<sub>3</sub>CN  
 Flow Rate: 1ml/min  
 Temp.: 30°C  
 Det.: UV at 230nm  
 Sample: 10µL, Quinidine, 50µg/ml

**Ordering information****Discovery C18 5µm Particles**

Prod. No.	ID (mm)	Length (cm)
50494721	2.1	5.0
569220-U	2.1	10.0
569229-U	2.1	12.5
50495521	2.1	15.0
504947-30	3.0	5.0
569221-U	3.0	10.0
569230-U	3.0	12.5
504955-30	3.0	15.0
504971-30	3.0	25.0
504947-40	4.0	5.0
569222-U	4.0	10.0
569231-U	4.0	12.5
504955-40	4.0	15.0
504971-40	4.0	25.0
504947	4.6	5.0
569223-U	4.6	10.0
569232-U	4.6	12.5
504955	4.6	15.0
504971	4.6	25.0
569224-U	10.0	25.0
569226-U	21.2	25.0

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Liquid Chromatography/ Mass Spectrophotometry: Techniques and Applications Kluwer Academic 26506

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**i** Information Request ..... 1002

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### Ordering information Hamilton HPLC Syringes

Needle Specification							Replacement Needles
Capacity	Model	Gauge	Length	Point	Prod No.	Qty.	Prod No.
<b>700 Series Syringes For Rheodyne, Valco VISF-2, Altex and SSI Injection Valves</b>							
25µL	702SNR	22s	2"	#3	58381	-	--
50µL	705SNR	22s	2"	#3	58382	-	--
100µL	710SNR	22s	2"	#3	58383	-	--
250µL	725SNR	22	2"	#3	58384	-	--
500µL	750SNR	22	2"	#3	26222-U	-	--
<b>800 Series LC Syringes For Waters U6K Loop Injector</b>							
25µL	802RNW	25s	5cm	#3	58391	1	58398
50µL	805RNW	25s	5cm	#3	58392	1	58398
100µL	810RNW	25s	5cm	#3	58393	1	58398
250µL	825RNW	25s	5cm	#3	58394	1	58399
<b>1700 Series Gastight Syringes – For Rheodyne, Valco VISF-2, Altex and SSI Injection Valves</b>							
25µL	1702RNR	22s	2"	#3	20886	3	58649
50µL	1705RNR	22s	2"	#3	20887	3	58649
100µL	1710RNR	22s	2"	#3	20888	3	58649
250µL	1725RNR	22	2"	#3	20889	3	58650-U
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- PEEK and Stainless Steel
- HPLC and LC
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### Ordering information

Description	Prod. No.
<b>Preparative Sample Injectors</b>	
Model 3725i, PEEK	57461
Model 3725i-038, SS	57463
<b>Replacement Components</b>	
Rotor Seal	57473
Stator Face Assembly	57474
Needle, PEEK for model 3725	57475
Needle, 16-Gauge SS for model 3725	57476
Fitting Set, 1/8" (1 nut & 1 ferrule)	57477
RheFlex Stainless Steel	57479
Ferrules, 1/8" (pk. of 5) Stainless Steel	57480-U
RheBuild Kit for 3725/3725i/3725-038/3725i-038	55043
<b>Sample Loops 1,2</b>	
PEEK	
2ml	57464-U
5ml	57465
10ml	57466-U
20ml	57467
Stainless Steel	
2ml	57468-U
5ml	57469
10ml	57470-U
20ml	57471

### Ordering information

Description	Prod. No.
Model 7125 Injector	58826
<b>Replacement Components</b>	
VESPEL Rotor Seal	58825-U
Needle Port Cleaner	58831
Valve Angle Bracket (for all metal Rheodyne valves)	55044
RheBuild Kit for 7125/7126	
<b>Sample Loops with Fittings</b>	
5µL	58840-U
10µL	58832
20µL	58833-U
50µL	58834
100µL	58835
200µL	58836
500µL	58837
1ml	58838
2ml	58839
5ml	57637

Note: Use VESPEL seals to pH 10, Tefzel seals to pH 14.

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## SPE Article

# A Case Study in SPE Method Development- Understanding the Dual Interaction Properties of Discovery DSC-SCX SPE Using Verapamil (and Metabolite) from Serum as a Test Example

A strong cation-exchange (SCX) SPE method was developed to selectively recover verapamil and its major metabolite, norverapamil from porcine serum for subsequent HPLC analysis. Initial experimentation using standards revealed that neither an increase in eluant pH (to neutralize the basic amine functional groups exhibited by verapamil and norverapamil) nor organic strength alone were sufficient to elute analytes from the SCX phase. Instead, a combination of the two strategies was required to completely elute the analytes of interest. This information was invaluable towards developing a rugged wash system for removing unwanted endogenous sample interferences co-extracted onto the SCX phase.

The extraction method, coupled with the Discovery C18 HPLC column, offers a powerful analytical approach for the quick and accurate analysis of verapamil and norverapamil from serum.

### Principles of Ion-Exchange SPE

Ion-exchange SPE utilizes electrostatic interaction between the analyte and sorbent functional groups to retain charged molecules from a variety of sample matrices. In order for electrostatic retention to occur, both the analyte and sorbent functional groups must be oppositely charged. Strict control of pH is necessary to manipulate the ionization states of both the analyte and sorbent's acidic/basic functional groups thereby controlling selectivity (retention and elution) during the SPE process.

Although electrostatic interaction is the primary mode of retention in ion-exchange SPE, secondary reversed-phase or mixed-mode interactions may take place. This is especially true when organic analytes are introduced to the sorbent in the presence of an aqueous sample matrix.

### Sample Characteristics and Phase Selection

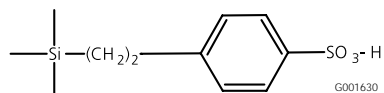
Verapamil is an anti-anginal/anti-arrhythmic calcium channel blocker used to treat cardiovascular disorders. Both verapamil and its major metabolite, norverapamil, carry secondary and tertiary amines, respectively making these basic compounds good candidates for cation-exchange (Figure A). Both compounds have a pKa of 8.6-8.9.

Discovery DSC-SCX (Figure B) is a polymerically bonded, benzene sulfonic acid functional group with an H<sup>+</sup> counter ion and exhibits a low pKa of <1. At virtually all pH levels, the SCX functional groups remained negatively charged.

As stated earlier, in order for electrostatic retention to occur, both sorbent and analyte functional groups must be oppositely

charged. Dropping the sample pH to at least 2 pH units below the analytes' pKa (pH = 6.6) should effectively ionize the analytes' amine functional groups.

Figure B. Structure of Discovery DSC-SCX

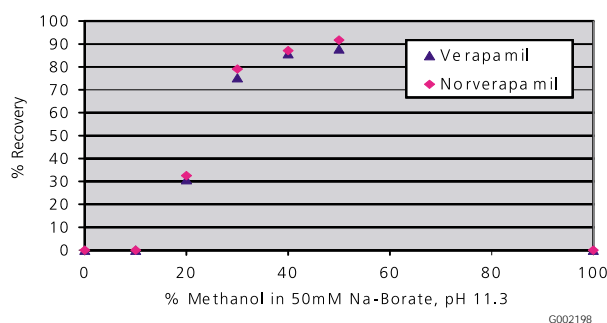


### Load Optimization & Wash/Elution Profile

In this study, 1ml working standards of both verapamil and norverapamil (5.0µg/ml) in 10mM ammonium formate, pH 3.1 were loaded onto Discovery DSC-SCX cartridges (100mg/1ml) previously conditioned and equilibrated with 1ml methanol and DI H<sub>2</sub>O. The load flow through eluate was then collected and analyzed via HPLC-UV. A lack of analyte presence in the load eluate indicated that adequate retention was observed under acidified aqueous conditions.

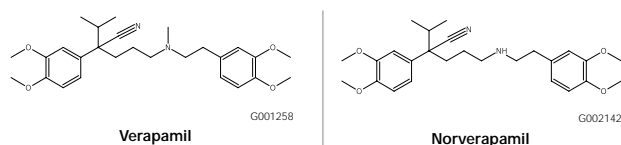
To determine application specific wash/elute parameters, Discovery DSC-SCX cartridges were conditioned and equilibrated with 1ml MeOH and DI H<sub>2</sub>O, and loaded with 1ml of the 0.5µg/ml verapamil/norverapamil low pH test mix. The respective tubes were washed/eluted with 1ml test solvents ranging from 0-100% methanol in 50mM sodium borate, pH 11.3. The wash/elute eluate was collected and analyzed via HPLC-UV. A graph relating organic strength to recovery was used to identify breakthrough (Figure C).

Figure C. Results of the Wash/Elute Profile for Verapamil and Norverapamil on Discovery DSC-SCX SPE



By profiling the major parameters affecting analyte retention and elution, application specific guidelines were established for defining, optimizing, and troubleshooting an extraction method. To elute verapamil and norverapamil from the SCX phase, disruption of the electrostatic interaction between SCX's sulfonic and the analytes' amino functional groups was necessary. By increasing the eluant pH to at least two pH units above the analytes' pKa, the compounds were effectively neutralized disrupting the ionic interaction retaining the compounds of interest. However, because of secondary hydrophobic interactions between the sorbent's phenyl group

Figure A. Structure of Verapamil and Norverapamil



and the analytes' carbon backbone, pH modification alone was not sufficient at eluting the analytes of interest. An organically modified buffer of appropriate pH was required for analyte elution (50% methanol in 50mM sodium borate, pH 11.3). Also learned from the wash/elute profile were two powerful wash steps. Both neat buffer and methanol can be used separately to wash off any co-retained hydrophilic and lipophilic interferences, respectively prior to analyte elution.

### Incorporation of Serum Sample Matrix

Porcine serum was incorporated into a method defined by data generated from precursory load optimization and wash/elute profile studies (Table 1).

Table 1.

#### Systematically Developed Method for Extracting Verapamil and Norverapamil from Serum Using Discovery DSC-SCX SPE (100mg/1ml)

1. Condition and equilibrate each cartridge with 1ml methanol and DI H<sub>2</sub>O (n=3)
2. Load 1ml 0.5µg/ml Verapamil and Norverapamil spiked porcine serum diluted in 10mM ammonium formate, pH 3.1 (1:1; v/v)
3. Wash with 1ml methanol
4. Wash with 1ml 50mM sodium borate, pH 11.3
5. Elute with 1ml 50% methanol in 50mM sodium borate, pH 11.3
6. Analyze eluate via HPLC-UV

### Simpler SPE Procedure, Cleaner Extracts, Reduced Analytical Run Times, & Excellent Recovery and Reproducibility

Figure D illustrates and compares chromatograms of both the SPE extract and external standards. Using the SPE method development approach, we were able to identify two powerful wash steps (neat methanol and buffer) to remove any co-retained lipophilic and hydrophobic interferences inherent with the serum sample matrix. As a result, the baseline was free of interfering background peaks that may have co-eluted with the two peaks of interest. Also, cleaner extracts allowed for shorter analytical run times (<4 min.). Unlike most reversed-phase protocols which require 100% organic elution that must be evaporated and reconstituted prior to HPLC analysis, a weaker solvent (50% methanol in 50mM sodium borate, pH 11.3) was

employed for final elution. This allowed for the direct injection/analysis of the final eluate reducing the number of extraction steps and minimizing overall processing time.

Average absolute recoveries and RSDs for both verapamil and norverapamil were  $102.9 \pm 1.2$  and  $95.9 \pm 2.3\%$ , respectively.

### Conclusion

When extracting ionized organic compounds from aqueous samples using ion-exchange SPE, retention rarely occurs exclusively via electrostatic interaction. Secondary hydrophobic interactions must be considered. Some researchers consider secondary or multiple interactions problematic often complicating the sample prep system. However, if studies are conducted to profile the major parameters affecting analyte retention and elution, highly selective SPE methods can be developed to specifically target and isolate analytes in very complicated/dirty sample matrices.

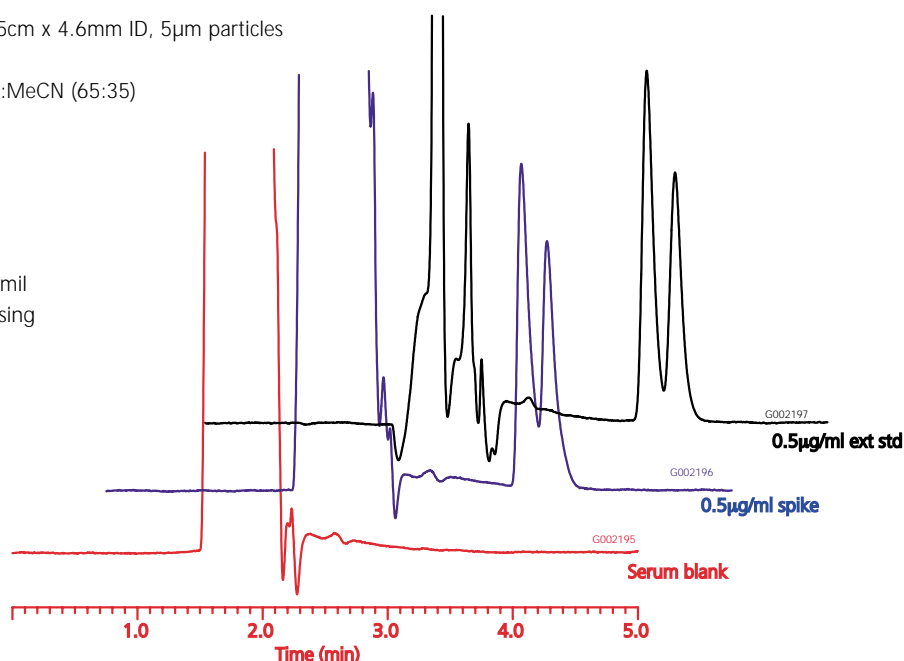
By profiling the affects of pH and % organic modifier, we were able to determine powerful wash conditions to remove both hydrophilic and lipophilic interferences. Cleaner extracts resulted in shorter analytical run times. Elution conditions using a more selective weaker solvent of appropriate pH allowed for direct injection thereby shortening overall processing time while maintaining high recoveries and low RSDs.

**i** Information Request .....1003

Figure D. Example Chromatograms of Extracts Generated from the Systematically Developed Method Using Discovery DSC-SCX SPE

Column: Discovery C18, 15cm x 4.6mm ID, 5µm particles  
 Cat. No.: 504955-U  
 Mobile Phase: 0.1% formic acid:MeCN (65:35)  
 Flow Rate: 1ml/min  
 Temperature: 35°C  
 Det.: UV, 267nm  
 Inj.: 10µL

Absolute Recovery of 0.5µg/ml Verapamil & Norverapamil from Porcine Serum Using Discovery DSC-SCX SPE (100mg/1ml)  
 Compound % Recovery ± RSD (n=3)  
 1. Verapamil 95.9 ± 2.3  
 2. Norverapamil 10.29 ± 1.2



## SPME Article

# Altitude Monitoring of Organic Pollutants by Solid Phase Micro Extraction (SPME)

By Elena Serena<sup>1</sup>, Gideon Czaczkes<sup>2</sup>, Nicola Perchiazzi<sup>3</sup> and Pietro Traldi<sup>1</sup>

<sup>1</sup> CNR-ISTM, Corso Stati Uniti 4, 35100 Padova (Italy) <sup>2</sup> Sky Light, via delle Palme 22/1, 53100 Padova (Italy) <sup>3</sup> Sigma-Aldrich s.r.l. Italia Via Gallarate 154, 20151 Milano (Italy)

In the last decade solid phase microextraction has proven to be a highly powerful tool for the analysis of volatile organic compounds (VOCs) present in the atmosphere<sup>(1)</sup>. This method is an extraction/concentration technique for pollutants in liquid or gaseous matrices, not requiring the use of any solvent<sup>(2)</sup>. It is based on the physico-chemical distribution principle of the analyte between two different phases:

- The solid one, present on the fused silica surface of the SPME device.
- The gaseous (or liquid) one in which the analyte(s) is dissolved.

Once the analyte(s) is concentrated on the SPME fiber, it is usually thermally desorbed inside a gas chromatographic (GC) injector, and analysed by GC/ECD or GC/MS systems. The SPME technique allows the use of fibres with solid phases of different thickness and polarities, to perform sampling selectively with respect to molecular weight, volatility and polarity of the analytes of interest. Usually the monitoring of volatile pollutants in air is carried out at ground level, as this is representative to which people are most exposed. However, considering that pollution at ground level is often the result of transport processes due to air stream from polluting sites, not necessarily close to the detection point, the monitoring at different altitudes, allowing to depict a three-dimensional map, is surely of high interest. In fact, it can be validly employed not only to describe the pollutants present in the site, but also to investigate origin and transport mechanisms.

To achieve this result, the most interesting approach (both from operative and economic points of view) is that based on the use of small-dimension, radio controlled, unmanned aircraft. Obviously these systems use d.c. electric motors, to avoid any contamination of the atmosphere under investigation. The collaboration between the Laboratory of Mass Spectrometry Researches of the CNR Institute of Molecular Science and Technologies (Padova, Italy) and the Sky Light Society (Padova) has led to the development of a system,



**Figure 1.**  
The radio controlled dirigible, d.c. electric motor driver, used for altitude monitoring

mounted on a small radio-controlled dirigible (see Figure 1), able to perform an altitude monitoring. To obtain an unequivocal description of the measurement point a GPS locator (Benefon GMS/GPS) and a Microsoft program (Mapoint 2002) are employed. This program is a powerful tool allowing the user to obtain homogeneous and personalized maps, linking the geographical description of the area with the parameters of interest. An example of the screen view is given in Figure 2.



**Figure 2.** Screen view of the Microsoft Mapoint 2002 software

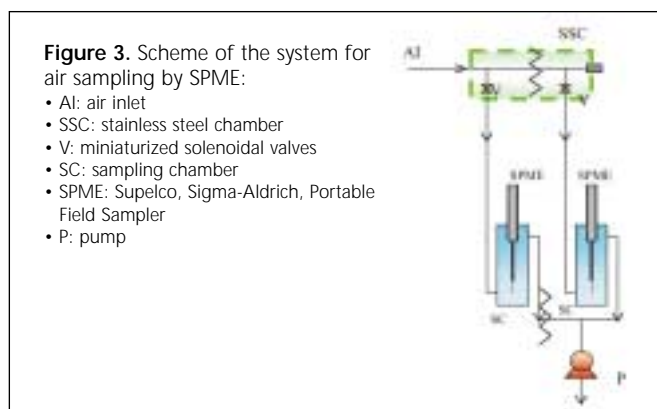
On the dirigible the following systems are mounted:

- CCD and/or Thermo IR camera;
- Microdust Pro (PM 10-PM 2.5)-(Casella cel);
- GPS, altimeter, barometer, thermometer;
- Sensors (CO<sub>x</sub>, O<sub>3</sub>, NO<sub>x</sub>)-(Microsens S.A.);
- Unit for VOCs analysis;

Using this systems it is possible to obtain the following data:

- Photos of the area of interest;
- Evaluation of dust dimension and concentration;
- Position, altitude, atmospheric pressure and temperature in the sampling point;
- CO<sub>x</sub>, O<sub>3</sub>, NO<sub>x</sub> concentrations;
- VOCs quali- and quantitative evaluation.

The unit for VOCs analysis was built around by the sampling



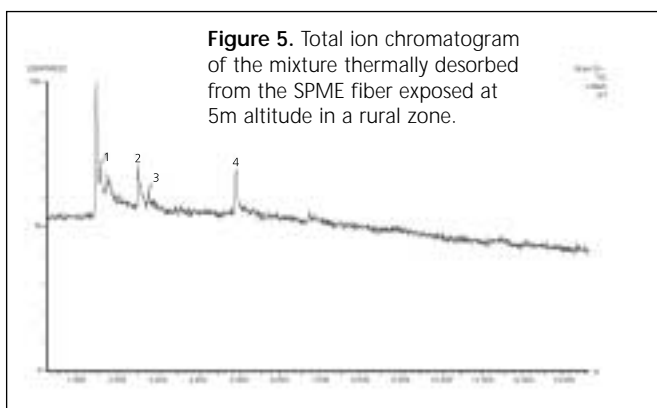
**Figure 4.**  
Photograph of the different components of the sampling system mounted inside the "gondola", placed on the bottom of dirigible.

device, based on the SPME Portable Field Sampler, schematized in Figure 3. It has been mounted inside a "gondola", mounted on the bottom of the dirigible (see Figure 4). A pump (Casella Cel, Bedford, UK, operating with flows in the range 0.8-11.5 L/min) is used to pump the air from outside. The air first reaches a stainless steel block, on which a series of miniaturized solenoidal valves (pneumax Veneto, Vicenza, Italy) are mounted. They can be radio operated and allow the air to be pumped into different chambers, in which SPME fibers are mounted (Supelco, Sigma-Aldrich, Portable Field Sampler, 65 mm Carboxen/PDMS).

The air volume interacting with the SPME fiber can be easily

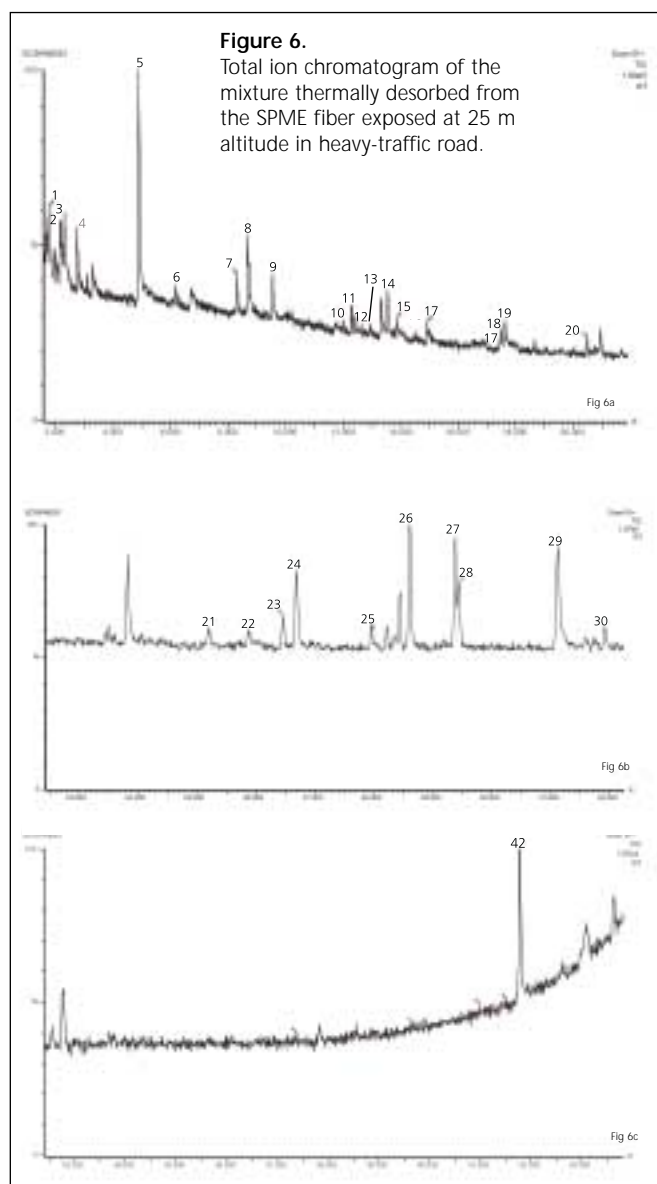
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calculated with respect to pumping time, as the flow is constant the pump operate in the constant flow conditions. The use of the system is particularly simple: the dirigible is placed at the altitude and position of interest, and air flow is directed into one of the SPME chambers for the desired time. Then the dirigible is moved to another position in which the second SPME chamber is employed, and so on. Once the dirigible has landed, the SPME fibers are retracted, to avoid their contact with a different environment to that of interest. They are brought to the lab, where GC/MS analyses are performed.



**Figure 5.** Total ion chromatogram of the mixture thermally desorbed from the SPME fiber exposed at 5m altitude in a rural zone.

Peak no.	R.T.	MW	Name	For	REV
1	1.766	58	Butane	515	861
2	2.166	58	Isobutane	456	878
3	2.316	60	Acetic acid ethyl ester	838	967
4	2.756	78	Benzene	826	969
6	4.989	92	Methyl-benzene	872	993
7	6.188	166	Tetrachloroethylene	504	878
9	8.319	106	ethyl-benzene	795	964
10	8.679	106	1,2-dimethyl-benzene	793	971
11	9.560	106	1,4-dimethyl-benzene	847	931
12	12.271	148	2,3,7-trimethyl-2,4,6-cycloheptatrienone	352	959
13	12.301	120	methyl,ethyl-benzene	684	971
14	12.411	120	2-methylethyl-benzene	542	890
15	12.941	120	methyl,ethyl-benzene	378	910
17	13.542	120	methyl,ethyl-benzene	596	976
18	13.882		Hydrocarbon	622	927
19	14.532	263	2-methyl-2-nitro-1-phenyl-1,5-heptandione	332	869
20	14.922	262	[R]-4-[5'-(3"-methyl-2"-butenyl)-2',6',6'-trimethyl-1'-cyclohexenyl]-3-buten-2-ol	636	939
21	17.463	86	2,2-dimethyl-butane	461	870
22	17.473	206	1,1-difluoro-dodecane	656	977
25	20.494		Hydrocarbon	638	986
29	25.198	216	Propanoic acid, 2-methyl-,2,2-dimethyl-1-(2-hydroxy-1-methylethyl)propyl ester	578	980
30	25.886	191	Propanoic acid, 2-methyl-,3-hydroxy-2,4,4-trimethylpentyl ester	619	987
31	26.456	270	bis(3,5,5-trimethylhexyl)ether	594	985
32	26.686		Hydrocarbon	899	981
33	27.967	194	Trans-6,10-dimethyl-5,9-undecadien-2-one	593	991
36	28.607	218	1-nonyl-benzene	772	969
37	29.377		Hydrocarbon	876	977
38	29.437	220	2,6-bis(1,1-dimethylethyl)-4-methyl-phenol	764	932
39	31.108	167	n-cyclohexyl-2-pyrrolidone	841	885
40	31.908		Hydrocarbon	652	930
42	41.769	228	4,4'-(1-methylethylidene)-bisphenol	537	848



**Figure 6.** Total ion chromatogram of the mixture thermally desorbed from the SPME fiber exposed at 25 m altitude in heavy-traffic road.

As an example, we report the data obtained by two different sampling cycles. The first has been performed at 50 m altitude, in a rural zone, not heavily polluted, being 50 m away from a light-traffic, country road. An air flow of 0.8 L/min was exposed 20 minutes to the SPME fiber (Supelco, Sigma-Aldrich, Portable Field Sampler 65 mm Carboxen/PDMS). The related total ion chromatogram is reported in Figure 5, in which the peaks due to acetaldehyde (peak 1), tetrahydrofuran (peak 2), benzene (peak 3) and toluene (peak 4) are detectable. The second sampling took place at an altitude of 25 m on a heavy-traffic road placed in the Padova industrial zone.

Air flow and sampling time were as before the same employed for the above described experiment (0.8 L/min for 20 min) but, as expected, in this case the total ion chromatogram (see Figure 6) shows a high number of pollutants. These have been identified by comparison with library data and are described in Table 1.

The data shown above confirms that SPME is a highly effective tool in air monitoring applications. The small size and the light weight of the portable SPME system make it particularly suitable for its use on small, radio aircrafts to be employed for air monitoring at different altitudes.

#### References

1. Augusto F, et al., Anal. Chem. 73, 481-486 (2001)
2. Pawliszyn J., Trends Anal. Chem. 14, 113-122 (1995)

# Supelclean:

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*For Further Information*

*Details of Supelclean products can be found in the main Supelco catalogue pages 157-159*

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KIT:	KIT RP	KIT NP	KIT IEX
<b>Prod. No.</b>	<b>57019</b>	<b>57074-U</b>	<b>57073</b>
Packing			
LC-Si	500mg/3ml	500mg/3ml	
LC-8	500mg/3ml		
LC-18	500mg/3ml		
LC-CN	500mg/3ml		500mg/3ml
LC-Diol	500mg/3ml	500mg/3ml	
LC-NH <sub>2</sub>	500mg/3ml	500mg/3ml	500mg/3ml
LC-Ph	500mg/3ml		500mg/3ml
LC-SAX	500mg/3ml		500mg/3ml
LC-SCX	500mg/3ml		500mg/3ml
LC-WCX	500mg/3ml		
LC-Alumina-A		1g/3ml	
LC-Alumina-B		1g/3ml	
LC-Alumina-N		1g/3ml	
Qty. Ea. Tube:	6	6	12

## Sigma-Aldrich TLC Plates

All silica plates are coated with 60Å pore size silica. Silica-based adsorbent layers incorporate a polymeric binder; highly purified silica gel plates incorporate a gypsum/polymer binder. Glass is the most widely used support material in TLC, but PET polyester plates offer an economical alternative to glass. PET plates are flexible, can be cut with scissors, and can be heated to 160°C to char spots. Aluminum foil-backed plates offer the advantages of polyester while tolerating even higher

temperatures. Concentrated mineral acids and ammonia should not be used with aluminum backed plates. Product Nos. Z292893 and Z292907 (highly purified silica gel on glass) were designed for analyses of aflatoxins. The special gypsum/polymer binder (mostly gypsum) used in these plates gives a softer layer than our other plates. This is ideal in applications that require removing individual spots by scraping. The gypsum binder also gives these plates slightly different selectivities from our other silica plates.

Ordering information						
Adsorbent Layer	Plate Dimensions (cm x cm)	Layer Thickness(µm)	Particle Size (µm)	Fluorescent Indicator	Qty.	Prod. No.
<b>BASIC ALUMINA ON PET POLYESTER</b>						
Aluminum oxide	20 x 20	200	<60	yes	25	Z234214-1PAK
Aluminum oxide	20 x 20	200	<60	no	25	Z234206-1PAK
<b>SILICA ON ALUMINUM FOIL</b>						
Silica gel	5 x 10	200	2-25	yes	50	Z193275-1PAK
Silica gel	10 x 20	200	2-25	yes	20	Z193283-1PAK
Silica gel	20 x 20	200	2-25	yes	25	Z193291-1PAK
<b>SILICA ON GLASS</b>						
Silica gel	5 x 20	250	5-17	no	100	Z122688-100EA
Silica gel	5 x 20	250	5-17	yes	100	Z122696-100EA
Silica gel	10 x 20	250	5-17	no	50	Z185310-50EA
Silica gel	10 x 20	250	5-17	yes	50	Z185329-50EA
Silica gel	20 x 20	250	5-17	no	25	Z122718-25EA
Silica gel	20 x 20	250	5-17	yes	25	Z122726-25EA
<b>SILICA ON PET POLYESTER</b>						
Silica gel	20 x 20	250	5-17	no	25	Z122777-25EA
Silica gel	20 x 20	250	5-17	yes	25	Z122785-25EA
<b>HIGHLY PURIFIED SILICA GEL, ACID WASHED, ON GLASS 2</b>						
Silica gel, highly purified	20 x 20	250	5-17	no	25	Z292893-1PAK
Silica gel, highly purified	20 x 20	250	5-17	yes	25	Z292907-1PAK

1 Manganese-activated zinc silicate. 2. Recommended for applications that require removing individual spots by scraping.

*For Further Information*

*Please refer to Supelco Catalogue pages 70-72 or to Sigma-Aldrich Laboratory Equipment Catalogue, pages 151-154*

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# GC Article

## Choosing the Proper Activated Alumina PLOT Column

The recent addition of Alumina sulfate and Alumina chloride columns to the Supelco family of GC capillary PLOT columns has prompted several customer inquiries on why we offer two alumina chemistries and how to choose between the two. We present the following information and applications to address these questions.

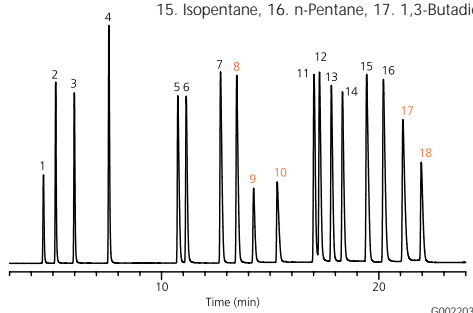
### Solution

Activated alumina PLOT columns are prepared using submicron, granular activated alumina (aluminum oxide) particles possessing a significant amount of mesopores. The use of sodium sulfate or potassium chloride as a desiccant and deactivating agent ensures that the activity of the alumina surface is effective in eluting (distilling) the unsaturated hydrocarbons after the saturated hydrocarbons. A small amount of micropores allows for the separation of methane from the C2 hydrocarbons, however surface chemistry plays the most significant role for these PLOT columns. The polarity of the aluminum oxide provides a unique selectivity, eluting/distilling small, unsaturated hydrocarbons after larger

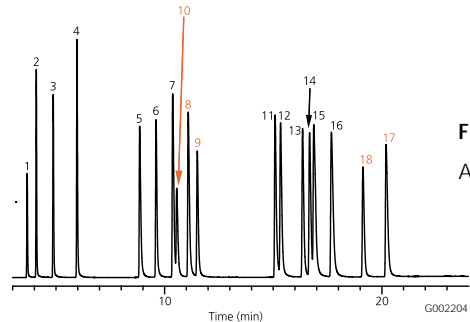
**Figure A.** 18 Component C1 to C5 Hydrocarbon Mix

Column 1: Alumina sulfate, 50m x 0.53mm ID  
 Cat. No.: 28324-U  
 Column 2: Alumina chloride, 50m x 0.53mm ID  
 Cat. No.: 28329-U  
 Oven: 35°C (2.5 minutes) to 150°C @ 5°C/minute (4.5 minutes)  
 Inj.: 200°C  
 Det.: FID, 230°C  
 Carrier Gas: Helium, 3.0 ml/minute  
 Injection: 10µL direct valve inlet  
 Valve: 150°C  
 Sample: Hydrocarbon mix in nitrogen (bulk) at 25ng on column

1. Methane
2. Ethane
3. Ethylene
4. Propane
5. Cyclopropane
6. Propylene
7. Isobutane
8. n-Butane
9. Propadiene
10. Acetylene
11. Trans-2-Butene
12. 1-Butene
13. Isobutylene
14. Cis-2-Butene
15. Isopentane
16. n-Pentane
17. 1,3-Butadiene
18. Propyne



**Figure A1.**  
Alumina sulfate



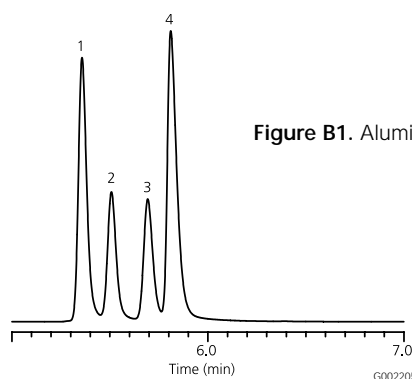
**Figure A2.**  
Alumina chloride

(or similar molecular sized) saturated hydrocarbons. This elution pattern is augmented with the alumina sulfate PLOT column, where acetylene elutes after n-butane, and methyl acetylene elutes after n-pentane and 1,3-butadiene.

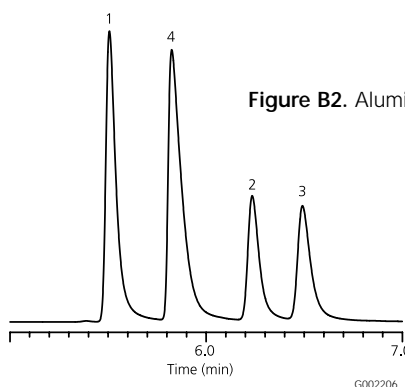
Use of the alumina chloride still provides these unsaturated/saturated elution profiles, but to a reduced

**Figure B.** Mineral Oil Gases

Column 1: Alumina sulfate, 50m x 0.53mm ID  
 Cat. No.: 28324-U  
 Column 2: Alumina chloride, 50m x 0.53mm ID  
 Cat. No.: 28329-U  
 Oven: 115°C (sulfate) / 95°C (chloride)  
 Inj.: 200°C  
 Det.: FID, 230°C  
 Carrier Gas: Helium, 3.0 ml/minute  
 Injection: 10.0µL direct valve inlet  
 Valve: 150°C  
 Sample: 1. 1-Butene (50ng on column) 2. Isobutylene (25ng on column)  
 3. Cis-2-Butene (25ng on column) 4. Neopentane (50ng on column)



**Figure B1.** Alumina sulfate



**Figure B2.** Alumina chloride

degree. For example, acetylene elutes after propane and propylene, but before n-butane (n-butane/acetylene is reversed for the alumina sulfate PLOT). The alumina chloride PLOTs are also the best choice for the analyses of Freons®. Examples of these elution patterns (i.e, the differences between the alumina sulfate and alumina chloride PLOTs) are illustrated in Figure A for 18 component analyses and Figures B for mineral oil gases.

### Ordering information

Alumina Chloride	Max. Temp.	Prod. No.
30m x 0.32mm ID	180°C	28326-U
50m x 0.32mm ID	180°C	28327-U
30m x 0.53mm ID	180°C	28328-U
50m x 0.53mm ID	180°C	28329-U

### Ordering information

Alumina Sulfate	Max. Temp.	Prod. No.
30m x 0.32mm ID	180°C	28321-U
50m x 0.32mm ID	180°C	28322-U
30m x 0.53mm ID	180°C	28323-U
50m x 0.53mm ID	180°C	28324-U

**i** Information Request .....1004

# Supelco GC Columns for Petrochemical Analyses



## Family of PLOT Capillary Columns

**NEW!** Alumina <sup>1005</sup>  
Improved durability reduces GC  
down time

Carboxen <sup>1006</sup>  
Designed for higher flow rates and rapid  
temperature programs resulting in faster  
analysis time

Mol Sieve 5A <sup>1007</sup>  
Patented adhesive eliminates particle loss,  
reducing noise spikes

Supel-Q <sup>1008</sup>  
True "Q" selectivity provides similar  
fingerprint to packed Q columns for  
method transfer

## Family of Petrocol Columns

Capillary columns for  
detailed hydrocarbon  
analyses  
Eliminates the need for unusual  
columns, special conditions, and  
prolonged analysis time

Capillary columns for  
simulated distillation  
Low column bleed for sharp  
return to baseline and more reliable  
boiling point data

## GPA Methodology Columns

**NEW!** Packed column sets for  
GPA methods 2261 and  
2177, and ASTM method 2597  
Highly reproducible for consistent  
method performance

Equity GC capillary columns  
for extended gas analysis for  
GPA methods 2186 and 2286  
Consistent resolution provides accurate  
identification and reliable quantitation

**i** Information Request.....1005, 1006, 1007, 1008

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## GC Article

# Fast GC Analysis Using the 0.10mm ID Equity-5

A current trend in gas chromatography has been decreased analysis times and increased sample throughput. Decreased run time can be achieved by decreasing column length, inner diameter (ID), stationary phase film thickness, increasing carrier gas linear velocity and oven ramp rate. This type of GC analysis, which is often termed "fast GC," utilizes narrow bore columns (<0.25mm ID) combined with thin films. Narrow bore columns offer a greater number of theoretical plates per meter than wider bore columns, and thus shorter lengths can be used while maintaining or improving the theoretical efficiency of the system. For example, comparing a 0.25mm ID column to a 0.10mm ID column with similar retention ( $k'$ ) and coating efficiency, the approximate plate numbers per meter are 2925 and 7300 respectively. For this reason, 0.10mm ID columns are a good choice for fast GC analysis.

Comparing 0.10mm ID with 0.25mm ID columns, the Van Deemter plots (plate height,  $H$  vs. linear velocity,  $\mu$ ) of 0.10mm ID columns have a higher  $\mu_{opt}$  and a more shallow increase in  $H$  with increasing  $\mu$  than 0.25mm ID columns. Consequently, a higher linear velocity can usually be used on these columns to shorten analysis time, without significantly affecting resolution. However, these columns require higher head pressures than 0.25mm ID columns to establish the same linear velocity. This limits the length of column that is practical for use in a conventional GC system. Helium, the most commonly used carrier gas, requires a head pressure of 55psi to achieve a linear velocity of 30cm/sec at 100°C on a 15m x 0.10mm ID x 0.10 $\mu$ m column. In comparison, the same length column with a 0.25mm ID requires only a 7.8psi head pressure to achieve the same linear velocity.

Hydrogen, an alternative carrier gas choice, has a lower viscosity than helium and will not require as high of a head pressure to achieve the same linear velocity. A 15m x 0.10mm ID x 0.10 $\mu$ m column run with hydrogen carrier would only require a head pressure of 25psi to achieve 30cm/sec at 100°C.

for hydrogen achieved at a higher average gas velocity than helium. The result is that using hydrogen carrier at a linear velocity near or just above its  $\mu_{opt}$  will result in a faster run time without a significant loss in resolution.

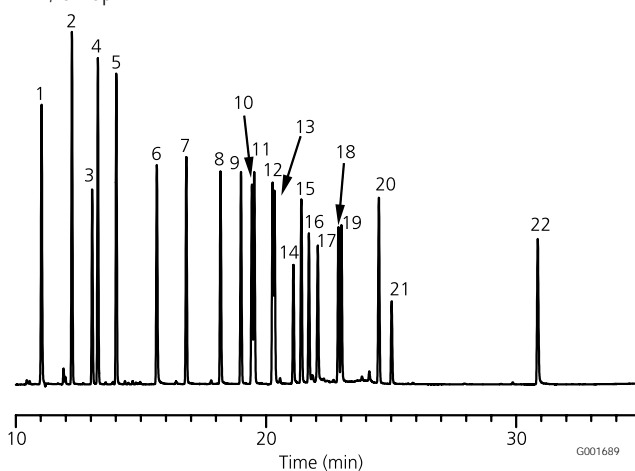
Fast oven ramp rates are also essential in decreasing run time, and all hold times during the temperature program should be as short as possible. The various GC systems available have different capabilities with regards to oven ramping. Before developing a fast GC method, it is advisable to check with the manufacturer of your GC to find out the temperature ramping capability of your system.

In this work, we compared the use of a 30m x 0.25mm ID x 0.25 $\mu$ m to a 15m x 0.10mm ID x 0.10 $\mu$ m Equity-5 for two common applications, organochlorine pesticides and PCBs. The run conditions established using the 0.10mm ID Equity-5 reduced the run time by 75%. Figure A illustrates the separation of 20 organochlorine pesticides and 2 surrogates on the 30m x 0.25mm ID x 0.25 $\mu$ m Equity-5. Figure B illustrates the same compound list on a 15m x 0.10mm ID x 0.10 $\mu$ m Equity-5 column. The total run time decreased from 32 to 6.5 minutes. Hydrogen carrier in constant flow mode and rapid oven ramp rates with no hold at the initial oven temperature were necessary to decrease the run time to this level. A 4mm ID liner was used to accommodate the expansion volume of the 2 $\mu$ L injection. One coelution was noted, endosulfan I and a-chlordane. Labs typically do this application as a dual column analysis with a second column of different selectivity. Since this secondary column would probably resolve this pair, the trade-off in resolution in this case could be acceptable for a 75% decrease in run time. After converting a conventional method to a "fast" method, the elution order of the analytes should always be verified. In this case, there was a change in elution order between endosulfan sulfate/DDT and endrin ketone/methoxychlor.

**Figure A.** Organochlorine Pesticides on the Equity-5, 30m x 0.25mm ID, 0.25 $\mu$ m

Column: Equity-5, 30m x 0.25mm ID, 0.25 $\mu$ m  
Cat. No.: 28089-U  
Oven: 100°C (2 min), 15°C/min to 160°C, 5°C/min to 300°C (10 min)  
Inj.: 225°C  
Det.: ECD, 310°C  
Carrier Gas: Helium, 30cm/sec @ 100°C  
Injection: 2 $\mu$ L, splitless (0.5 min)  
Liner: 4mm ID double taper  
Sample: 50ppb of a 22 component chlorinated pesticide standard (Cat. No. 46845-U)

Compounds:	1. 2,4,5,6-Tetrachloro-m-xylene (surr.)	12. 4,4'-DDE
	2. a-BHC	13. Dieldrin
	3. b-BHC	14. Endrin
	4. g-BHC	15. Endosulfan II
	5. d-BHC	16. 4,4'-DDD
	6. Heptachlor	17. Endrin aldehyde
	7. Aldrin	18. Endosulfan sulfate
	8. Heptachlor epoxide	19. 4,4'-DDT
	9. g-BHC	20. Endrin ketone
	10. Endosulfan I	21. Methoxychlor
	11. a-Chlordane	22. Decachlorobiphenyl (surr.)



Another benefit of hydrogen carrier gas over helium is its higher diffusivity. The Golay theory for open tubular columns predicts that optimum gas velocity is proportional to diffusivity. This means that the optimum linear velocity for hydrogen will be higher than helium. In addition, the Van Deemter plot for hydrogen is flatter than that of helium, with the minimum  $H$

In the case of the PCB analysis, the time savings were similar to the pesticides. A chromatogram of a mixture of Aroclors® 1016 and 1260 on a 30m x 0.25mm ID x 0.25 $\mu$ m Equity-5 column is presented in Figure C. Analysis times of 38-45 minutes are not uncommon for this application. If the same mix is rerun on a 15m x 0.10mm ID x 0.10 $\mu$ m Equity-5 with the

same run conditions used for the pesticide analysis (Figure D), the analysis can be done in under 8 minutes. Even with the significantly reduced run time; resolution is sufficient to provide excellent pattern recognition of both Aroclors.

We have seen here that reducing column length and ID, increasing oven ramp rates and using hydrogen as a carrier gas can significantly reduce GC run time. The 15m x 0.10mm ID x 0.10µm Equity-5 can be used for such a purpose. If you are in need of increasing GC sample throughput in your laboratory, consider letting us help you convert your current method to a faster version.

For further assistance, please contact technical service at [uktechsv@europe.sial.com](mailto:uktechsv@europe.sial.com) or call 0800 424342 / 814-359-3041.

Ordering information	
Description	Prod. No.
<b>Narrow Bore GC Equity 5</b> 30m x 0,25mm ID x 0,25µm df	28089-U
<b>Fast GC Equity 5</b> 15m x 0,10mm ID x 0,10µm df	28083-U
<b>Other phases available in Fast GC dimension are:</b>	
Equity 1, Equity 5, Equity 1701, TCEP, Supelcowax 10, Omegawax, SP2560	
Please contact our local Representatives and Specialists for any information on Fast GC.	

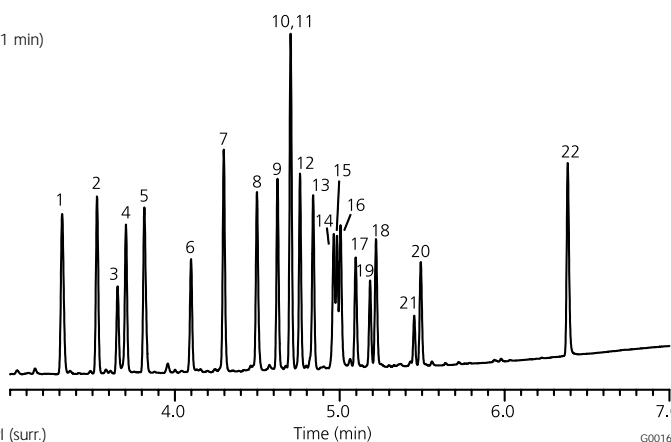
**i** Information Request.....1009

**Figure B.** Organochlorine Pesticides on the Equity-5, 15m x 0.10mm ID, 0.10µm

Column: Equity-5, 15m x 0.10mm ID, 0.10µm  
 Cat. No.: 28083-U  
 Oven: 100°C (0 min), 50°C/min to 200°C (0 min), 35°C/min to 360°C (1 min)  
 Inj.: 225°C  
 Det.: ECD, 360°C  
 Carrier Gas: Hydrogen, 30cm/sec constant  
 Injection: 2µL, splitless (0.75 min)  
 Liner: 4mm ID single taper  
 Sample: 50ppb of a 22 component chlorinated pesticide standard (Cat. No. 46845-U)

Compounds:

1. 2,4,5,6-Tetrachloro-m-xylene (surr.)	12. 4,4'-DDE
2. a-BHC	13. Dieldrin
3. b-BHC	14. Endrin
4. g-BHC	15. Endosulfan II
5. d-BHC	16. 4,4'-DDD
6. Heptachlor	17. Endrin aldehyde
7. Aldrin	18. Endosulfan sulfate
8. Heptachlor epoxide	19. 4,4'-DDT
9. g-Chlordane	20. Endrin ketone
10. Endosulfan I	21. Methoxychlor
11. a-Chlordane	22. Decachlorobiphenyl (surr.)

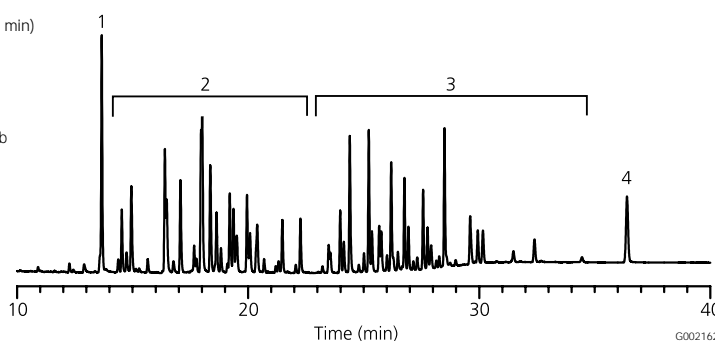


**Figure C.** Aroclors 1016 and 1260 on the Equity-5, 30m x 0.25mm ID, 0.25µm

Column: Equity-5, 30m x 0.25mm ID, 0.25µm  
 Cat. No.: 28089-U  
 Oven: 100°C (2 min), 15°C/min to 160°C, 5°C/min to 300°C (10 min)  
 Inj.: 225°C  
 Det.: ECD, 310°C  
 Carrier Gas: Helium, 30cm/sec @ 100°C  
 Injection: 2.0µL, splitless (0.5 min)  
 Liner: 4mm ID double taper  
 Sample: Aroclor Mix 1 standard at 75ppb with surrogates at 7.5ppb (Cat. No. 46846-U)

Compounds:

- 2,4,5,6-Tetrachloro-m-xylene (surr.)
- Aroclor 1016
- Aroclor 1260
- Decachlorobiphenyl (surr.)

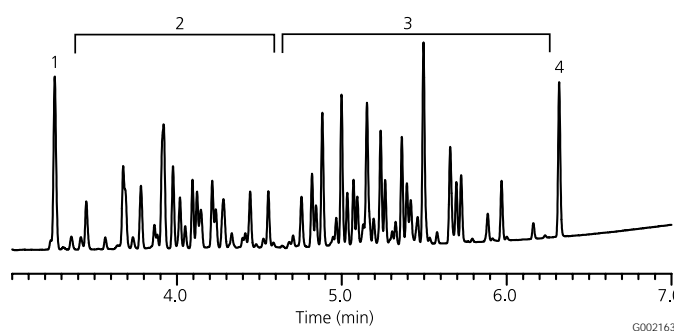


**Figure D.** Aroclors 1016 and 1260 on the Equity-5, 15m x 0.10mm ID, 0.10µm

Column: Equity-5, 30m x 0.25mm ID, 0.25µm  
 Cat. No.: 28083-U  
 Oven: 100°C (0 min), 50°C/min to 200°C (0 min), 35°C/min to 360°C (1 min)  
 Inj.: 225°C  
 Det.: ECD, 360°C  
 Carrier Gas: Hydrogen, 30cm/sec constant  
 Injection: 2µL, splitless (0.75 min)  
 Liner: 4mm ID single taper  
 Sample: 200ppb of Aroclors 1016 & 1260 with surrogates at 20ppb (Cat. No. 46846-U)

Compounds:

- 2,4,5,6-Tetrachloro-m-xylene (surr.)
- Aroclor 1016
- Aroclor 1260
- Decachlorobiphenyl (surr.)



## GC Article

# Conditioning Molecular Sieve Columns Removes Trapped Moisture, Improves Chromatography

Analysis of permanent gases is routinely performed using Molecular Sieve 5A or 13X packed, micropacked or PLOT columns. The separation of gases is achieved because of the interaction of the gases with pores present in the molecular sieve material. The pores also trap water vapor from the carrier gas or injected with samples. As water begins to occupy the internal pore volume of the packing, the retention times of gases such as oxygen, nitrogen, methane and carbon dioxide will decrease. The ability of the column to separate these compounds will also decrease.

Conditioning of the column at elevated temperature removes the water and will often restore column performance. The amount of conditioning required is dependent on the amount of water that is present on the column and the degree of separation required. The following chromatograms compare the effects of two different durations of conditioning at elevated temperature.

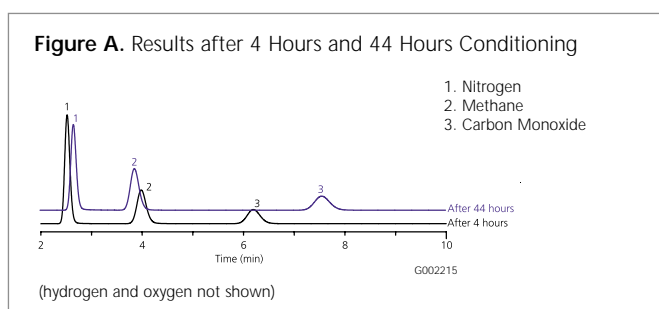


Figure A shows the analysis of a helium sample containing 1% v/v of nitrogen, methane and carbon monoxide on a 12 ft x 1/8" SS column packed with 45/60 mesh Molecular Sieve 5A. The packing material had become saturated with water and the gases were eluting too quickly and were inadequately separated. The analyst baked the column at 250°C for four hours in an attempt to restore the original performance of the column. This significantly increased the separation of the compounds, but did not restore the original chromatography (black line). The column received an additional 40 hours of conditioning at 250°C. The extended conditioning returned the column to its original performance (blue line).

**Table 1. Relative Retention Times**

Reconditioning Time	Nitrogen	Methane	CO
0 hrs ('wet' mole sieve packing)	2.48	4.22	5.37
After 4 hrs @ 250°C	2.73	4.33	6.73
After 44 hrs @ 250°C	3.24	4.71	9.22

Another way to look at the change is to divide the retention times of the analytes by that of hydrogen (peak not shown in Figure 1), a relatively unretained peak, yielding the relative retention times of the analytes. The changes in relative retention times (see Table 1) due to the presence of water in the Molecular Sieve 5A become very clear.

While either of the chromatograms shown in Figure A could be acceptable, it is up to the analyst to determine the extent of conditioning needed to produce the chromatography required to meet the analytical objective.

## GC Article

# Why do I Need High Purity Solvents for Environmental Analysis?

Most of the organic pollutants to be determined in environmental analysis are extracted from matrices like water or soil samples by means of solvents. The composition of the analytes is quantitatively analysed after a concentration step. In order to get meaningful results it is essential that for the used solvents extremely low limits referring to the substances to be analysed are guaranteed.

The PESTANAL® solvents were developed especially for the application in residue analysis of pesticides and other low-volatile, environmentally relevant substances by means of GC/ECD or GC/PND. As polychlorinated biphenyls (PCBs) are also detected in the GC/ECD test, the PESTANAL® solvents are suitable for analysis of this class of substances as well.

Besides a general high grade of purity the specifications are tailor-made to the special requirements in residue analysis of pesticides, metabolites, preservatives and other low-volatile, environmentally relevant substances. Therefore, the specification refers mainly to the corresponding retention ranges for GC/ECD and GC/PND analyses.

Moreover, besides their suitability for residue analysis by means of GC/ECD and GC/PND, parameters as assay, non-volatile matter and water content are specified for each solvent.

As these values might vary depending on the solvent, they are indicated in the product range.

The high purity solvents are produced in a plant using cutting-edge technology. They are produced in large, homogenous lots involving several purification steps. Filling occurs under clean-room conditions in contaminant-free, functional packaging materials.

In addition to the 1 l and 2.5 l glass bottles the solvents of the PESTANAL® line are also available in returnable packagings as the 7 l stainless steel bottle and the 18 l and 45 l stainless steel barrels.

**Table 1. Specifications**

Method	GC/ECD	GC/PND
Retention range	Pentachlorobenzene to DDT	4-Chloraniline to Coumaphos
single signals	max. 5 pg/ml	max. 5 pg/ml
Standards	Pentachlorobenzene, Lindan, Heptachlor, Aldrin, 4,44-DDD, 4,44-DDT	4-Chloraniline, Parathionethyl, Altrazin, Coumaphos

*For Further Information*

<http://www.sigma-aldrich.com/pestanal>

# New!

## SGT Super Clean™ Gas Filter

Supelco introduces a new range of "point-of-use" gas purifiers, the Super-Clean-Gas Filters from SGT.

Figure C. Shipping & Flush base of He-specific triple filter



General picture SGT Purifier



In GC and GC/MS the gas quality has a significant influence on the analytical performance of the system. A purer and/or more reliable gas supply leads to longer life time of the analytical column, less column bleed, decreases baseline noise, increases sensitivity, and eliminates spikes. Gas purifiers ensure the reliable supply of pure gas and also offers the possibility to use initially less pure and therefore less expensive gas. The SGT glass/metal, diffusion proof cartridge system filters purify delicate carrier, fuel or compressor & other gases for your GC & GC/MS system from hydrocarbons, moisture and oxygen up to better than 6.0 gas (99.9999%) quality, independent from the original gas quality. The systems have been extensively tested with a GC-MS system by the R.I.C. - Research Institute for Chromatography, Belgium (Prof. P. Sandra, Dr. F. David).

### SGT Super-Clean-Gas filters provide the following benefits:

- High Flexibility - Tailor-made combinations of a variety of one-purpose and/or multipurpose filters (1-4 position baseplates for various demands and applications).
- Easy and fast exchange of filters - Change (possible during instrument operation) within seconds "fast-connect" diffusion-proof design. (see Figure A)
- Visual saturation indication of Oxygen, Moisture and Hydrocarbons.
- No plastic housing - Out-coming gas guaranteed 6.0 grade or higher. Completely inert and diffusion-proof.
- TUV approved safety device - Pressure resistant up-to 150psi/11bar. (German-)
- 100% Compatible with Agilent's Super Clean™ Gas Filter Systems.

### Super-Clean Triple filter

The newest product of the SGT line is the Helium specific Super-Clean™ Triple filter. This is a "3-in-1" filter containing adsorber beds to trap hydrocarbons, moisture (indicated) and oxygen (indicated). The especially treated and He pre-conditioned adsorbents allow fast operation after cartridge

exchange (4x faster than inline filter). The enclosed special shipping & flush base (Figure C) allows to precondition the cartridge with other gases (e.g. Ar, H<sub>2</sub> etc.).

Bottomline: Just 1 Filter is needed to purify the GC/MS gas from Hydrocarbons, Oxygen and Moisture. This saves time & money and guarantees best analytical results.

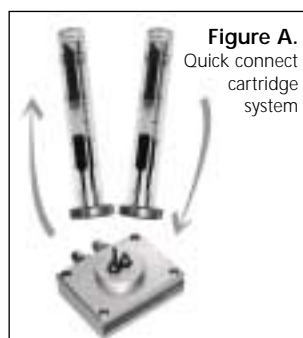


Figure A. Quick connect cartridge system



Figure B. SGT Triple filter kit (Cartridge & Base plate)

### Ordering information

Description	Prod. No.
<b>Helium specific version</b>	
GC-MS Kit for He - gas specific (Baseplate + 1 triple filter)	SU861040
Replacement Triple GC-Filter for Helium (Oxygen/Moisture/Charcoal), 1ea.	SU861027
<b>Non Helium specific version</b>	
GC-MS,ECD,FID,NPD-carrier gas Kit (Baseplate + 1 triple filter)	SU861041
Replacement Triple GC-Filter (Oxygen/Moisture/Charcoal), 1ea.	SU861026

Other available ready to use Super-Clean-Gas Filter systems are:  
GC-FID Kit 4 cartridges (ultra capacity O<sub>2</sub>, moisture 2x charcoal)  
GC-FID Kit 3 cartridges (triple + 2x combi cartridges charcoal/moisture )  
LC/MS kit 2 cartridges (2x charcoal for N<sub>2</sub> purification) High flow capacity, no indicator

**i** Information Request.....1010

## NEW! Improved Performance from Packed GC Columns Used for the Analysis of Natural Gas and Natural Gas Liquids In Reference to GPA and ASTM Methods

Packed GC columns continue to be used in valved-GC configurations for the determination of BTU content in natural gas and natural gas liquids as prescribed in Gas Processors

Association Methods 2261, 2177, and the American Society of Testing and Materials (ASTM) Test Method 2597. Column sets used for these analyses must meet performance based criteria cited in the methods. This poster presents data demonstrating how optimized column manufacturing procedures produce column sets that provide highly reproducible analyses that exceed GPA and ASTM method requirements.

**i** Information Request.....1011

# PRIMUS - A project of



## New Certified Standards for Demanding Ion Chromatography applications

### Introduction

Today, Ion Chromatography is an important technique within the analytical laboratory used for the aqueous determination of ions down to the ppt range. It is the only technique that can provide quantitative analysis of ions at this level. Moreover, the development of modern IC apparatus and columns makes this technique a fast, uncomplicated and reliable tool!

The quality of IC standards has needed to improve to keep up with advances in the available hardware. The selection of appropriate standards for the calibration of the IC system is important, and Fluka already offers a wide range of IC standards from single element for anion and cation analytic to multielement standards. These standards are frequently used by the environmental, food and beverage industries.

Some industries, e.g. Pharma or Semiconductor, may need to work with even higher qualifications and certified standards to comply with GMP. This trend is furthermore reinforced by official regulations of the European community within the guideline 96/23/EG, that regulates the use of certified reference materials (CRM)<sup>1</sup>. The American FDA (Food and Drug Administration) demands in their inspection Guide for analytic methods the usage of reference standards. And ISO (International Organization for Standardization) published more than 6 papers about the topic reference materials.

CRMs guarantee the validation standard and means that they have to be traceable to a SI unit and standard deviation has to be determined. In accordance to ISO Guide 17025<sup>2</sup>, the preparation of a CRM can only be carried out by metrological institutes, such as NIST (National Institute of Standards and Technology), EMPA (Swiss Federal Laboratories for Materials Testing and research) or BAM in Germany (Federal Institute for Materials Research and Testing).

### Single- and Multielement Standards developed in collaboration with EMPA and BAM

For Ion Chromatography the number of CRM's is small. Fluka fills this gap and a few months ago launched the first CRM's for Ion Chromatography (Table 1). The development was carried out in collaboration with EMPA, located in St. Gallen, Switzerland. Based on the competence of this highly regarded metrological institute the traceability to SI is guaranteed and standard deviation is determined. Therefore standards are produced completely at EMPA and give you the guarantee of certification you desire.

Following a positive customer response to these standards we were also asked to produce certified multielement standards in Ion Chromatography. Today we are pleased to offer you two new certified multielement standard solutions, **PRIMUS** (Primary Multiions Standards for Ion Chromatography) (Table 2).

Table 1. Anionic standard solutions for ion chromatography (1000 mg/kg)

Prod. No.	Brand	Product	Produced from	Medium	Pack Size
87603	Fluka	Chloride	NaCl	H <sub>2</sub> O	100 ml
80218	Fluka	Sulfate	Na <sub>2</sub> SO <sub>4</sub>	H <sub>2</sub> O	100 ml
87969	Fluka	Bromide	NaBr	H <sub>2</sub> O	100 ml
86576	Fluka	Nitrate	NaNO <sub>3</sub>	H <sub>2</sub> O	100 ml
80373	Fluka	Fluoride	NaF	H <sub>2</sub> O	100 ml
81193	Fluka	Phosphate	NaH <sub>2</sub> PO <sub>4</sub>	H <sub>2</sub> O	100 ml

### High precision weighing and high purity Salts for highest quality

EMPA determine the purity of the starting materials based on high purity salts (Table 2). The content of these salts is determined by three methods for each salt and is done in collaboration with BAM in Germany. The results are verified and cross-checked at state-of-the-art levels to ensure the quality you require.



The really novel part is the production procedure: First the solid salts are determined and then they are dissolved in high purity water. Although it sounds simple, one of the core competences of EMPA is the extremely precise and accurate weighing of these salts. This allows us to offer our customers standards at a very low content of ion species in these CRMs (10mg/kg for each ion).

To ensure the quality of our product: Each bottle is delivered in a sealed mylar bag to avoid loss of water and to keep the concentration stable over more than two years. All standards are delivered with a certificate of analysis, including the detailed methods for quantitative analyses and standard deviation.

Table 2. Multielement standard solutions for ion chromatography (10 mg/kg)

Prod. No.	Brand	Product	Ions	Produced from	Medium	Pack Size
89316	Fluka	certified multication standard solution	Lithium, Sodium, Potassium, Magnesium, Calcium	Li <sub>2</sub> CO <sub>3</sub> , NaCl, KCl, MgO, CaCO <sub>3</sub>	H <sub>2</sub> O, HCl	50 ml
89886	Fluka	certified multication standard solution	Fluoride, Chloride, Bromide, Nitrate, Phosphate, Sulfate	NaF, NaCl, NaBr, NaNO <sub>3</sub> , Na <sub>2</sub> HPO <sub>4</sub> , Na <sub>2</sub> SO <sub>4</sub>	H <sub>2</sub> O	50 ml

References

- (1) Commission decision of 12 August 2002 implementing Council Directive 96/23/EC concerning the performance of analytical methods and the interpretation of results, Official Journal of the European Communities, L 221/8
- (2) ISO Guides: 30 (1992) Terms and definitions used in connection with reference materials; 31 (2000): Reference materials – contents of certificates and labels; 32 (1997): Calibration in analytical chemistry and use of certified reference materials; 33 (2000): Uses of certified reference materials; 34 (2000): General requirements for the competence of reference material producers; 35 (1989): Certification of reference materials – General statistical principles
- (3) 3.1.1.2. Trueness.  
In this paragraph, the determination of trueness (one component of accuracy) is described. Trueness can only be established by means of certified reference material (CRM). A CRM be used whenever available.

## Did you know that ...

each new Metrohm IC apparatus contains a pack of our multication and multianion standards for validation. Metrohm highly recommends the use of our standards for the internal calibration of their instruments.

**i** Information Request .....1012

advanced

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- The low bleed you expect for lower detection limits
- The column life you count on for increased productivity

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Capillary GC Columns in our new brochure.

### Ordering information

#### Equity-1 Capillary GC Columns

Phase: bonded; poly(dimethylsiloxane)

Temp. Limits: 0.25 and 0.32mm ID: -60°C to 325/350°C  
0.53mm ID: -60°C to 300/320°C (<=1.5µm Df)  
-60°C to 260/280°C (>1.5µm Df)

Prod. No.	Length (m)	D <sub>f</sub> (µm)
<b>0.20mm ID</b>		
28041-U	12	0.33
28042-U	25	0.33
28043-U	10	1.2
<b>0.25mm ID</b>		
28044-U	30	0.10
28045-U	15	0.25
28046-U	30	0.25
28047-U	60	0.25
28048-U	15	1.0
28049-U	30	1.0
28050-U	60	1.0
28052-U	100	1.0
<b>0.32mm ID</b>		
28053-U	30	0.10
28054-U	15	0.25
28055-U	30	0.25
28056-U	60	0.25
28057-U	30	1.0
28058-U	60	1.0
28060-U	100	1.0
28061-U	30	2.0
28062-U	30	5.0
28063-U	60	5.0
<b>0.53mm ID</b>		
28064-U	15	0.10
28065-U	30	0.10
28067-U	15	0.5
28068-U	30	0.5
28069-U	15	1.0
28071-U	30	1.0
28072-U	15	1.5
28073-U	30	1.5
28074-U	60	1.5

### Ordering information

#### Equity-5 Capillary GC Columns

Phase: bonded; poly(5% diphenyl/95% dimethylsiloxane)

Temp. Limits: 0.25 and 0.32mm ID: -60°C to 325/350°C  
0.53mm ID: -60°C to 300/320°C (<=1.5µm Df)  
-60°C to 260/280°C (>1.5µm Df)

Prod. No.	Length (m)	D <sub>f</sub> (µm)
28075-U	15	3.0
28076-U	30	3.0
28077-U	60	3.0
28079-U	15	5.0
28081-U	30	5.0
28082-U	60	5.0
<b>Equity-1701 Capillary GC Columns</b>		
Phase: bonded; poly(14% cyanopropylphenyl/86% dimethylsiloxane)		
Temp. Limits: 0.25 and 0.32mm ID: subambient to 280°C 0.53mm ID: subambient to 260°C		
Prod. No.	Length (m)	D <sub>f</sub> (µm)
<b>0.10mm ID</b>		
28083-U	15	0.10
<b>0.20mm ID</b>		
28084-U	15	0.20
28085-U	30	0.20
28086-U	60	0.20
28087-U	12	0.33
<b>0.25mm ID</b>		
28088-U	15	0.25
28089-U	30	0.25
28090-U	60	0.25
28092-U	30	0.5
28093-U	15	1.0
28094-U	30	1.0
28095-U	60	1.0
<b>0.32mm ID</b>		
28096-U	15	0.25
28097-U	30	0.25
28098-U	60	0.25
28099-U	30	0.32
28195-U	30	0.5
28199-U	30	1.0
28251-U	60	1.0
<b>0.53mm ID</b>		
28252-U	15	0.5

# OFFER

**Free** Buy a column and get a pack of 5 liners of your choice from the Supelco catalogue (page 261-266) for free. (Offer limited to one column per customer)

Promotional code: F83 Offer valid until 15th April 2004

### Ordering information

Prod. No.	Length (m)	D <sub>f</sub> (µm)
28259-U	30	0.5
28263-U	60	0.5
28264-U	30	1.0
28265-U	15	1.5
28267-U	30	1.5
28268-U	30	3.0
28269-U	60	3.0
28278-U	15	5.0
28279-U	30	5.0
28293-U	60	5.0

#### Equity-1701 Capillary GC Columns

Phase: bonded; poly(14% cyanopropylphenyl/86% dimethylsiloxane)

Temp. Limits: 0.25 and 0.32mm ID: subambient to 280°C  
0.53mm ID: subambient to 260°C

Prod. No.	Length (m)	D <sub>f</sub> (µm)
<b>0.25mm ID</b>		
28371-U	15	0.25
28372-U	30	0.25
28373-U	60	0.25
28374-U	15	1.0
28378-U	30	1.0
28379-U	60	1.0
<b>0.32mm ID</b>		
28381-U	15	0.25
28382-U	30	0.25
28384-U	60	0.25
28386-U	15	1.0
28387-U	30	1.0
28388-U	60	1.0
<b>0.53mm ID</b>		
28389-U	15	0.5
28391-U	30	0.5
28393-U	15	1.0
28394-U	30	1.0
28395-U	15	1.5
28396-U	30	1.5

**i** Information Request.....1013

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