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The Reporter

THE TECHNICAL NEWSLETTER FROM SUPELCO

Mass Spectral Column Bleed in Nitrogen-Containing Polar-Embedded HPLC Stationary Phases

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Introduction

Liquid chromatography-mass spectrometry (LC-MS) has become one of the most important tools in the analytical chemistry laboratory over the past decade. The structural information obtainable along with sensitive detection has made the technique indispensable in pharmaceutical, environmental and a wealth of other scientific disciplines.

HPLC column bleed is a major source of background signal in LC-MS analyses. This phase bleed occurs when the bonded phase elutes from the column during the analysis. The bleed may originate from acid hydrolysis of the bonded phase at low mobile phase pH values or from dissolution of the silica substrate under more basic conditions. Most column manufacturers recommend using silica-based columns between pH 2 and 7.5, however, hydrolysis of C18 phase

may occur using mobile phases at pH 2-3 and slow dissolution of the silica substrates occurs in the commonly used pH range.

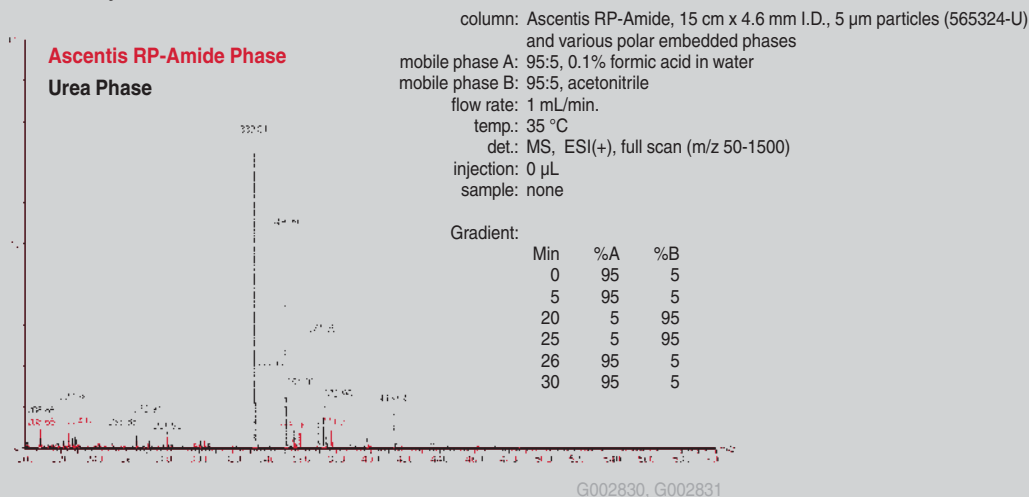
In this report, we examine the extent of stationary phase bleed for several commercially available, nitrogen-containing polar-embedded phases. The study utilizes a low pH mobile phase to induce hydrolysis of the bonded phase along with gradient elution to ensure elution of any liberated phase material. Prior to analysis, each of the columns used in this study were exhaustively washed and stored in 50:50 methanol: water for two days. This procedure reduces the probability of observing bleed from sources other than that due to stationary phase instability.

Experimental

Bleed analyses were performed on a Waters® (Milford, MA USA) 2795 HPLC system coupled to a Waters Micromass ZQ mass spectrometer via

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Figure A. Accumulated Mass Spectra for Ascentis™ RP-Amide and Urea Polar Embedded Stationary Phases



Liquid Chromatography

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an electrospray interface. The interface was operated in positive ion mode and an m/z range of 50 to 1500 was acquired. Acetonitrile and water (Riedel-de Haën®, Seelze, Germany) were of LC-MS grade. Formic acid obtained from the same supplier was of HPLC grade. Prior to each column bleed analysis, a blank run using no column in line was acquired to establish responses due to system impurities. Each column was subsequently subjected to five gradient cycles. HPLC conditions are shown in Figure A.

Results and Discussion

The information-rich LC-MS experiments are difficult to present in their entirety. For brevity, the combined mass spectra for each run were obtained by accumulating the spectra over the time range of 14-16 minutes. It is in this time frame where the greatest concentration of mass responses due to phase bleed is observed. In addition, the spectral responses in this report are presented in the range of m/z 200-600. Presentation of the data in this range avoids complications due to abundant mass responses at low m/z ranges originating from system contamination.

The mass spectrum obtained using the Ascentis RP-Amide stationary phase is compared to the urea polar-embedded phase response in Figure A. The Ascentis RP-Amide spectrum demonstrates a limited number of mass responses due to column bleed in this region.

Table 1 lists the significant mass responses that were not observed in blank runs for the Ascentis RP-Amide column and several commercially available amide, carbamate, and urea polar-embedded phases. The Ascentis RP-Amide phase exhibits three mass responses that can be attributed to stationary phase bleed (m/z 355, 359 and 377). The analysis from a commercially available urea polar-embedded phase shows numerous intense mass responses in the investigated region, indicating substantial bleed. For the carbamate phase, the mass responses are significantly

lower in intensity than the urea column; however, there are additional mass responses that may be attributed to phase bleed. This increases the probability of analyte interference by phase bleed that may lead to increased difficulty in spectral interpretation. A second commercially available amide phase exhibits the same number of mass responses as the Ascentis RP-Amide, however, there is a significant increase in response intensity resulting in higher background.

Conclusions

In this study we have examined LC-MS bleed characteristics of nitrogen-containing polar-embedded stationary phases. The results demonstrate that the Ascentis RP-Amide phase exhibits fewer and less intense mass spectral bleed responses when compared to other commercially available polar-embedded columns. Low stationary phase bleed results in improved detection of trace impurities, facilitates mass spectral interpretation, and minimizes downtime due to source contamination.



Related Information

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3	2.1	10	565301-U
3	2.1	15	565302-U
5	2.1	5	565303-U
5	2.1	10	565304-U
5	2.1	15	565305-U
5	2.1	25	565306-U

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Table 1. Mass Responses and Intensities Attributed to Stationary Phase Bleed

Column	Intensity at Mass to Charge Response (m/z)					
Ascentis RP-Amide (m/z)	355	359	377			
Intensity (cps)	6.14E+05	5.79E+05	7.25E+05			
Urea Phase (m/z)	228	271	332	350	372	413
Intensity (cps)	1.66E+06	1.19E+06	1.15E+07	8.07E+06	4.17E+06	1.61E+06
Carbamate Phase (m/z)	249	273	291	355	369	397
Intensity (cps)	1.74E+06	2.18E+06	1.38E+06	1.25E+06	1.35E+06	1.37E+06
Amide Phase (m/z)	355	369	397			
Intensity (cps)	1.32E+06	1.30E+06	1.52E+06			

Carbon Adsorbent Kits for Sample Prep & Method Development

Daniel Vitkuske
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Carbon based adsorbents exhibiting a broad range of adsorption strengths and surface areas are becoming increasingly popular with analytical chemists and research scientists performing thermal/solvent desorption and purge and trap applications for environmental monitoring. Newly developed methodology for solid phase extraction (SPE) using carbon adsorbents has also shown effective performance for liquid sample preparation (1,2,3). Because there are a wide variety of carbon based adsorbents and choosing the proper adsorbent to use for an application is critical to the success of an analysis, we have developed three carbon adsorbent kits along with an *Adsorbent Selection Guide* (see Related Information) to aid researchers with the development of air sampling traps or tubes. Two of the kits are composed of graphitized carbon blacks-Carbotrap™ and Carbopack™ and the third kit contains carbon molecular sieves-Carboxen™ and Carbosieve™.

The Carbotrap Kit consists of five (20/40 mesh) graphitized carbon black adsorbents while the Carbopack Kit consists of six (60/80 mesh) graphitized carbon black adsorbents with a range of surface areas and adsorption strengths. Selecting an adsorbent of the proper surface area and desorption strength

is critical to efficiently trapping and releasing the compounds of interest. Carbotrap and Carbopack are high purity graphitized carbon blacks that are exclusive to Supelco. A surface area range from 5 – 240 m²/g for the graphitized carbons included in the kits will allow a broad range of adsorbent strength options. In addition, the graphitized carbon selection of Carbopack F, C, Y, and B are nonporous adsorbents while Carbopack Z and X have some porosity and offer increased adsorption strength. Of particular interest is Carbopack X, a newly developed graphitized carbon that displays an extended analyte retention response when compared to Carbopack B. For example, the recovery of 1,3-Butadiene from Carbopack X is excellent even at large sample volumes, which in turn extends the method detection limits. Finally, all of the graphitized carbons presented in these kits are hydrophobic and are good choices when sampling in an environment where high humidity exists or when extracting organics from aqueous environments.

The Carbon Molecular Sieve Kit contains Carboxen and Carbosieve adsorbents. Both high purity spherical polymer carbons are exclusive to Supelco. Table 1 shows the physical properties of the adsorbents for each kit. Most of the Carboxen

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Table 1. Physical Characteristics of Supelco Carbon Adsorbents

	BET surface area [▲] , m ² /g	Density, g/mL	Porosity, cc/g			Micropore Diameter, Å
			micro-	meso-	macro-	
Carbotrap Kit (20/40 mesh graphitized carbon black)						
Carbotrap F	5	0.69	-	-	-	-
Carbotrap C	10	0.68	-	-	-	-
Carbotrap Y	24	0.45	-	-	-	-
Carbotrap B	100	0.37	-	-	-	-
Carbotrap X	240	0.43	-	0.62	-	100
Carbopack Kit (60/80 mesh graphitized carbon black)						
Carbopack F	5	0.64	-	-	-	-
Carbopack C	10	0.68	-	-	-	-
Carbopack Y	24	0.42	-	-	-	-
Carbopack B	100	0.35	-	-	-	-
Carbopack Z	220	0.18	-	1.73	-	255
Carbopack X	240	0.41	-	0.62	-	100
Carbon Molecular Sieve Kit						
Carboxen-1016	75	0.40	-	0.34	-	-
Carboxen-569	485	0.58	0.20	0.14	0.10	5-8
Carboxen-1021 [▼]	600	0.62	0.30	-	-	5-8
Carboxen-1018 [▼]	675	0.60	0.35	-	-	6-8
Carbosieve S-III [□]	975	0.61	0.35	0.04	-	4-11
Carboxen-1003	1000	0.46	0.38	0.26	0.28	5-8
Carbosieve G	1160	-	0.49	0.02	-	6-15
Carboxen-1000	1200	0.48	0.44	0.16	0.25	10-12
Carboxen-1012	1500	0.50	-	0.66	-	19-21

[▲] Brunauer, Emmett, Teller (BET) surface area calculations

[▼] microporous, monoporos carbon sieve

[□] closed pore structure

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GC

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Figure A. Supelco Carbon Molecular Sieve Sample Kit



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(continued from page 3)

grades, with the exception of Carboxen-1018 and 1021, are multi-porous adsorbents designed specifically to efficiently retain and release only analytes with low boiling points. The Carbosieve adsorbents have a unique pore structure in combination with a high surface area and are very strong adsorbents. Both Carboxen and Carbosieve may require having a bed of weaker adsorbent, such as Carbopack, placed in front, to prevent analytes with high boiling points from reaching the pores of the molecular sieve adsorbent during sampling. In addition, within this kit we offer our newest adsorbent, Carboxen-1016, a graphitized polymer carbon. This carbon molecular sieve adsorbent demonstrates excellent performance across both a wide range of analytes and sample volumes.

The proper selection of the adsorbent components of adsorptive multi-bed traps is critical to the efficient retention and release of targeted analytes within the sample matrix. The Carbon Adsorbent Kits were developed to provide the analyst the largest practical number of adsorbent bed combinations in order to ensure nearly complete analyte coverage. The combination of the new Carbon Adsorbent Kits and Supelco's practical *Adsorption Selection Guide* allows the researcher to increase productivity by reducing the time necessary to develop and customize new multi-bed tubes for air and liquid sample preparation applications.

References

- Care and Use Manual for Supelco Multi-Layer Silica Gel Column and Dual-Layer Carbon Reversible Column, Supelco Data Sheet T70218 (2002).
- Y. Kemmochi, K. Tsutsumi, A. Arikawa, H. Nakazawa, J. Chromatogr. A, 977 (2002) 155-161.
- M. Concejero, L. Ramos, B. Jimenez, B. Gomara, E. Abad, J. Rivera, M.J. Gonzalez, J. Chromatogr. A, 917 (2001) 227-237.

Carbon Adsorbent Kits

Description	Cat. No.
Carbopack Kit (60/80 mesh graphitized carbon black)	13026-U
Carbotrap Kit (20/40 mesh graphitized carbon black)	13027-U
Carbon Molecular Sieve Kit	13028-U



Related Information

For more information on adsorbents, request *Characterization of Adsorbents for Sample Preparation Process*, T402026 (EQG) and *A Tool for Selecting an Adsorbent for Thermal Desorption Applications*, T402025 (HKZ).

Did you know...?

The inlet liner used for a capillary split/splitless injection is critical to the proper transfer, vaporization and movement of the injected sample through the liner. New FocusLinners™ are the recommended inlet liners for improving the reproducibility of a capillary analysis. The key to the improved reproducibility provided by these liners is the proper positioning of the glass wool in the liner and due to the positioning, the ability to wipe the end of the syringe needle during the injection.



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Description	Qty.	Cat. No.
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Empty fritted tube, 6 mm O.D. x 4.5", 4 mm I.D.	3	20380-U
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VOST Stack Sample Tube		
Empty VOST tube, 16 mm O.D. x 5" (1/4" O.D. ends)	1	21993
Thermal Desorption Tubes for PerkinElmer Instruments		
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Empty SS tubes with TMX brass storage caps, 1/4" O.D. x 3.5", 5 mm I.D.	50	28015-U
Empty glass tube without caps, 1/4" O.D. x 3.5", 4 mm I.D.	1	25084
Empty glass tubes with TMX brass storage caps, 1/4" O.D. x 3.5", 4 mm I.D.	50	28013-U
Thermal Desorption Tubes for Tekmar®-Dohrmann Instruments		
Empty SS tube, 1/4" O.D. x 7" long, 5 mm I.D.	1	20920-U
Empty SS tube, 1/2" O.D. x 7" long, 12 mm I.D.	1	20924
Empty glass tube, 1/4" O.D. x 7" long, 4 mm I.D.	1	20918
Empty glass tube, 1/2" O.D. x 3.5" long, 10 mm I.D.	1	20922
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Empty SS non-fritted tubes	1	28276-U
Empty glass fritted tubes	1	28286-U
Empty glass non-fritted tubes	1	28287-U

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Vicki Yearick

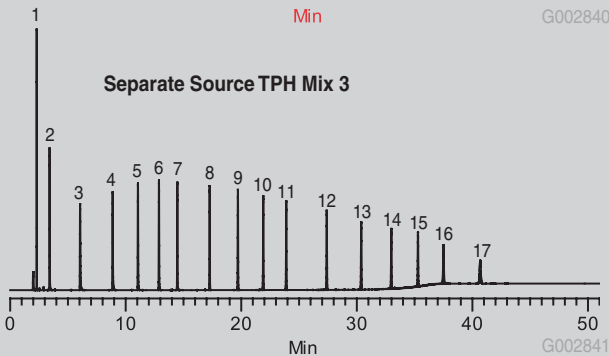
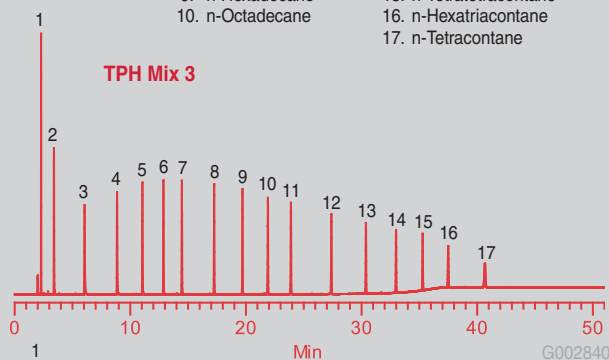
vyearick@sial.com

Supelco is pleased to introduce 300 new analytical standards for environmental analyses. These new formulations resulted from working closely with key customers to develop environmental standards that provide better solutions for popular, everyday analyses. The new formulations include volatiles, pesticides, herbicides, semi-volatiles, Aroclors®, and hydrocarbons and are appropriate for use with current US EPA 500, 600, and 8000 series, and CLP methodologies. They are being offered in an assortment of neat, single component and multi-component solutions, and kits. Separate Source™ standards for several mixes will also be available.

Figure A. Separate Source Total Petroleum Hydrocarbon (TPH) Standards

sample: TPH Mix 3, 1000 µg/mL each in CS₂ (861394-U)
 sample: Separate Source TPH Mix 3,
 1000 µg/mL each in CS₂ (8S61394-U)
 column: Equity-5, 60 m x 0.53 mm I.D., 0.5 µm (28263-U)
 oven: 40 °C (5 min.) to 350 °C (20 min. hold) @ 10 °C/min.
 inj.: 250 °C
 det.: 360 °C
 carrier gas: helium
 injection: 1.0 µL, direct on-column

- | | | |
|-------------|------------------|-------------------------|
| 1. Hexane | 5. n-Decane | 11. n-Eicosane |
| 2. Heptane | 6. n-Undecane | 12. n-Tetracosane |
| 3. n-Octane | 7. n-Dodecane | 13. n-Octacosane |
| 4. n-Nonane | 8. n-Tetradecane | 14. n-Dotriacontane |
| | 9. n-Hexadecane | 15. n-Tetratetracontane |
| | 10. n-Octadecane | 16. n-Hexatriacontane |
| | | 17. n-Tetracontane |



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P000392

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Glass Magnet Sheet	57269

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P000126

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2 mL amber glass	100	27064
2 mL clear glass w/markings spot	100	27062-U
2 mL amber glass w/markings spot	100	27066-U
Shell Style Inserts		
0.35 mL clear glass, 6 x 31 mm	100	24715
Conical Style Inserts		
0.25 mL glass, 6 x 31 mm	100	24717
0.15 mL glass w/top spring, 6 x 29 mm	100	24719
0.20 mL glass w/bottom spring, 6 x 29 mm	100	24721
0.20 mL polypropylene w/bottom spring, 6 x 29 mm	100	24722

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Improved SPME Fiber Life and Reproducibility with All Metal Fiber Assemblies

Robert Shirey

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Solid Phase Microextraction (SPME) has grown significantly over the last 10 years. The CTC CombiPAL™ is a valuable tool for high volume sample analysis using SPME. However, the sample agitator on the CombiPAL, which increases sample adsorption efficiency, can significantly stress the fiber, leading to fiber damage and shorter fiber life.

A new SPME fiber assembly has been developed that contains a special metal alloy in the needle, plunger, and fiber core. This metal alloy adds significantly greater strength resulting in 7 to 10 times longer fiber assembly life. The new material also allows us to improve the fiber manufacturing process, resulting in better inter and intra-lot reproducibility leading to greater overall reproducibility in analytical results. While some customers may be concerned that certain analytes might break down by contact with metal in a hot injection port, we have demonstrated that this new metal alloy is equally inert when compared to existing fused silica and StableFlex materials.

The increased assembly life results from a fiber assembly made of individual components such as the plunger, needle and fiber core that utilize this special metal alloy. In a side-by-side evaluation under the same conditions, the metal assemblies were able to perform a minimum of 350 extraction/desorption cycles without breaking compared to 30-40 cycles for the silica based fibers with stainless steel assemblies.

Using the metal fiber as the core material also allowed us to optimize the fiber coating process. The new continuous process controls many variables. Carboxen-PDMS fibers have been particularly difficult to coat reproducibly due to many bonding variables. Table 1 shows the comparison of lots of Carboxen-PDMS fibers prepared with the old and new coating process. As Table 1 shows, the relative standard deviation of the assemblies made by the new process have less than half the variation as compared to those made by the older process.

Another significant advantage of the metal alloy is that it is an inert metal that does not contain any iron. None of the components in the assembly exposed to the heated injection port contain iron. Thus analytes that may break down by contact with metal in a hot injection port are not an issue with this assembly. It is generally understood that certain amines can react with metals and break down. To verify the inertness of

Table 1. Improved Reproducibility of All Metal Fiber Assemblies

	Ethane	Propane	Butane	Pentane	Hexane
Old Process (n=8)					
Avg. Response	125	1699	6462	12371	16224
Std. Deviation	20	601.1	1777.9	2045.1	2243.2
% RSD	16%	35%	28%	17%	14%
New Process (n=6)					
Avg. Response	202	3375	11083	16233	19294
Std. Deviation	16.1	328.3	513.4	789.7	974.7
% RSD	8%	10%	5%	5%	5%

the new fiber, a sample containing amines was extracted with the various fiber core types. The results of our evaluation indicate that the response using the fibers with metal alloy cores were similar to those with the fused silica and StableFlex cores.

Table 2. Relative Response of Amines on 3 Fiber Core Materials

Fiber Core	Methylamine		Dimethylamine		Diethylamine	
	Ratio	% RSD	Ratio	% RSD	Ratio	% RSD
Metal	1.14	5.8	4.85	4.7	38.12	3.4
Fused Silica	1.02	6.6	4.21	4.4	38.67	2.9
StableFlex	1.04	6.7	5.43	6.2	37.49	3.6

The merging of a new fiber assembly with a metal fiber core and an improved coating process has created a superior SPME fiber assembly. Customers should not only enjoy longer assembly life, but better reproducibility between fibers.



Related Information - SPME Vials and Closures

While the new assembly can puncture 3 mm thick vial septa, we highly recommend using vial seals designed for Solid Phase Microextraction. SPME vial seals have thinner 1.5 or 1.6 mm septa and special cap designs to obtain a secure seal. Our evaluations have determined that butyl rubber vial closures can never be recommended for use with any SPME assembly with automation. For a detailed summary of the recommended vials and closures for SPME, contact Supelco Technical Service at 800-359-3041 / 814-359-3041, or email techservice@sial.com

Did you know...?

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SPME

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POSTER SESSIONS - located in the southeast corner of the convention center.

Time	Title, Author
Monday, Feb. 28, 2005	
Morning	<i>New Carbo-pack X Adsorbent for 24-Hour Diffusive Sampling of 1,3-Butadiene</i> , W. Betz
Morning	<i>New Portable SPME Passive Air Monitoring and Field Sampling Device</i> , R. Shirey
Morning	<i>A Tool for Selecting an Adsorbent for Thermal Desorption Applications</i> , J. Brown
Morning	<i>Fast Gas Chromatography, Guidelines and Applications</i> , L. Sidisky
Morning	<i>Reversed-Phase Retention of Glycopeptides and Detection by Mass Spectrometry</i> , H. Brandes
Afternoon	<i>Pharmaceutical Metabolite Analysis Using 100% Aqueous-Compatible HPLC Stationary Phases</i> , C. Santasania
Afternoon	<i>Decreasing the Analysis Time of Pharmaceutical Solvents with SPME</i> , K. Stenerson

Tuesday, Mar. 1, 2005	
Morning	<i>Starting Points in Chromatographic Method Development: A Structure-Based Approach</i> , D. Bell
Morning	<i>Molecular Interactions Contributing to Alternative Selectivity of Polar-Embedded HPLC Stationary Phases</i> , D. Bell
Morning	<i>Designed Selectivity Enhancement</i> , W. Campbell
Morning	<i>The Development of a New Resilient SPME Fiber Assembly Containing Coated Metal Fibers</i> , R. Shirey
Morning	<i>Novel Polyethylene Glycol Coatings for LC and GC Solid Phase Microextraction</i> , C. Linton
Afternoon	<i>The Importance of Ion-Exchange Capacity in Mixed-Mode SPE Technology for Pharmaceutical Bioanalysis</i> , A. Trinh

Time	Title, Author
Tuesday, Mar. 1, 2005 (contd.)	
Afternoon	<i>Enhanced Selectivity from Mixed-Mode SPE Technology for Pharmaceutical Bioanalysis</i> , D. Bell
Afternoon	<i>Flash Chromatography Method Development</i> , M. Ye
Afternoon	<i>Purification of Products of Organic Synthesis Using Flash Chromatography</i> , M. Ye

Wednesday, Mar. 2, 2005	
Morning	<i>Analysis of Multi-Pesticide Residues in Vegetables, Food and Fruits by SPE/GC-MS</i> , M. Ye
Morning	<i>Apply SPE and LC-MS to Determine Acrylamide in Potato Chips</i> , C. Aurand

ORAL SESSIONS

Time	Location	Title, Author
Tuesday, Mar. 1, 2005		
8:50 AM	S220C	<i>FAME Analysis Using High Speed GC</i> , L. Sidisky
1:30 PM	S220D	<i>Molecular Interactions Contributing to Aqueous Normal Phase Retention of Basic Analytes on Fluorinated and Other Polar HPLC Stationary Phases</i> , D. Bell
2:30 PM	S220C	<i>Zirconia: The Ideal Substrate for Ion-Exchange LC and LC-MS</i> , R. Henry

Trademarks

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Patent

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