

the Reporter

EUROPE

Volume 16, May 2005 International issue

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EDITORIAL

Supelco Carbon Adsorbents on Cassini-Huygens Mission to Saturn

Dear Reader,

Recent developments in the aerospace industry demonstrate conclusively that Supelco will go to the ends of the universe to provide innovative solutions to difficult analytical problems.

On the 15th of October, 1997 the Cassini-Huygens spacecraft lifted off from Launch Complex 40 at Cape Canaveral on Florida's Atlantic coast. While eager engineers all over the world followed Cassini-Huygens trajectory and safe ascent through the atmosphere, eager chemists at Supelco knew it would be years until the fruits of their labors would be put to the test. On board that spacecraft were two of Supelco's innovative Carboxen™ carbonaceous adsorbents, called into service by NASA and European Space Agency (ESA) scientists to help collect and analyze the atmosphere on Saturn and Saturn's moon, Titan. In January 2005, Cassini-Huygens began sending data back to Earth.

One of the Carboxens™, a carbon molecular sieve, is used in the enrichment cell of an Ion and Neutral Mass Spectrometer (INMS) onboard the Cassini orbiter. Gaseous components in Saturn's atmosphere, including elements, isotopes and molecules, are collected, concentrated and separated by the Carboxen™. The separated components are then sent to the INMS for identification. Supelco researchers prepared this carbon for the University of Paris, where scientists were studying and testing the INMS system for the ESA. One example of the data supplied by this instrument is the ratio of hydrogen isotopes, important information in the scientific debate over the Big-Bang theory.

The other Carboxen™, a graphitized polymeric carbon, is onboard the Huygens probe that landed on Titan on January 14th. During its two and one-half hour descent and for ninety minutes after it landed on the surface, the probe transmitted data to the Cassini orbiter. This Carboxen™ is also contained in an enrichment cell, this time in a GC/MS instrument, where it is

used for collecting, concentrating and separating light hydrocarbons, such as methane and acetylene. Scientists believe that the Titan atmosphere is composed of hydrocarbons and data provided by this instrument will further their understanding. Supelco researchers prepared this Carboxen™ for scientists at the NASA-Goddard Space Flight Center in Greenbelt, Maryland.

The vast majority of our chromatography products are used by analysts to solve problems right here on Earth; environmental remediation, treatments for disease, forensic investigations, air and water monitoring and measuring the safety of our food supply, to name just a few. However, with the launch of the Cassini-Huygens Mission on the 15th of October 1997, our products are now firmly entrenched in solving problems and answering questions beyond Earth.

To learn more about the Cassini-Huygens Mission, see <http://saturn.jpl.nasa.gov>. To find out how Carboxens™ or any of our innovative products can help you with your analytical challenges, please call us.



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European Sales Development Manager
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HPLC ARTICLE

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

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Introduction

Liquid chromatography-mass spectrometry 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 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 (LC-MS CHROMASOLV®, Fluka/Riedel-de Haën) 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.

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

Column: Ascentis RP-Amide, 15 cm x 4.6 mm I.D., 5 µm particles (565324-U) and various polar embedded phases
 mobile phase A: 95:5, 0.1% formic acid in water
 mobile phase B: 95:5, acetonitrile
 flow rate: 1 ml/min.
 temp.: 35 °C
 det.: MS, ESI(+), full scan (m/z 50-1500)
 injection: 0 µL
 sample: none

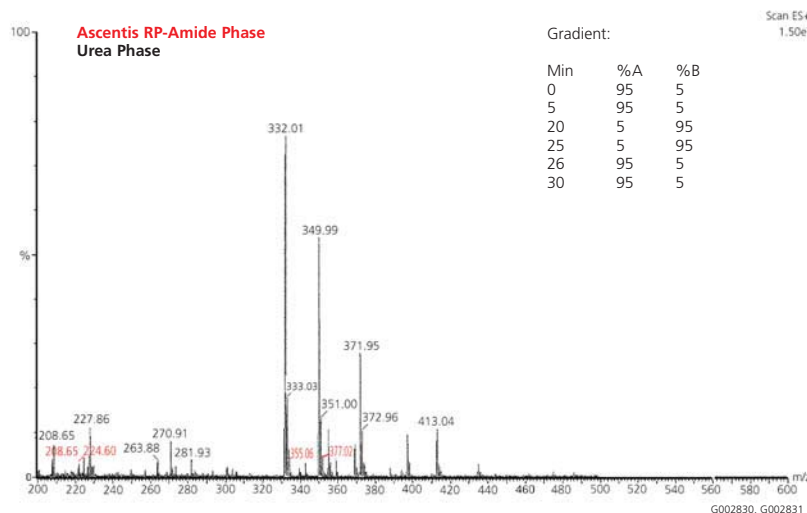


Table 1. Mass Responses and Intensities Attributed to Stationary Phase Bleed

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			

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 minor 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.

Ordering information

Prod No.	Particle Size (μm)	I.D. (mm)	Length (cm)
565300-U	3	2.1	5
565301-U	3	2.1	10
565302-U	3	2.1	15
565303-U	5	2.1	5
565304-U	5	2.1	10
565305-U	5	2.1	15
565306-U	5	2.1	25

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APPLICATION REPORT 200

LC-MS Analysis of Carnitine on Discovery® HS F5

Carnitine is an essential co-factor of fatty acid metabolism and a constituent of striated muscle tissue and the liver. An isocratic method is described suitable for LC-MS analysis.

Key Words

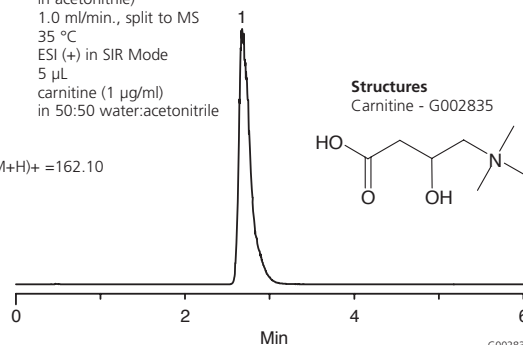
carnitine, 439584, 541-15-1 LC-MS, Discovery HS F5, 567513-U

Author: Carmen T. Santasania, Acquisition System: W2795
Notebook Reference: 1548-90

Conditions

column: Discovery HS F5, 5 cm x 4.6 mm I.D., 5 µm particles (567513-U)
mobile phase: 20:80, (10:90 0.1 M formic acid in water):(10:90 0.1 M formic acid in acetonitrile)
flow rate: 1.0 ml/min., split to MS
temp.: 35 °C
det.: ESI (+) in SIR Mode
injection: 5 µL
sample: carnitine (1 µg/ml) in 50:50 water:acetonitrile

Peak IDs
1. Carnitine (M+H)⁺ =162.10



APPLICATION REPORT 201

LC-MS Analysis of the Fungicide Iminoctadine on Discovery® HS F5

Iminoctadine is a guanidine fungicide used on fruits, vegetables and grains. A reversed gradient method is described suitable for LC-MS analysis.

Key Words

iminocadine, 13516-27-3, 39202-40-9, fungicide, LC-MS, Discovery HS F5, 567513-U)

Author: Carmen T. Santasania, Acquisition System: W2795
Notebook Reference: 1548-93

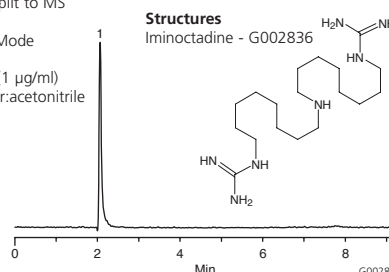
Conditions

column: Discovery HS F5, 5 cm x 4.6 mm I.D., 5 µm particles (567513-U)
mobile phase: A:B, 250 mM ammonium formate (pH 3.7 with formic acid): acetonitrile
flow rate: 1.0 ml/min., split to MS
temp.: 35 °C
det.: ESI (+) in SIR Mode
injection: 5 µL
sample: iminocadine (1 µg/ml) in 50:50 water:acetonitrile

Gradient:

Min	%A	%B
0	95	5
5	95	5
20	5	95
25	5	95
26	95	5
30	95	5

Peak IDs
1. Iminocadine (M+H)⁺ =356.34



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Ordering information

Discovery HS F5	
3µm Discovery HS F5 HPLC Columns	
567501-U 2.1	3.3 (call to order)
567500-U 2.1	5
567502-U 2.1	10
567503-U 2.1	15
567505-U 3.0	3.3 (call to order)
567542-U 3.0	15 (call to order)
567530-U 4	5
567531-U 4	10
567532-U 4	15
567509-U 4.6	3.3 (call to order)
567504-U 4.6	5
567506-U 4.6	10
567507-U 4.6	15
5µm Discovery HS F5 HPLC Columns	
567508-U 2.1	5
567510-U 2.1	10
567511-U 2.1	15
567512-U 2.1	25
567533-U 4	5
567534-U 4	10
567535-U 4	15

Discovery HS F5	
567536-U 4	25
567513-U 4.6	5
567515-U 4.6	10
567516-U 4.6	15
567517-U 4.6	25
567518-U 10	5
567519-U 10	10
567537-U 10	15
567520-U 10	25
567521-U 21.2	5
567539-U 21.2	10
567522-U 21.2	15
567523-U 21.2	25
10µm Discovery HS F5 HPLC Columns	
567524-U 10	5
567538-U 10	10
567525-U 10	15
567526-U 10	25
567527-U 21.2	5
567540-U 21.2	10
567528-U 21.2	15
567529-U 21.2	25

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Water blends

LC gradient testing in UV and MS, metal impurities (Na < 2 ppm, K, Mg, Ca < 0.5 ppm), UV-transmittance, additive content: 0.093-0.107 TFA, FA, AA (w/v), ammonium acetate (w/v), pH: effective +/- 0.1

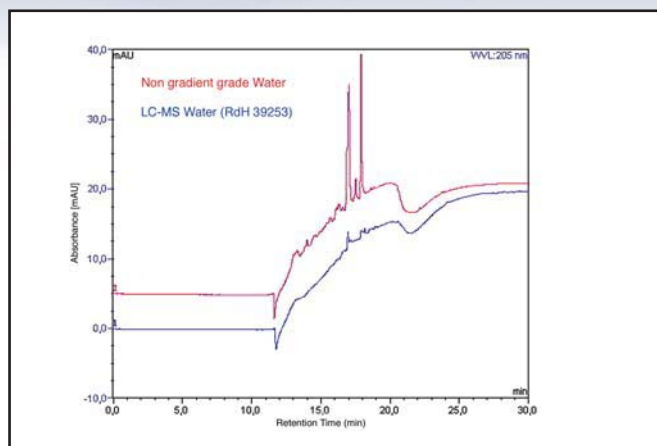
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34978	Water with 0.1% TFA	2.5 L
34976	Acetonitrile with 0.1% R TFA	2.5 L
34974	Methanol with 0.1% TFA	2.5 L
34673	Water with 0.1% formic acid	2.5 L
34668	Acetonitrile with 0.1% formic acid	2.5 L
34675	Water with 0.1% acetic acid	2.5 L
34678	Acetonitrile with 0.1% acetic acid	2.5 L
34672	Methanol with 0.1% acetic acid	2.5 L
34674	Water with 0.1% ammonium acetate	2.5 L
34669	Acetonitrile with 0.1% ammonium acetate	2.5 L
34670	Methanol with 0.1% ammonium acetate LC-MS CHROMASOLV®	2.5 L

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SPME ARTICLE

Improved SPME Fiber Life and Reproducibility with All Metal Fiber Assemblies

Robert Shirey bshirey@sial.com

Use of 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.

We have recently developed a new SPME fiber assembly 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 users 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 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.

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



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%

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

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...?

To completely eliminate septum coring, we highly recommend the use of Merlin Microseals™ or other septum-less inlet sealing devices.

 Information Request.....1605

SPME ARTICLE

Using SPME-GC-MS to characterize volatile components of honey as indicators of botanical origin

Antonella Verzera, Salvatore Campisi, Mario Zappalà

Istituto di Industrie Agrarie, Università di Catania.

Ivana Bonaccorsi, Dipartimento Farmaco-chimico, Università di Messina.

With introduction by Walter Gmelin, Sigma-Aldrich (wgmelin@sial.com).

Introduction

Like wine, olive oil and other natural liquids, honey is a complex mixture comprising a diverse population of compounds. Each compound gives the honey some particular nuance to its organoleptic properties or contributes to its nutraceutical value. Carbohydrates, from simple sugars to long-chain polysaccharides, account for 70-80% of honey by weight and give it its sweetness, viscosity and texture. Antioxidant properties of honey have been attributed to ascorbic acid, enzymes, minerals and polyphenolic compounds. Honey also contains organic acids, proteins and minerals. A wide variety of volatile organic compounds give honey its characteristic flavors and aromas. Using the unique GC-MS fingerprint of these volatile compounds to help identify the botanical source of the honey is the focus of the research reported herein.¹

The Analytical Challenge

Any flowering plant from which bees can harvest nectar can be a honey source. Unadulterated honey from a single plant source is termed unifloral. Plant nectars lend the honey its color, taste, aroma and other characteristics. Because plant nectars vary in composition, so do the honeys from which they are made. Examples of organoleptic properties of some honey varieties are found in Table 1. As with any natural product, there is the temptation to adulterate expensive honey with cheaper honey and pass it off as the genuine article. The challenge faced by analytical chemists in the honey industry and regulatory agencies is to provide an unambiguous determination of the botanical source of the honey sample.

Table 1. Organoleptic properties of honey samples analyzed in this study

Honey type	Colour	Physical state	Flavour
<i>Chestnut</i> (<i>Castanea sativa</i>)	Dark amber with reddish hues	Liquid or slowly granulating	Bitter, intense, astringent
<i>Orange</i> (<i>Citrus sinensis</i>)	White to light yellow	Granulating, medium crystals	Medium, sweet, floreal, persistent
<i>Eucalyptus</i> (<i>Eucalyptus camaldulensis</i>)	Beige with greyish hues	Complete irregular granulation, medium fine crystals	Intense and moderately persistent, soft caramel, mushrooms
<i>Sulla</i> (<i>Hedysarium coronarium</i>)	White to ivory	Complete regular granulation, medium fine crystals	Medium, not persistent, herbaceous, slightly fruity
<i>Wildflowers</i>	Amber ivory	Complete regular granulation, fine crystals	Medium, not persistent, herbaceous, sweet, floreal.

Because the current methods to estimate the botanic origin are useful but not confirmatory, we sought to develop an analytical method for its determination based on the volatile organic fraction. The method should be simple, rapid, sensitive and give unambiguous results. By using solid phase microextraction (SPME) followed by GC-MS analysis these objectives were met with success. We analyzed three samples of each of the

following unifloral honeys: orange (*Citrus sinensis* L.), eucalyptus (*Eucalyptus camaldulensis* L.), chestnut (*Castanea sativa* L.), sulla (*Hedysarium coronarium* L.), and wildflowers. Honey samples were supplied by Sicilian apiarists from different geographic areas of eastern Sicily.

Extraction of Volatile Organic Fraction using SPME

SPME was used to extract the organic volatile component of the honey without adding solvents that can alter its composition and reduce the reliability of the analytical results. For complete description of SPME, please see the "Further Reading" section at the end of this article. The SPME fiber used in this experiment was coated with polydimethylsiloxane/divinylbenzene (PDMS/DVB) with a 65 µm film thickness (Supelco).

The honey sample (16 grams) and 2 g NaCl were dissolved in 7 ml of water in a 40 ml glass headspace vial. The vial was equipped with a Mininert® valve (Supelco) which served to prevent loss of volatile components and allow insertion of the fiber without the need for a septum. The vial was gently heated to 30°C and equilibrated for 30 minutes. After equilibration, the SPME fiber was exposed to the vapor phase above the sample for 25 minutes with stirring. To ensure reproducible sampling, care was taken to place the fiber in the same location in the headspace during each exposure. Each sampling of the different varieties of honey was performed in triplicate.

Description

SPME fiber assembly, polydimethylsiloxane/divinylbenzene (PDMS/DVB) fiber, 65 µm film thickness

Manual SPME holder

Glass vials, 40 ml headspace

Mininert® valve

Hot plate for SPME extraction

SUPELCO WAX 10 Capillary GC column, 60 m x 0.25 mm i.d., 0.25 µm film thickness

GC-MS Analysis

After sampling, the SPME fiber was introduced onto the GC injector in splitless mode and held for 3 minutes to permit the complete desorption of the analytes from the fiber onto the column. The capillary GC column was a polyethylene glycol bonded Carbowax 20M phase, 60 m x 0.25 mm i.d., 0.25 µm film thickness. The GC-MS instrument was a STAR 3400 CX gas chromatograph, interfaced with a SATURN 3 ion trap mass spectrometer (Varian). The injector temperature was 220 °C. The GC oven temperature program was 45 °C for 10 seconds, increasing to 250 °C at a rate of 2 °C/min. The carrier gas was helium at a constant pressure of 10 psi. Transfer line temperature was 200 °C. MS acquisition range was 40–650 *m/z* with a scan rate of 2 µm/sec. The MS library was NIST 92.

Optimizing SPME Extraction Parameters

The complexity and high sugar content of honey are challenges to SPME optimization. Several key experimental parameters were studied in order to maximize sensitivity, reproducibility and fiber lifetime:

Liquid vs. headspace sampling: Inserting the SPME fiber

directly into the liquid sample resulted in better sensitivity, but the method lacked repeatability. Also, the high sugar content caused the fiber to become unusable after only three analyses.

Chemistry (polarity) of SPME fiber coating: PDMS/DVB was the most suitable fiber coating for the volatile components of honey. Nonpolar stationary phases such as PDMS gave very low recovery of all components.

Sample preparation: The honey was diluted with water to decrease its viscosity and to facilitate release of compounds of interest from the sugars. The addition of NaCl to the sample increased the extraction recovery by decreasing the solubility of hydrophobic compounds, forcing them into the headspace.

Sampling temperature: The temperature used during the extraction was 30 °C to avoid artifacts and Maillard (browning) reactions.

SPME fiber exposure time: A sampling time of 25 minutes was optimal for the components of interest.

Headspace volume-sample volume ratio: The headspace-sample volume ratio of about 1:1 provided the highest amount of extracted compounds.

Results

Figures 1-5 show the GC-MS traces of SPME extracts from five different unifloral honey samples. Spectral data, linear retention indices (LRI)^a and standard injection were used to identify the components. Using the SPME-GC-MS method, 113 unique components were identified and found to include aliphatic and aromatic compounds, acyclic and monocyclic monoterpenes and their oxygenated derivatives, furan derivatives and sulfur and nitrogen-containing compounds. A list of the 113 compounds and methods used for their identification appear in Table 3.

Because the aim of the investigation was to see if volatile organic compounds could be used to determine botanical origin, a correlation was sought between the types of compounds or ratios between key compounds identified by GC-MS and the variety of honey. Most of the compounds reported in Table 3 were found in all the honey samples analyzed. However, there were some distinct markers and the ratios between certain compounds differed widely between varieties. The variation in peak heights in the GC-MS traces in Figures 1-5 attests to the differences. Some interesting findings related to specific honeys were:

1. Eucalyptus honey (Figure 1) is characterized by high levels of nonanol, nonanal, nonanoic acid, 5-hexen-2-ol, and 2,3-dimethyl-5-hexen-2-ol. Borneol and dihydrocarveol are unique to this variety of honey.
2. Orange honey (Figure 2) is distinctive because of its high level of hotrienol (3,7-dimethyl-1,5,7-octatrien-3-ol). Methyl anthranilate is also a possible marker for orange honey.
3. Wildflower honey (Figure 3) has a high amount of hexanol, linalol, hexanoic acid, and 3-methylbutanoic acid. Markers of wildflower honey may be myrcenol and cis-carveol.
4. Sulla honey (Figure 4) contains high levels of hexanol, hexanoic acid, linalol, nonanal, terpinen-4-ol and α -terpineol.
5. Chestnut honey (Figure 5) has a high amount of nonanal, nonanol, benzaldehyde and camphor. Markers for chestnut honey are acetophenone, 2-aminoacetophenone and 1-phenylethanol.

Figure 1. SPME-HRGC/MS chromatogram of a eucalyptus honey.

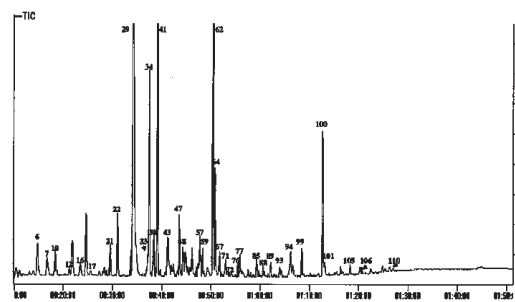


Figure 2. SPME-HRGC/MS chromatogram of an orange honey.

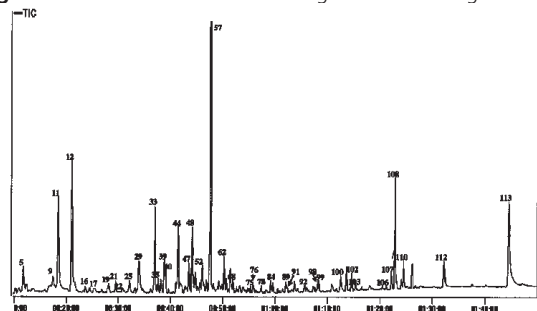


Figure 3. SPME-HRGC/MS chromatogram of a wild flower honey.

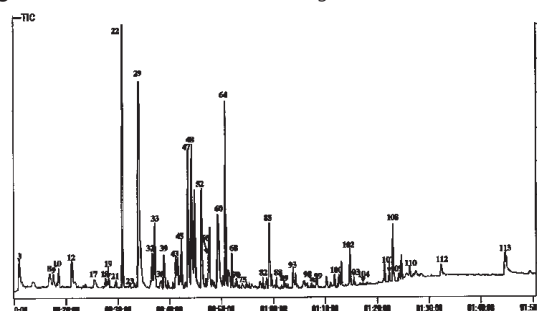


Figure 4. SPME-HRGC/MS chromatogram of a sulla honey.

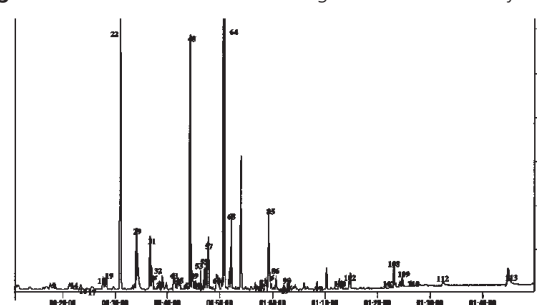
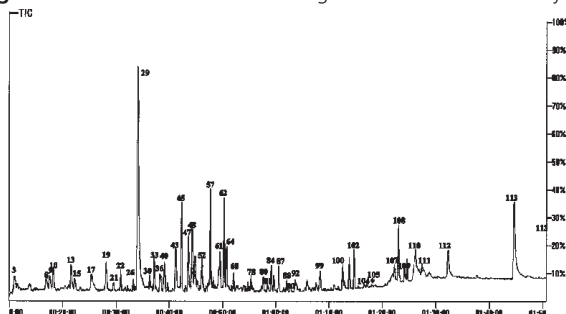


Figure 5. SPME-HRGC/MS chromatograms of a chestnut honey.



Repeatability of the Method

An analytical method is not valuable if it cannot be repeated. To demonstrate method reproducibility, SPME-GC-MS was performed on three different samples of the same orange blossom honey under identical conditions. The peak area for each component identified during the three different analyses was tabulated and the RSD was calculated. The average value of the resulting RSD was approximately 10%, with maximum values less than 20% for trace components. Table 3 shows the RSD for fifteen components from the three samples of orange blossom honey.

Conclusions

The results of this investigation suggest that SPME followed by GC-MS analysis can be used to identify compounds in the volatile fraction of honey and use them to confirm its botanical origin. The technique is simple – it requires instrumentation found in most modern analytical laboratories, rapid – samples can be prepared, extracted and analyzed in less than one hour, selective – distinct differences in the levels of certain compounds or the ratio of individual compounds were seen between honey from different botanical sources, and reproducible – the %RSD values for triplicate measurements were less than 10% for most of the compounds identified.

(a) The LRI of a compound is an expression of its retention time on a gas chromatographic column relative to a homologous series of n-alkanes

References

(1) Verzera, A.; Campisi, S.; Zappalà, M.; Bonaccorsi, I.; *American Laboratory*; July 2001; 18-21.

Further Reading

- SPME users guide, CD etc.)
- Table and Figures used with permission from reference 1.
- Figure 1 SPME-GC-MS chromatogram of eucalyptus honey
- Figure 2 SPME-GC-MS chromatogram of orange blossom honey
- Figure 3 SPME-GC-MS chromatogram of wildflower honey
- Figure 4 SPME-GC-MS chromatogram of sulla honey
- Figure 5 SPME-GC-MS chromatograms of chestnut honey

Table 2.

Repeatability of the SPME-GC-MS Method: Peak area and % RSD for fifteen components from three different samples of orange blossom honey

Compounds	% RSD*
1,8-Cineole	8.3
(E)-2-Pentenol	11.1
Hexanol	10.7
n-Nonanal	7.8
Linalool oxide (cis furanoid)	9.4
Acetic acid	4.5
Furfural	3.5
Linalool oxide (trans furanoid)	10.8
n-Decanal	8.8
Menthofurane	9.8
Benzaldehyde	7.7
Linalol	5.3
Nonanol	4.7
Monanoic acid	7.1
Benzoic acid	3.4

(* Mean as a percentage of the standard deviation of the triplicate analyses.)

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Table 2 Compounds Identified in Honey Samples and Their Methods of Identification

Compound	LRI	Std.	MS	Ref.	Literature LRI
1 Toluene	1033	x	x	x	1036(d)
2 Dimethyldisulphide	1062	x	x	x	
3 Hexanal	1077	x	x	x	1080(d)
4 (Z)-2-Pentenal	1088		x		1105(d)
5 Undecane	1096	x	x	x	1100(b)
6 2,5-Dimethyl-3-hexanone	1145		x		
7 2-Heptanone	1173		x	x	1172(a)
8 Heptanal	1176	x	x	x	1183(a)
9 Limonene	1185	x	x	x	1187(c)
10 3-Methyl-1-butanol	1194		x	x	1196(a)
11 1,4-Cineole	1198		x		1185(c)
12 1,8-Cineole	1230	x	x		1228(c)
13 3-Methyl-3-buten-1-ol	1236	x	x	x	1248(d)
14 Pentanol	1239	x	x	x	1249(a)
15 1,3,5,7-cyclooctatetraene	1244		x		
16 1-Methylethyl benzene	1256		x	x	1265(b)
17 Octanal	1278	x	x	x	1284(d)
18 4-Methyl-1-pentanol	1299		x		1316(d)
19 (E)-2-Pentenol	1306		x		1313(d)
20 (Z)-2-Pentenol	1314		x		1321(d)
21 6-Methyl-5-hepten-2-one	1324		x		1335(a)
22 Hexanol	1342	x	x	x	1346(a)
23 (E)-3-Hexenol	1351		x	x	1365(d)
24 Dimethyltrisulphide	1357		x	x	
25 cis-Rose oxide	1358		x	x	
26 (Z)-3-Hexenol	1370		x	x	1378(a)
27 2-Nonanone	1374		x		
28 4-Methyl-3-pentenol	1375		x		1390(d)
29 Nonanal	1380	x	x	x	1380(a)
30 (E)-2-Octenal	1407		x		1423(a)
31 3-Methyl-1-hexanol	1413		x		
32 a-p-Dimethylstyrene	1417		x	x	
33 cis-Linalol oxide (furanoid)	1420	x	x	x	1432(a)
34 5-Hexen-2-ol	1425		x		
35 1,3,8-p-Menthatriene	1429		x		
36 Acetic acid	1436	x	x	x	1451(b)
37 1-Octen-3-ol	1437		x		1452(d)
38 Heptanol	1439	x	x		1445(a)
39 Furfural	1447	x	x	x	1450(a)
40 trans-Linalol oxide (furanoid)	1450		x	x	1451(c)
41 2,3-Dimethyl-5-hexen-2-ol	1451		x		
42 2-Decanone	1476		x		
43 Decanal	1480	x	x	x	1487(a)
44 Menthofurano	1487	x	x		1503(c)
45 Benzaldehyde	1496	x	x	x	1509(a)
46 2-Decanol	1499		x		
47 Camphor	1517	x	x	x	1518(c)
48 Linalol	1529	x	x	x	1547(a)
49 Octanol	1538	x	x		1539(a)
50 2-Methyl propanoic acid	1549		x	x	1571(b)
51 1,3-butandiol	1556		x	x	1576(b)
52 Terpinen-1-ol	1562		x		1576(c)
53 Isophorone	1563		x	x	1600(c)
54 Terpinen-4-ol	1577	x	x		1601(c)
55 Myrcenol	1581		x		1585(c)
56 a,4-Dimethyl-3-cyclohexen-1-acetaldehyde	1585		x	x	
57 Hotrienol	1589		x	x	1601(a)
58 Nonanetrile	1593		x		
59 Butanoic acid	1599	x	x	x	1600(a)
60 Phenylacetaldehyde	1618	x	x	x	1640(a)
61 Acetophenone	1621		x	x	
62 Nonanol	1636	x	x	x	
63 5-Methyl-5-ethenyldihydro-2(3H)-furanone	1639		x	x	
64 3-Methyl butanoic acid	1645	x	x	x	1639(a)
65 Furfuryl alcohol	1656	x	x	x	1662(b)

Table 2 Compounds Identified in Honey Samples and Their Methods of Identification

Compound	LRI	Std.	MS	Ref.	Literature LRI
66 Neral	1662	x	x		1676(a)
67 4-Oxoisophorone	1663		x	x	
68 a-Terpineol	1670	x	x	x	1685(c)
69 Heptadecane	1681	x	x	x	1700(b)
70 Dodecanal	1685	x	x		1687(a)
71 Borneol	1686	x	x	x	1698(a)
72 Dihydrocarveol	1698		x		1713(c)
73 cis-Linalol oxide (pyranoid)	1708		x	x	1737(a)
74 Carvone	1714	x	x		1715(c)
75 (E)-2-Undecenal	1722		x		
76 Decanol	1736	x	x		1735(a)
77 Methyl salicylate	1744		x	x	1748(a)
78 Myrtenol	1762		x		
79 Methyl hexanoic acid	1775		x		1789(a)
80 2-Hydroxy-ethyl benzoate	1780		x		
81 1-Phenylethanol	1782	x	x	x	
82 b-Damascenone	1788		x	x	
83 cis-Carveol	1800	x	x		1820(d)
84 Nerol	1803	x	x		1808(d)
85 Hexanoic acid	1813	x	x	x	1821(a)
86 p-Cymen-8-ol	1818		x		1846(c)
87 Geranyl acetone	1825		x		
88 Benzyl alcohol	1844	x	x	x	1878(c)
89 Phenylethyl alcohol	1878	x	x	x	1907(c)
90 Methyl heptanoate	1893		x		
91 Benzeneacetonitrile	1893		x		
92 2,6-Dimethyl-3,7-octadien-2,6-diol	1914		x	x	
93 Heptanoic acid	1918		x		1913(a)
94 2-Methyldihydro-2(3H)-furanone	1971		x	x	
95 Methyl octanoate	1977		x		
96 4-Methyldihydro-2(3H)-furanone	1982		x		
97 2,6-Dimethyl-3,7-octadien-1,6-diol	1995		x	x	
98 g-Nonalactone	2019		x	x	
99 Octanoic	2022	x	x	x	2025(a)
100 Nonanoic	2124	x	x	x	2128(b)
101 Hexanedioic	2137		x	x	
102 Methyl decanoate	2178		x		
103 Methyl anthranilate	2197	x	x		
104 Decanoic acid	2227	x	x		
105 Octadecanal	2271	x	x		
106 Hexadecanol	2332	x	x		
107 Benzoic acid	2380	x	x	x	2431(a)
108 Methyl decenoate	2397		x	x	
109 Dodecanoic acid	2430	x	x	x	2477(d)
110 2-Acetyl benzoic acid	2489		x	x	x
111 3-Aminoacetophenone	2518		x	x	x
112 Yetradecanoic acid	2634	x	x	x	x
113 Hexadecanoic acid	2832	x	x		

LRI = Linear retention indices

St = Standard injection

MS = Mass spectra

Ref. = Reference data

Lit. LRI = Linear retention indices from literature references (see below)

(a) Boelens, M.H.; *Perf. Flav.*; **1995**; 20:23–51.(b) Shimoda, M.; Wu, Y.; Osajima, Y.; *J. Agric. Food Chem.*; **1996**; 44:3913–18.(c) Umano, K.; Nakahamara, K.; Shoji, A.; Shibamoto, T.; *J. Agric. Food Chem.*; **1999**; 47:3702–5.(d) Davies, N.W.; *J. Chromatogr.*; **1990**; 503:1–24.

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52783-U	DSC-MCAX,	100mg/3ml
52784-U	DSC-MCAX,	300mg/3ml
52786-U	DSC-MCAX,	300mg/6ml
52788-U	DSC-MCAX,	1g/6ml
575639-U	DSC-MCAX, 96-Well	25mg/well
575640-U	DSC-MCAX, 96-Well	50mg/well
575641-U	DSC-MCAX, 96-Well	100mg/well

GC ARTICLE

Carbon Adsorbent Kits for Sample Prep & Method Development

Robert Shirey bshirey@sial.com

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 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,

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Table 1. Physical Characteristics of Supelco Carbon Adsorbents

	BET surface area [▲] , m ² /g	Density, g/ml	micro-	Porosity, cc/g meso-	macro-	Micropore Diameter, Å
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, monoporous carbon sieve

□ closed pore structure



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

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

Ordering information

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

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Ordering information

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24340	Kit I
24328-U	Kit II

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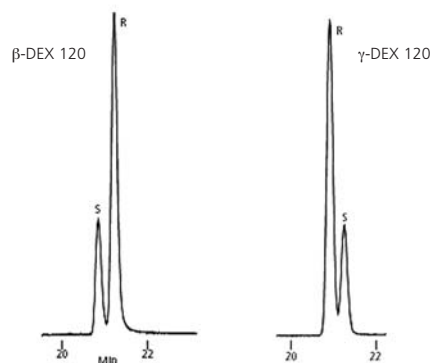
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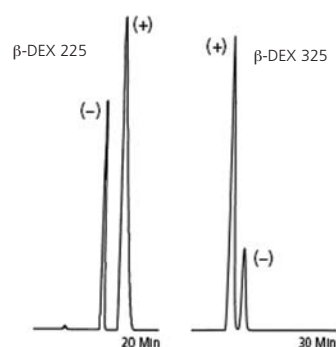
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 det.: FID, 300°C
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Column: α -DEX 120 and γ -DEX 120, 30m x 0.25 mm I.D., 0.25 μ m
 Oven.: 130°C
 Inj.: 250°C
 det.: FID, 300°C
 Carrier gas: helium, 35 cm/sec
 Sample: helium, 35 cm/sec



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PRODUCT INFORMATION

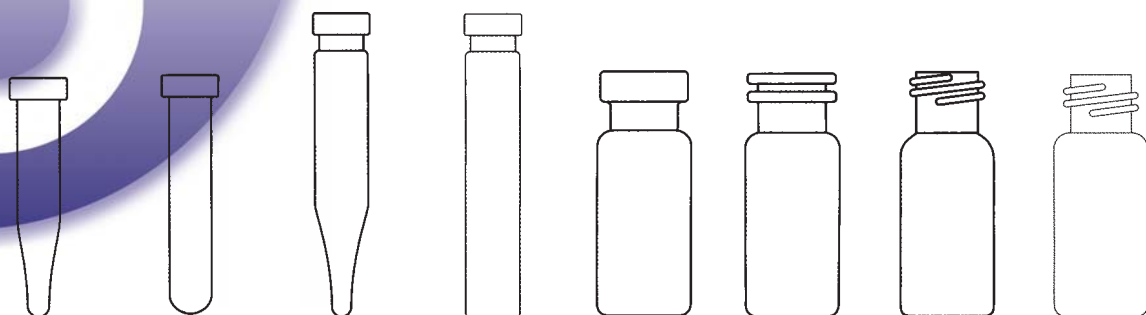
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Vials	Volume/Pk Size	0.3ml	0.3ml	0.7ml	0.7ml	1.5ml	1.5ml	1.5ml	1.5ml
Dimensions		31.5 x 5.5mm	31.5 x 5.5mm	40 x 7mm	40 x 7mm	32 x 11.6mm	32 x 11.6mm	32 x 11.6mm	32 x 11.6mm
Neck		Crimp Neck ND 8	Crimp Neck ND 8	Crimp Neck ND 8	Crimp Neck ND 8	Crimp Neck ND 11	Snap Ring, ND11	Short Thread ND9	Screw Neck ND8
Opening		Standard	Standard	Standard	Standard	Wide, 6mm	Wide, 6mm	Wide, 6mm	Standard 4.6mm
Bottom		Tip	Round	Tip	Flat	Flat	Flat	Flat	Flat
Clear Glass	pk. 100	SU860057	SU860056	SU860058	SU860060	854000	SU860068	SU860069	854982
	pk. 1000			SU860025	SU860023	854964	854974	854972	854997
Amber Glass	pk. 100			SU860059	SU860061	SU860063	SU860089	SU860088	SU860083
	pk. 1000			854027	SU860024	854981	854993	854994	854983
Clear Glass Marking Spot	pk. 100					SU860064	SU860081	854165	854171
	pk. 1000					854500			
Amber Glass Marking Spot	pk. 100					854998	SU860082	SU860033	854172
CAP /SEPTA*									
PTFE / Silicon	pk. 100	27359	27359	27359	27359	SU860094	SU860093	SU860092	SU860076
	pk. 1000	27372	27372	27372	27372	SU860016		SU860019	854985
PTFE / Natural Rubber	pk. 100	SU860077	SU860077	SU860077	SU860077	854140	SU860090	854161	SU860091
	pk. 1000					854980-U	854975	854973	854984
PTFE / Synthetic Rubber	pk. 100					27102-U			
	pk. 200	33135-U	33135-U	33135-U	33135-U				
	pk. 1000	33136	33136	33136	33136	33233-U			
PTFE / Butyl	pk. 100								
PTFE / Silicon / PTFE	pk. 100	SU860087	SU860087	SU860087	SU860087	SU860080		SU860079	
	pk. 1000	SU860028	SU860028	SU860028	SU860028			SU860020	
Pharma-Fix									
Magnetic, Pharma-Fix	pk. 100								
Magnetic, PTFE / Silicon	pk. 100					SU860094			
	pk. 1000					SU860018			
Magnetic, PTFE / Silicon / PTFE	pk. 100					SU860095			
	pk. 1000					SU860017			
SPME, Magnetic, Black Viton	pk. 100								
SPME, Magnetic, Silicon /PTFE	pk. 100								
INSERT									
0,1ml, 31 x 6mm	pk. 100					SU860067	SU860067	SU860067	
	pk. 1000					854988	854988	854988	854995
0,1ml, 28 x 6mm, plastic spring	pk. 100					SU860066	SU860066	SU860066	
	pk. 1000					854110	854110	854110	

- ✓ Huge range
- ✓ Great prices
- ✓ Outstanding Service

24hr delivery:

More than 95% of our products are delivered next day.



Vials	Vol/Pk Size	4ml	4ml	5ml	10ml	10ml	20ml	20ml	20ml	20ml
Dimensions		45 x 14.75mm	44.6 x 14.65mm	38.25 x 22mm	46 x 23mm	46 x 22.5mm	75.5 x 23mm	75 x 22.5mm	75.5 x 22.5mm	75.5 x 22.5mm
Neck		Screw Neck ND 13	ND15 with PE plug	Crimp, ND 20	Crimp Neck ND 20	Crimp, ND 20	Crimp, ND 20	Crimp, ND 20	Crimp, ND 20	Crimp, 5.2 mm DIN long neck
Opening		Standard	Standard	Standard	Standard	Standard	Standard	Standard	Standard	Standard
Bottom		Flat	Flat	Round	Flat	Round	Round	Flat	Round	HS-Bottom
Clear Glass	pk. 100	854190	854189	SU860065	SU860029	854180-U	SU860049	SU860030	854181-U	SU860051
	pk. 1000									
Amber Glass	pk. 100	854986								
	pk. 1000									
Clear Glass Marking Spot	pk. 100									
	pk. 1000									
Amber Glass Marking Spot	pk. 100									
CAP /SEPTA*										
PTFE / Silicon	pk. 100	SU860078		854996	854996	854996	854996	854996	854996	854996
	pk. 1000									
PTFE / Natural Rubber	pk. 100	854987								
	pk. 1000									
PTFE / Synthetic Rubber	pk. 100									
	pk. 200									
	pk. 1000									
PTFE / Butyl	pk. 100			854979	854979	854979	854979	854979	854979	854979
PTFE / Silicon / PTFE	pk. 100									
	pk. 1000									
Pharma-Fix				SU860084	SU860084	SU860084	SU860084	SU860084	SU860084	SU860084
Magnetic, Pharma-Fix	pk. 100					854178-U			854178-U	SU860014
Magnetic, PTFE / Silicon	pk. 100					854179-U			854179-U	SU860015
	pk. 1000									
Magnetic, PTFE / Silicon / PTFE	pk. 100									
	pk. 1000									
SPME, Magnetic, Black Viton	pk. 100									
SPME, Magnetic, Silicon /PTFE	pk. 100									SU860053
INSERT										
0,1ml, 31 x 6mm	pk. 100									
	pk. 1000									
0,1ml, 28 x 6mm, plastic spring	pk. 100									
	pk. 1000									



GC PRODUCT INFORMATION

Cross Reference List for Hamilton GC Syringes



Ordering information

Prod No.	Description	Volume (µL)	Needle	Needle Length (mm)	Agilent/HP No.
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Autosampler Syringes

Hamilton Syringes for Agilent/HP 7673 & 7683

Hamilton 700 Series Microliter Syringe, Fixed Needle

21310-U	75 ASN	2.5 L	26s ga (cone tip)	43 (1.71 in.)	5183-4728
21314	75 ASN	2.5 L	26s ga (cone tip)	43 (1.71 in.)	5183-4728
24570-U	75 ASN	2.5 L	23s-26s ga (cone tip)	43 (1.71 in.)	5181-1273
24571	75 ASN	2.5 L	23s-26s ga (cone tip)	43 (1.71 in.)	5181-8810
21313	701 ASN	2.5 L	23s ga (cone tip)	43 (1.71 in.)	9301-0713
21317	701 ASN	2.5 L	23s ga (cone tip)	43 (1.71 in.)	9301-0725
24573	701 ASN	2.5 L	23s-26s ga (cone tip)	43 (1.71 in.)	5181-1267
24574	701 ASN	2.5 L	23s-26s ga (cone tip)	43 (1.71 in.)	5181-3360

Prod No.	Description	Volume (µL)	Needle	Needle Length (mm)	Hamilton No.
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Autosampler Syringes: Hamilton Syringes for Agilent/HP 7670, 7671, & 7672

Hamilton Syringe, 700 Series

20734	701N	10	26s ga (bevel tip)	51 (2 in.)	80300
20779	701N	10	26s ga (bevel tip)	51 (2 in.)	80366

Hamilton Syringe, 7000 Series Modified Microliter

20750	7001	1	25s ga (bevel tip)	70 (2.75 in.)	80135
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Hamilton Syringes for CTC/Leap Technologies

Prod No.	Description	Volume (µL)	Needle	Needle Length (mm)	Hamilton No.
	Sampling	GC	Syringes, PAL Instruments		
28613-U	75N	5	26s ga (cone tip)	51 (2 in.)	203189
28614-U	701N	10	26s ga (bevel tip)	51 (2 in.)	203072
28615-U	701N	10	26s ga (cone tip)	51 (2 in.)	203205
28649-U	1702N	25	26s ga (cone tip)	51 (2 in.)	203043

Prod No.	Description	Volume (µL)	Needle	Needle Length (mm)	Hamilton No.
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700 Series Syringes

700 Series, N (Cemented Needle)

26200-U	75N	5	26s ga (bevel tip)	51 (2 in.)	87900
20734	701N	10	26s ga (bevel tip)	51 (2 in.)	80300
20735	702N	25	22s ga (bevel tip)	51 (2 in.)	80400
20736	705N	50	22s ga (bevel tip)	51 (2 in.)	80500
20737	710N	100	22s ga (bevel tip)	51 (2 in.)	80600
20738	725N	250	22s ga (bevel tip)	51 (2 in.)	80700
20739	750N	500	22 ga (bevel tip)	51 (2 in.)	80800

700 Series, RN (Removable Needle)

20919	75RN		26s ga (bevel tip)	51 (2 in.)	87930
20697	701RN	10	26s ga (bevel tip)	51 (2 in.)	80330
20787	702RN	25	22s ga (bevel tip)	51 (2 in.)	80430
20788	705RN	50	22s ga (bevel tip)	51 (2 in.)	80530
20790-U	710RN	100	22s ga (bevel tip)	51 (2 in.)	80630
24538-U	725RN	250	22s ga (bevel tip)	51 (2 in.)	80730
24539	750RN	500	22s ga (bevel tip)	51 (2 in.)	80830

800 Series Syringes

Hamilton Microliter Syringes, 800 Series, Removable Needle

26201	85RN	5	26s ga (bevel tip)	51 (2 in.)	84851
20797	801RN	10	26s ga (bevel tip)	51 (2 in.)	84853
21493	802RN	25	26s ga (bevel tip)	51 (2 in.)	84855

900 Series Syringes

Economical version of 800 series syringes.

20907-U	95RN	5	26s ga (bevel tip)	51 (2 in.)	87925
20909-U	901RN	10	26s ga (bevel tip)	51 (2 in.)	80370

Prod No.	Description	Volume (ml)	Size	Needle Length (mm)	Hamilton No.
Hamilton Gastight Syringes					
1000 Series, RN (Removable Needle)					
22192-U	1001RN	1	22 ga (bevel tip)	51 (2 in.)	81330
22193-U	1002RN	2.5	22 ga (bevel tip)	51 (2 in.)	81430
22194-U	1005RN	5	22 ga (bevel tip)	51 (2 in.)	81530
22195	1010RN	10	22 ga (bevel)	51 (2 in.)	81630
1000 Series, LTN (Fixed Needle)					
20740-U	1001LTN	1	22 ga (bevel tip)	51 (2 in.)	81317
20692	1005LTN	5	22 ga (bevel tip)	51 (2 in.)	81517
20693	1010LTN	10	22 ga (bevel tip)	51 (2 in.)	81617
1000 Series, TLL (Teflon Luer Lock)					
20997	1001TLL	1	-	not included	81320
20998	1002TLL	2.5	-	not included	81420
20999	1005TLL	5	-	not included	Tekmar 14-0069- 52
21000-U	1010TLL	10	-	not included	81620
20683	1025TLL	25	-	not included	Tekmar 14-0070- 52
20707	1050TLL	50	-	not included	85020
21967	1100TL	100	-	not included	86020
Hamilton Gastight Syringes: 1700 Series Gastight Syringes					
1700 Series, N (Cemented Needle)					
20972	1701N	10	26s ga (bevel tip)	51 (2 in.)	80000
20973	1702N	25	22s ga (bevel tip)	51 (2 in.)	80200
20687	1705N	50	22s ga (bevel tip)	51 (2 in.)	80900
20688	1710N	100	22s ga (bevel tip)	51 (2 in.)	81000
20689	1725N	250	22s ga (bevel tip)	51 (2 in.)	81100
1700 Series, RN (Removable Needle)					
20780-U	1701RN	10	26s ga (bevel tip)	51 (2 in.)	80030
20782	1705RN	50	22s ga (bevel tip)	51 (2 in.)	80930
20783	1710RN	100	22s ga (bevel tip)	51 (2 in.)	81030
20784	1725RN	250	22s ga (bevel tip)	51 (2 in.)	81130
20785-U	1750RN	500	22 ga (bevel tip)	51 (2 in.)	81230
20781	1702RN	25	22s ga (bevel tip)	51 (2 in.)	80230
Prod No.	Description	Volume (µL)	Size	Needle Length (mm)	Hamilton No.
7000 Series Modified Microliter Syringes					
22185-U	7000.5	0.5	25 ga (bevel tip)	70 (2.75 in.)	86259
20750	7001	1	25s ga (bevel tip)	70 (2.75 in.)	80135
20731	7001	1	25s ga (blunt tip)	70 (2.75 in.)	80100
20979	7101	1	22s ga (bevel tip)	70 (2.75 in.)	86211
20751	7002	2	25 ga (bevel tip)	70 (2.75 in.)	88411
24592	7102	2	23 ga (bevel tip)	70 (2.75 in.)	88511
20980-U	7105	5	24 ga (bevel tip)	70 (2.75 in.)	88011
20728	7105	5	24 ga (blunt tip)	70 (2.75 in.)	88000

OFFER

FREE CD Case

Buy any listed GC syringe and get a CD case for free.

Promotional code: xxx

Offer valid until 31st July 2005



Hamilton Syringe Replacement Parts



Point Style No.1 - Cone Tip

Recommended for use with pre-drilled septa. The shape of this needle has been developed for multi-injections on the Agilent/HP 7673A autosampler.



Point Style No.2 - Bevel Tip

The bevel tip (22° on Hamilton syringes, 20° on SGE syringes) is designed for optimum septum penetration and to prevent septum coring.



Point Style No.3 - Blunt Tip

The 90° blunt tip has chamfered and polished edges that eliminate damage to the valve's rotor seal and stator face. This style also can be used for pipetting of liquids.



Point Style No.5 - Cone Tip, Side-port Hole

Liquid samples can be filled and dispensed through the side hole, and septum damage is minimized by the solid domed tip.

Prod No.	Description	Volume (µL)	Size	Needle Length (mm)	Hamilton No.
Needles for Hamilton RN Syringes, Point Style #1 (Cone Tip)					
21327-U	cone tip	5-10	23s ga	43 x 0.63	80457
21326	cone tip	5-10	26s ga	43 x 0.47	80458
24582	cone tip	5-10	23s - 26s ga	43 x 0.47	80460
Needles for Hamilton RN Syringes, Point Style #2 (Bevel Tip)					
20760-U	bevel tip	2.5-100	26s ga	51 (2 in.)	7758-02
20762	bevel tip	2.5-100	26 ga	51 (2 in.)	7758-04
20761	bevel tip	2.5-100	22s ga	51 (2 in.)	7758-03
Z288993-1PAK	bevel tip	2.5-100	22 ga	51 (2 in.)	7758-01
23940	bevel tip	250-10000	26s ga	51 (2 in.)	7779-02
23941	bevel tip	250-10000	26 ga	51 (2 in.)	7779-04
20798	bevel tip	250-10000	22s ga	51 (2 in.)	7779-03
20799	bevel tip	250-10000	22 ga	51 (2 in.)	7779-01
Needles for Hamilton RN Syringes, Point Style #3 (Blunt Tip)					
20859	blunt tip	2.5-100	26s ga	51 (2 in.)	7768-01
58398	blunt tip	2.5-100	25s ga	50 (1.97 in.)	80426
58649	blunt tip	2.5-100	22s ga	51 (2 in.)	7770-01
26708	blunt tip	250-10000	26s ga	51 (2 in.)	7780-01
58399	blunt tip	250-10000	25s ga	50 (1.97 in.)	80726
20862	blunt tip	250-10000	22s ga	51 (2 in.)	7780-03
58650-U	blunt tip	250-10000	22 ga	51 (2 in.)	7780-04
26710-U	blunt tip	250-10000	16 ga	51 (2 in.)	80780
26709	blunt tip	250-10000	26 ga	51 (2 in.)	7780-02
Needles for Hamilton RN Syringes, Point Style #5 (Cone Tip, Side port Hole)					
20878	cone tip, side-port	2.5-100	26s ga	51 (2 in.)	7784-07
20879	cone tip, side-port	2.5-100	22s ga	51 (2 in.)	7784-05
26711	cone tip, side-port	250-10000	26s ga	51 (2 in.)	7784-03
26712	cone tip, side-port	250-10000	26 ga	51 (2 in.)	7784-04
26713	cone tip, side-port	250-10000	22s ga	51 (2 in.)	7784-01
20880	cone tip, side-port	250-10000	22 ga	51 (2 in.)	7784-02
Hamilton Needles for LT and TLL Syringes					
20757	Metal hub	-	28 ga (bevel tip)	51 (2 in.)	90028
21746	Metal hub	-	22 ga (bevel tip)	51 (2 in.)	90022
21748-U	Metal hub	-	22s ga (bevel tip)	51 (2 in.)	90038
20756	KEL-F hub	-	28 ga (bevel tip)	51 (2 in.)	90128
21749	KEL-F hub	-	22s ga (bevel tip)	51 (2 in.)	90138
21747	KEL-F hub	-	22 ga (bevel tip)	51 (2 in.)	90122
21741	Metal hub	-	22 ga (blunt tip)	51 (2 in.)	91022
-	-	-	-	-	-
21744	KEL-F	-	22 ga (blunt tip)	51 (2 in.)	90134
Z288977--	KEL-F hub	-	22s ga (blunt tip)	51 (2 in.)	90534
1PAK	-	-	-	-	-
20803	Metal hub	-	22 ga (cone tip, side-port)	51 (2 in.)	90222
21743	KEL-F hub	-	22s ga (cone tip, side-port)	51 (2 in.)	90438
21742-U	KEL-F hub	-	22 ga (cone tip, side-port)	51 (2 in.)	90422

Consistent, Reproducible Performance

Choose Equity for all your:

- Special Purpose GC/MS Analyses
- Demanding Environmental Methods
- General Purpose Applications

Equity-1, Equity-5, and Equity-1701

- The resolution you need for accurate compound identification
- The response you require for reliable quantitation
- The low bleed you expect for lower detection limits
- The column life you count on for increased productivity



OFFER

25% OFF Equity GC Columns
(same or smaller dimensions)

Promotional Code: V60

Offer valid until 31st July 2005

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 _i (µm)
0.20mm ID		
28041-U	12	0.33
28042-U	25	0.33
28043-U	10	1.2
0.25mm ID		
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
0.32mm ID		
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
0.53mm ID		
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

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

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 _i (µm)
0.10mm ID		
28083-U	15	0.10
0.20mm ID		
28084-U	15	0.20
28085-U	30	0.20
28086-U	60	0.20
28087-U	12	0.33
0.25mm ID		
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
0.32mm ID		
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
0.53mm ID		
28252-U	15	0.5

Ordering information

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 _i (µm)
0.25mm ID		
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
0.32mm ID		
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
0.53mm ID		
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

Information Request.....1608

STANDARDS ARTICLE

New Environmental Standards Offering 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 multicomponent solutions, and kits. Separate Source™ standards for several mixes will also be available.

Certification and Documentation

Each standard is backed by certification and documentation.

- A certificate of analysis (COA) summarizes the QA analysis of the final product for purity, component identity, and concentration.
- Certificate of composition (COC) provides a summary of gravimetric preparation of the final product.
- A 16 Chapter MSDS is provided with the first purchase of each product.
- **FREE** data packets are available upon request.
- Online COAs can be accessed for free, at anytime, through our website www.sigma-aldrich.com

Figure 1. Separate Source Total Petroleum Hydrocarbon (TPH) Standards

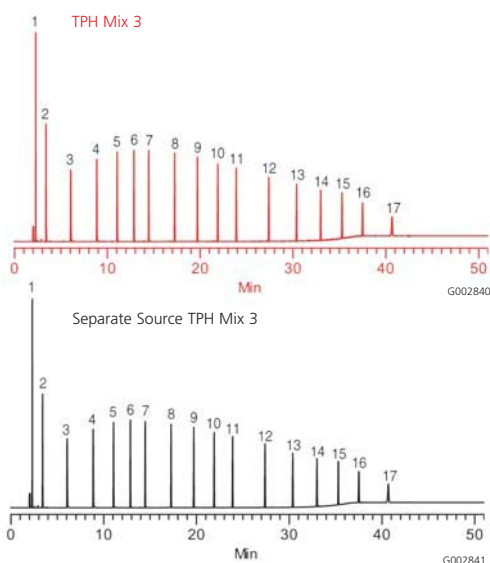
sample: TPH Mix 3, 1000 µg/ml each in CS2 (861394-U)
 sample: Separate Source TPH Mix 3, 1000 µg/ml each in CS2 (8561394-U)
 column: Equity-5, 60 m x 0.53 mm I.D., 0.5 µm (28263-U)
 oven: 40 °C (5 min.) to 350 °C (20 min. hold) @ 10 °C/min.
 inj.: 250 °C
 det.: 360 °C
 carrier gas: helium
 injection: 1.0 µL, direct on-column

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

Separate Source Standards

This new standards line includes over two dozen Separate Source standards. Separate Source products have identical formulations, but are prepared from independently sourced raw materials, and are independently quality controlled. An example is shown in Figure 1. These standards provide the convenience of working with Supelco as a single vendor while enabling you to meet independent audit requirements of the US EPA.

i Information Request.....1607

**Quality - Built into Every Product**

These new environmental standards are manufactured using the same quality processes and practices that we employ for all of our chemical standard products.

- Only the highest purity raw materials are used; most are 99%.
- Precise gravimetric measurements and volumetric dispensing tolerances no greater than 0.5% of target.
- Gravimetric measurements are made on balances that have been calibrated with NIST traceable weights.
- Quantitative testing is performed by GC or HPLC using a reference batch method.
- Shelf life is determined by both accelerated and real-time stability studies.

STANDARDS ARTICLE

New Standards from Sigma-Aldrich – driving solutions for Analytical Science

Sigma-Aldrich is arguably the world's leading supplier of analytical standards and we continually drive the development of new standards. Based on your feedback we extended our product range to include the new analytical standards presented in the table below.

All standards can be found on the web and on our updated Standards CD, Version 2.0. which is available upon request.

New Standards on Pesticides

Ordering information

Prod No.	Description	Brand
33972	3,5,6-Trichlor-2-pyridinol	Pestanal
33674	Acetamiprid	Pestanal
33863	Bensultap	Pestanal
33875	Boscalid (Nicobifen)	Pestanal
33879	Boscalid-solution, 10 ng/µl in Toluol	Pestanal
33876	Clethodim-solution, 10ng/µl in Toluol	Pestanal
33878	Cyazofamid-solution, 10 ng/µl in Toluol	Pestanal
33968	Diclosulam	Pestanal
33898	Esprocarb-R	Pestanal
33976	Ethiprole	Pestanal
33965	Fenamidon	Pestanal
33872	Fenoxanil	Pestanal
33951	Fentrazamid-Metabolit-solution	Pestanal
33877	Imibenconazole-solution, 10 ng/µl in Toluol	Pestanal
33969	Indoxacarb	Pestanal
33970	Mepanipyrim	Pestanal
33966	Oxadiargyl	Pestanal
33890	PCB No.209-solution, 10 µg/ml in Heptan	Oekanal
33889	PCB No.30-solution 10 µg/ml in Heptan	Oekanal
33891	PCB-Mix nach DIN EN 12766,-solution 10 µg/ml in Heptan	Oekanal
33698	Profoxydim-Li	Pestanal
33985	Propoxycarbazone Na salt	Pestanal
33897	Thiacloprid-amid	Pestanal
33873	Tritosulfuron	Pestanal

New Standards for Clinical, Forensic and Veterinary Analysis

Ordering information

Prod No.	Description	Brand
34010	2-NP-AHD-13C3	Vetranal
34009	2-NP-AMAZ-D5	Vetranal
34008	2-NP-AOZ-D4	Vetranal
34011	2-NP-SCA-13C, 15N2	Vetranal
34006	AHD-13C3	Vetranal
33952	Alcohol standard solution 0,25 mg/ml	Vetranal
33953	Alcohol standard solution 0,3 mg/ml	Vetranal
33957	Alcohol standard solution 0,5 mg/ml	Vetranal
33958	Alcohol standard solution 0,8 mg/ml	Vetranal
33959	Alcohol standard solution 0,9 mg/ml	Vetranal
33961	Alcohol standard solution 1 mg/ml	Vetranal
33962	Alcohol standard solution 2 mg/ml	Vetranal
33963	Alcohol standard solution 3 mg/ml	Vetranal
33964	Alcohol standard solution 4 mg/ml	Vetranal
34019	Carbofuran-D3	Vetranal
33975	CARPROFEN	Vetranal
33989	CEFALEXIN	Vetranal
34001	Ceftiofur hcl	Vetranal
33931	Clioquinol	Vetranal
33988	CLOPIDOL (oder Meticlorpindol)	Vetranal
33973	CLORSULON	Vetranal
33894	Clotrimazole	Vetranal
33874	Cyazofamid	Vetranal
33984	DIFLOXACIN-HCL	Vetranal
34018	Diuron-D6	Vetranal
33993	Doramectin	Vetranal
33981	FEBANTEL	Vetranal
33930	Fleroxacin	Vetranal
34003	HMMNI	Vetranal
34004	Iprnidazol-OH	Vetranal
34017	Isoproturon-D6	Vetranal
34005	m-Chloramphenicol	Vetranal
34007	Metronidazol-OH	Vetranal
33893	Microcystin-Lösung 10 mg/l in Methanol	Vetranal
33899	Norfloxacin	Vetranal
33987	OLAQUINDOX	Vetranal
33895	Piracetam	Vetranal
33979	ROBENIDINE HCL	Vetranal
33967	Sparfloxacin	Vetranal
33864	Tilmicosin	Vetranal
33986	TIOPRONIN	Vetranal

Offer

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Information Request.....1607

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Ascentis RP-Amide is a new generation, highly stable, polar embedded RP phase that provides orthogonal selectivity to C18 phases and increased resolution for analysis of polar compounds.

Ascentis RP-Amide

Ascentis RP-Amide is a new generation ultra low bleed, polar embedded RP phase that provides orthogonal selectivity and increased resolution for HPLC and LC-MS analysis of polar compounds. The Ascentis RP-Amide is the first choice in polar embedded HPLC phases.

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- 100% aqueous compatibility
- Ultra low bleed, LC-MS compatible
- Unique selectivity

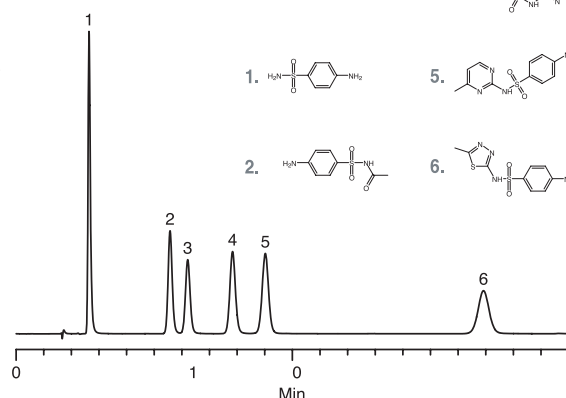
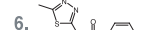
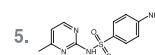
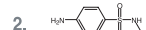
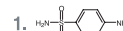
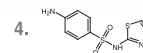
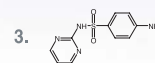
Phase Specifications

USP Code:	Pending
Bonded Phase:	Amido embedded reversed-phase
Endcapped:	Yes
Particle Shape:	Spherical
Particle Purity:	<5 ppm metals
Particle Size:	3µm, 5µm
Pore Size:	100 Å
Surface Area:	450 m ² /g
Carbon Load:	19.5%
pH Range:	2 to 8
Temp. Range:	≤70°C

Ascentis RP-Amide Delivers Excellent Performance in the Analysis of Antibiotic Sulfa Drugs

column: Ascentis RP-Amide, 15 cm x 4.6 mm I.D.,
5 µm particles (565324-U)
mobile phase: 85:15, 1% acetic acid in water:CH₃OH
flow rate: 1.0 ml/min
temp.: 35°C
det.: UV, 254 nm
injection: 10 µL
sample: as indicated in 1% acetic acid in water

1. Sulfanilamide
2. Sulfacetamide
3. Sulfadiazine
4. Sulfathiazole
5. Sulfamerazine
6. Sulfamethizole



Trial Offer

Free, no obligation 30-Day Trial on any Ascentis Amide C16 column.

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