

the Reporter

EUROPE

Volume 19, January 2006 International issue

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New NOGE (Novalac glycidyl ethers) Standards


SIGMA-ALDRICH

EDITORIAL

Analysis of Semivolatiles by US EPA Method 8270D Using Supelco's New Low Bleed GC Column - SLB-5ms

Dear Reader,

When capillary GC was in its infancy, the column was not the limiting factor in determining the limit of detection. However, over the last decade, the other components (autosamplers, injection ports, detectors, data systems) that make up a GC system have become more sophisticated, allowing analysts to achieve a lower and lower limit of detection. Using modern instrumentation, the column may in fact play a prime role in determining the lowest possible limit of detection. For this reason, today's chemists require capillary columns specifically designed to allow analysts to achieve the low detection limits specified by their applications.

New SLB-5ms

SLB columns are designed for GC and GC-MS analysts who require a low bleed, inert, durable, and consistent capillary column for trace analyses. All SLB columns incorporate a combination of unique advances in polymer synthesis, proprietary surface deactivation chemistry, and innovative manufacturing processes.

Varying functionality, no cleanup, quick turn-around times

US EPA Method 8270D for semivolatiles is a formidable challenge for analysts. They must achieve very low detection limits for a large number of analytes, each with varying functionality, in extracts from dirty samples with no cleanup, with quick turn-around times. The column that is used must be up to the task. The new SLB-5ms capillary column was evaluated for use in performing this method. The resulting chromatogram is presented on page 17 in this issue.

I hope you enjoy reading this issue of The Reporter, and can put the information to good use in your laboratory.

Sincerely,



Mike Buchanan
Product Manager, Gas Separations

E-mail: mbuchanan@sial.com

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29th International Symposium on capillary Chromatography.

Palazzo dei Congressi -
Riva Del Garda - Italy
May 29 - June 2, 2006



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HPLC ARTICLE

Separation of the Positional Isomer Quinocide from the Anti-Malarial Drug Primaquine Using a Discovery® HS F5 HPLC Column

I. Brondz and U. Klein Department of Biochemistry, University of Oslo, Oslo, Norway

Introduction

Malaria is one of the most deadly diseases on earth with an estimated death rate of about 2.7 million people per year. It is especially widespread in Africa where the death toll is highest among children. Malaria is caused by the protozoa *Plasmodium vivax* and other *Plasmodium ssp.* The drug primaquine di-phosphate is used for causative treatment of malaria infections. Primaquine (CAS 90-34-6) and its positional isomer quinocide (CAS 525-61-1), are highly toxic substances. Both substances are anti-malarial drugs having a number of side effects.

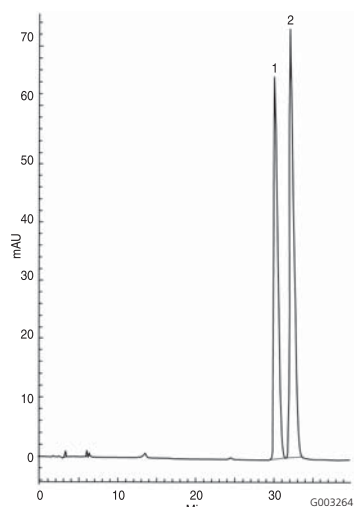
It has previously been assumed that the main contaminant of primaquine is the enantiomer, and a possible separation of the racemate has been reported (1). In some publications, the separation of the positional isomer from primaquine was mistaken as separation of the stereoisomer. Separation of isomers should be supported by MS analysis. We were able to show by using liquid chromatography-mass spectroscopy (LC-MS) that the main contaminant of primaquine is the positional isomer quinocide (2).

The Discovery HS F5 column has unique selectivity for the positional isomers of primaquine and excellent separation characteristics (Figure 1) compared to other stationary phases.

Figure 1. Separation of Quinocide from Primaquine on Discovery HS F5 Column

column: Discovery HS F5, 25 cm x 4.6 mm I.D., 5 µm particles (567517-U)
 mobile phase: 50:50, acetonitrile (34998):20 mM ammonium acetate (238074) in distilled water, pH 7.0
 flow rate: 1.0 ml/min.
 temp.: 20 °C
 det.: 268 nm, reference at 300 nm
 inj.: 20 µl

1. Quinocide
 2. Primaquine

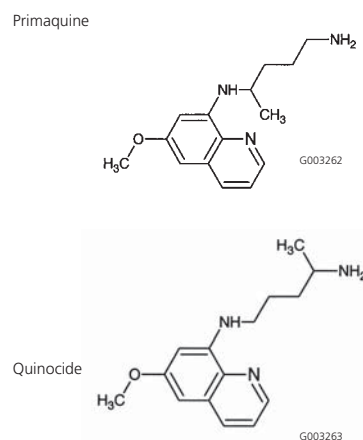


Materials and Methods

For HPLC analysis, an Agilent® 1100 chromatograph with diode array detector (DAD) was used. A Discovery HS F5 column, 25 cm x 4.6 mm I.D., 5 µm particle size was utilized to enhance the separation.

Baseline separation was achieved with isocratic conditions at a

Figure 2. Structures of Primaquine and Quinocide



flow rate of 1.0 ml/min. The mobile phase composition finally chosen was acetonitrile: 20 mM ammonium acetate in distilled water, pH 7.0, 50:50. The analytes were detected at 268 nm with reference at 300 nm. This method resulted in baseline separation of the positional isomer quinocide from the drug primaquine (Figure 1) resulting in a simple and reproducible method.

Conclusions

The anti-malarial drug primaquine is an important human drug, especially in third world countries. Precise quantification of toxic contaminants in this therapeutic agent is therefore of great value. We found that in pharmaceutical samples of primaquine, the concentration of quinocide was as high as 5.1% (2). In the *British Pharmacopoeia* 2000 (3) and in the *European Pharmacopoeia*, 2001, 3rd Supplement (4) related substances are allowed to be present in the drug at a maximum of 3%.

The structure of primaquine and quinocide are shown in Figure 2. The Discovery HS F5 column in isocratic mode gave baseline separation of the positional isomers primaquine and quinocide. Separation of positional isomers is supported by co-chromatography of primaquine and quinocide shown in Figure 1 and by previous LC-MS (2). Baseline separation gives the opportunity to perform precise quantification of quinocide in primaquine. Because the quinocide concentration in primaquine samples tends to be high and quinocide is more toxic and less stable than primaquine, some previously published investigations should be reconsidered.

References

1. R.C. Elderfield, W.J. Gensler, J.D. Head, H.A. Hageman, C.B. Kremer, J.B. Wright, A.D. Holley, B. Williamson, J. Galbreath, L. Wiederhold III, R. Flohardt, S.M. Kupchan, T.A. Williamson and O. Biratein. *J. Am. Chem. Soc.* 1946, 68:1524.
2. I. Brondz, D. Mantzilas, U. Klein, D. Ekeberg, E. Hvattum, M.N. Lebedeva, F.S. Mikhailitsyn, G.D. Soulimanov, J. Roe, *J. Chromatography B*. 2004. 800:211.
3. *British Pharmacopoeia*, 2000. v1:1285-6. 4. *European Pharmacopoeia*, 3rd ed. Supplement 2001. 1323-4.
4. *European Pharmacopoeia*, 3rd ed. Supplement 2001. 1323-4.

For information, please contact
 Wayne Way, wway@sial.com

Did you know?

The Discovery HS F5 bonded phase is one of the most versatile HPLC phases on the market today. In particular, the Discovery HS F5 phase exhibits reversed-phase and normal-phase retention for polar analytes as well as ion-exchange behavior. At low percent organic, retention decreases with increasing organic following reversed-phase behavior. However, at high percent organic, retention increases with increasing percent organic following normal-phase behavior. The result is a "U-shape" retention profile for these compounds.

Moreover, due to the ion-exchange interactions available using Discovery HS F5, the choice and concentration of buffer salts is critical to adequately control retention and separation compared to alkyl phases such as C18. Therefore, use the features of the Discovery HS F5 to your advantage to:

- Improve LC-MS detection by using higher percent organic mobile phase
- Use mobile phase concentration to alter selectivity at high percent organic
- Use buffer choice and concentration to alter selectivity for bases and cations

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Offer limited to 1 unit per customer

Promotional code: U78

Offer valid until 28th of February 2006

Ordering information

Prod No.	Description
567517-U	Discovery HS F5 column, 25 cm x 4.6 mm I.D., 5 μ m

For a complete listing of all Supelco products, log on to our website: sigma-aldrich.com

i Information Request 1901

HPLC ARTICLE

New Ascentis™ C8: Enhanced Hydrophobic Retention and Unique Hydrophilic Selectivity Make it Ideal for Method Development and LC/MS Applications

Introduction

Supelco is pleased to introduce the newest member of our Ascentis line of premiere HPLC columns for small molecule separations: Ascentis C8. This new product expands the user's choice of stationary phase selectivity to optimise chromatographic resolution, and complements the well established C18 and the orthogonally-selective RP-Amide phases. Ascentis C8 is highly reproducible, highly retentive and exhibits enhanced selectivity towards polar compounds. It excels in both highly aqueous and highly organic mobile phases. Like all members of the Ascentis family, Ascentis C8 is available from microbore (1.0 mm I.D.) to preparative (up to 50 mm I. D. on request) dimensions and is especially suited for LC/MS applications.

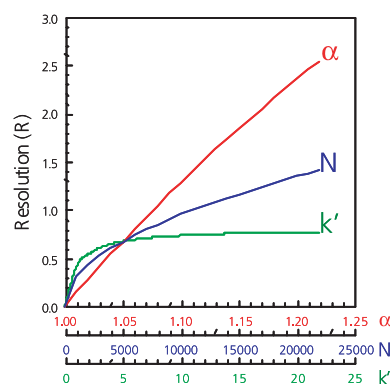
The Ascentis family

The Ascentis family of HPLC columns continues the tradition of innovative yet practical HPLC column technology from Supelco. Ascentis was developed at Supelco's R&D laboratories in Bellefonte, Pennsylvania, U.S.A. using a powerful three-way combination of ultra pure silica, proprietary "Surface-Optimised Technology" and a unique approach to endcapping. The "Surface-Optimised Technology" gives high, yet stable bonded phase coverage. The result is one of the most inert, low-bleed and reproducible HPLC materials available today. Ascentis is ideal for LC/MS applications because it is extremely low bleed and has high reversed-phase retentivity that allows the use of high organic content mobile phases.

Leveraging stationary phase selectivity to improve chromatographic resolution

Resolution in HPLC is governed by column efficiency (N), retention (k or k') and chromatographic selectivity (α). Of these three parameters selectivity has the greatest overall effect on resolution (1). The relationship between selectivity and resolution is nearly linear and does not have a limit as do the relationships between resolution and efficiency or retention (Figure 1).

Figure 1. Effect of selectivity (α) on resolution in HPLC. (from reference 1)



Selectivity is influenced by both the composition of the mobile phase and the stationary phase. With the Ascentis HPLC column product line now comprising three different bonded phase chemistries, optimal resolution can be achieved by using Ascentis

C18, the standard in RP-HPLC, the unique polar-embedded Ascentis RP-Amide, which gives orthogonal selectivity to C18, or the new Ascentis C8 phase.

Materials and methods

The comparison of retention of pentylbenzene between Ascentis C8 and competitive C8 phases was carried out as described by Euerby, *et al* (2). The instrument was a Waters 2690 HPLC system equipped with a 2996 photodiode array detector. Experiments to demonstrate the differences between the three Ascentis phases were performed using 150 x 4.6mm I.D. columns packed with 5µm particles. Solvents were of high purity LC-MS CHROMASOLV® grade and standards were from Sigma-Aldrich (see Product Listing for details). Separation was performed on an Agilent 1100 HP HPLC system with an 1100 multiple wavelength detector. Details are given in the chromatograms.

Enhanced hydrophobic retention of Ascentis™ C8

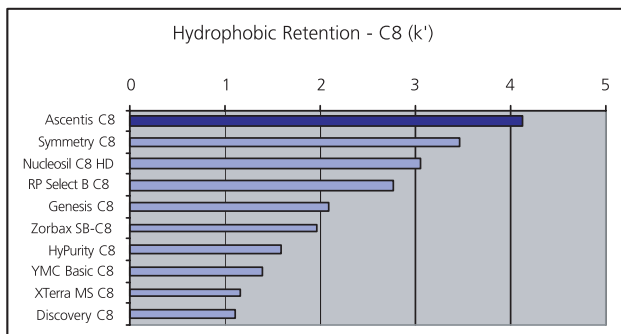
The properties and specifications of Ascentis C8 are given in Table 1. The comparison of Ascentis C8 with competitive C8 phases according to their hydrophobic retention is shown in Figure 2. Capacity factor (k or k') of pentylbenzene was determined as described by Euerby (2) and compared with published data. The results in Figure 2 show that the Ascentis C8 column has the largest capacity factor of the columns tested, indicating its superior hydrophobic retentivity. High capacity is important to HPLC for three reasons. First, within a certain range, increasing k increases resolution (see Figure 1). Second, increased hydrophobic retention permits use of higher organic LC/MS mobile phases that desolvate more rapidly. Third, high capacity means higher sample loads in preparative separations.

Table 1: Properties of and specifications of Ascentis C8

USP Code:	L7
Bonded Phase:	Octyl
Endcapped:	Yes
Particle Shape:	Spherical
Particle Purity:	<5 ppm metals
Particle Size:	3µm, 5µm
Pore Size:	100Å
Surface Area:	450 m ² /g
Carbon Load:	16%
pH Range:	2 to 8
Temp. Range:	≥ 70°C

Figure 2. Comparison of hydrophobic retention on C8 HPLC columns.

column: 150 x 4.6 mm I.D., 5 µm
 mobile phase: (20:80) water:methanol
 temperature: 40°C
 detection: UV at 254 nm



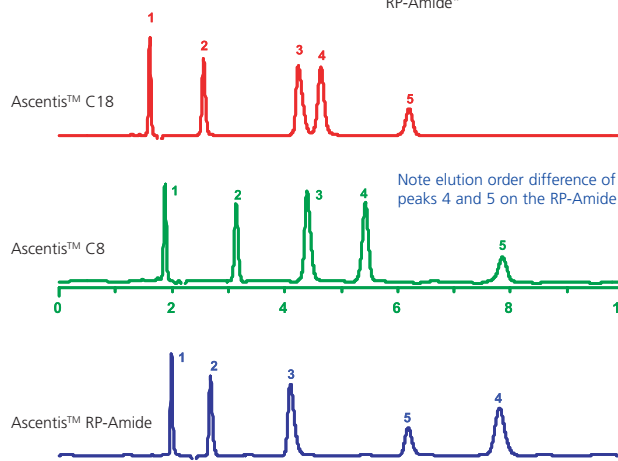
Unique selectivity and retention of polar compounds on Ascentis C8

Besides being unique among C8 phases, Ascentis C8 is also different from the other Ascentis phases. This is demonstrated in the separation of low molecular weight organic acids shown in Figure 3. Three observations are noteworthy. First, all the Ascentis phases gave excellent resolution and peak shape of these often difficult analytes. Second, the different elution patterns show that the Ascentis C8 has different selectivity than the C18 and RP-Amide. The order of elution of the organic acids is the same for Ascentis C8 and C18. However, on the Ascentis RP-Amide column elution order of acrylic acid and fumaric acid is reversed, demonstrating the orthogonal selectivity of the polar embedded RP-Amide phase. Third, the Ascentis C8 is more retentive toward these polar compounds than the C18.

Figure 3. Organic acids demonstrate selectivity differences between Ascentis C18, C8 and RP-Amide phase columns.

column: 150 x 4.6 mm I.D., 5 µm
 mobile phase: (97:3) 0.1% TFA: methanol.
 temperature: ambient
 flow rate: 1 ml/min.
 detection: UV at 220 nm.

"1. L-Tartaric Acid, 2. Lactic Acid, 3. Citric Acid, 4. Fumaric Acid, 5. Acrylic Acid. Elution order of Fumaric Acid and Acrylic Acid has been swapped on the polar embedded phase RP-Amide"



Conclusions

Ascentis C8 is the latest addition to the Ascentis HPLC column product line. Like all Ascentis columns it provides:

- Excellent peak shape for difficult compounds
- Low bleed and high retentivity for LC/MS
- Rugged, reproducible separations

However, Ascentis C8 also provides:

- Increased hydrophobic retention compared to competitive C8 phase
- Different selectivity than Ascentis C18 and RP-Amide phases
- Enhanced retention of hydrophilic compounds compared to C18

The Ascentis family of HPLC columns, which includes the new Ascentis C8, leverages all three variables of the resolution equation, efficiency, retention, but most importantly, selectivity, to give users maximum flexibility in optimizing HPLC separations.

For more information on Ascentis, please call or consult our website: www.sigma-aldrich.com/ascentis

References

1. Zhao, J. H.; Carr, P. W.; *Analytical Chemistry*, 1999, 71(14), 2623-2632.
 2. Euerby, M. R.; Peterson, P.; *J. Chromatogr. A*, 2003, 994, 13-36.

Contact your local sales office. Website: sigma-aldrich.com/supelco

Prod No.	Particle Size (µm)	Length (cm)	I.D. (mm)
Ascentis C8 HPLC Columns			
581412-U	3	5	1.0
581435-U	3	10	1.0
581436-U	3	15	1.0
581400-U	3	5	2.1
581401-U	3	10	2.1
581402-U	3	15	2.1
581403-U	3	3	3
581404-U	3	5	3
581405-U	3	10	3
581406-U	3	5	4.6
581407-U	3	10	4.6
581408-U	3	15	4.6
581420-U	5	5	2.1
581421-U	5	15	2.1
581422-U	5	25	2.1
581423-U	5	5	4.6
581424-U	5	15	4.6
581425-U	5	25	4.6

Ascentis C8 guard columns

581427-U	5	2	4 (kit) ¹
581426-U	5	2	4 (2/pk) replacement cartridges

¹Kit contains holder and one cartridge

Ascentis C18 and RP-Amide HPLC Columns

Ascentis C18²

581324-U	5	15	4.6
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Ascentis RP-Amide²

565324-U	5	15	4.6
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²(Dimensions used in this study. Other dimensions and particles sizes are available. Please call or consult our website: www.sigma-aldrich.com/ascentis)

Prod No.	Description	Pack Size
LC-MS CHROMASOLV® Solvents		
39253	Water	1 l
34978	Water with 0.1% TFA	2.5 l
34966	Methanol	1 l, 2.5 l

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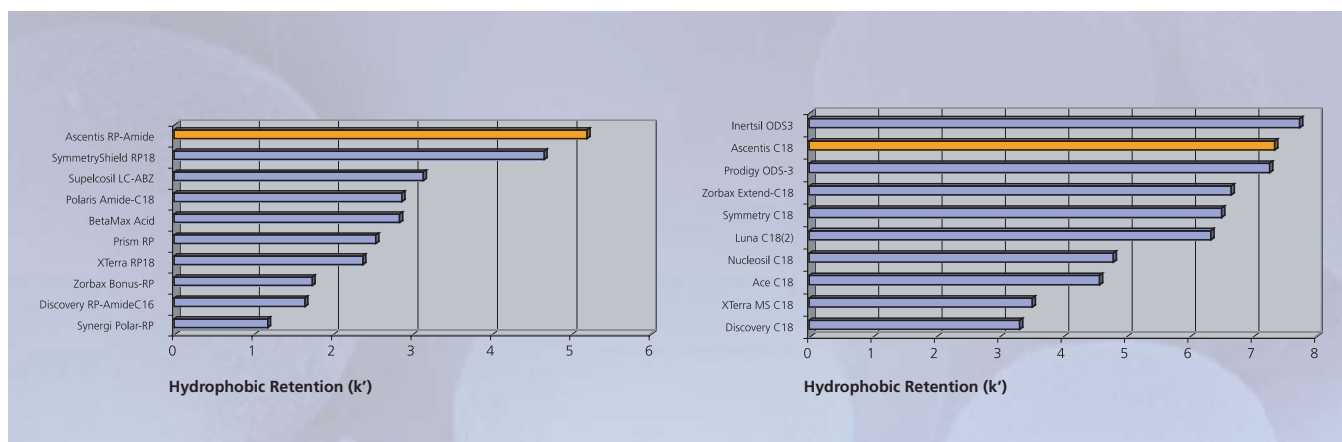
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Promotional code: U89

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Ascentis C8, C18 and RP-Amide for Better Separations!



Hydrophobic Retention

As shown, Ascentis C18, C8 and Ascentis RP-Amide columns are at the top of their class for hydrophobic retention, enabling the user to achieve better separations.

For details on hydrophobic retention see HPLC article on page 4.

¹Ascentis C18 and Ascentis RP-Amide data developed by the Supelco/Sigma-Aldrich Applications Laboratory utilising the Euerby methodology. All other data obtained from Euerby, M. R. and Petersson, P. (2003) J. Chromatogr. A, 994, 13-36.

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 Ascentis, Discovery, SUPELCOSIL - Sigma-Aldrich
 BetaMax, Prism RP-Select B - Merck Genesis - W. R. Grace YMC Basic - YMC, Co.
 - Thermo Electron Corporation
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 Luna, Prodigy, Synergi, Polar-RP - Phenomenex
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- High UV-transmittance for combination with UV and diode array detection
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- Particle tested

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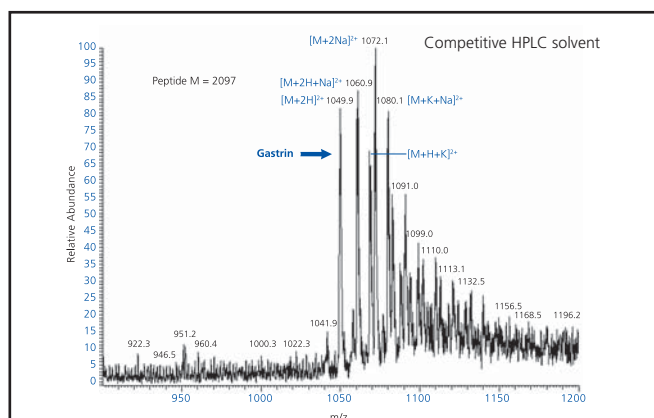
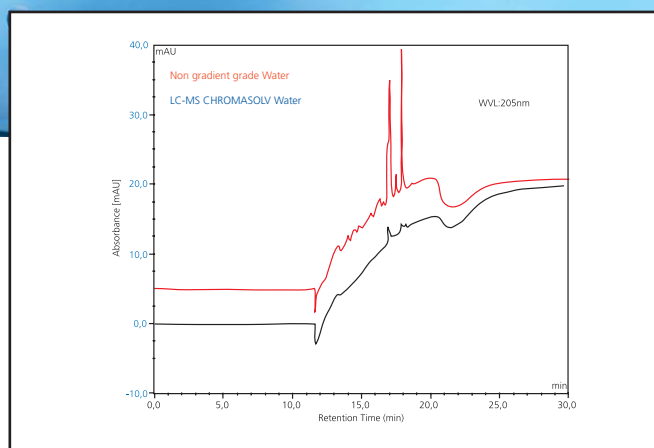
For product information and additional customized solvents, please contact your local office or visit.

www.sigma-aldrich.com/lc-ms-solvents

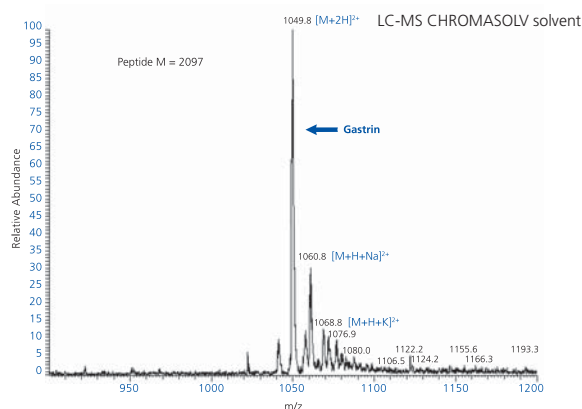
Ordering information

Prod No.	Description	Pack Size
39253	LC-MS CHROMASOLV® Water	1l
34967	LC-MS CHROMASOLV® Acetonitrile	1l / 2.5l
34966	LC-MS CHROMASOLV® Methanol	1l / 2.5l
34965	LC-MS CHROMASOLV® 2-Propanol	1l / 2.5l
34972	LC-MS CHROMASOLV® Ethylacetate	1l / 2.5l

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Cluster ions arising from solvent impurities interfere with sensitive LC-MS analyses



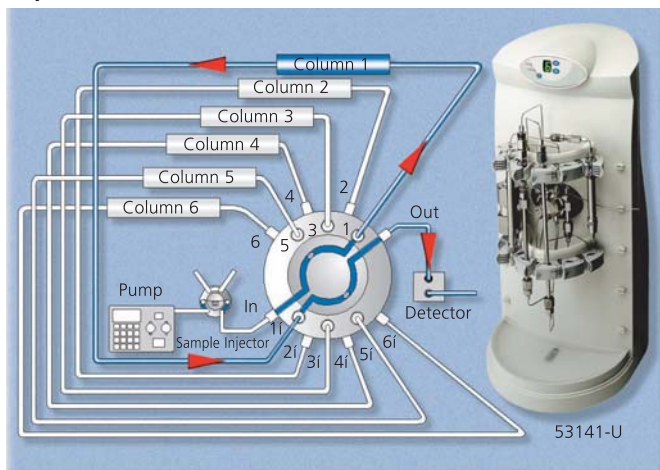
LC-MS CHROMASOLV® solvents are free from metal ions that cause cluster ion formation, giving clean, sensitive LC-MS results every time.

HPLC ARTICLE

Automated Column Switching Facilitates Method Development

SupelPRO™ Automated Fluidics Instruments Complement Method Development on Ascentis™ and Discovery™ HPLC Columns

Supelco's SupelPRO series are precision, electronically controlled, motorised valve instruments for repetitive fluid switching operations. Each SupelPRO instrument is self contained and incorporates a 2-position or multi-position port valve. Standard 2-position models incorporate Level Logic control (type of electrical signal for switching), multi-position models include additionally a 4-line BCD (binary coded decimal) for remote control e.g. by the HPLC instrument. Power requirements: 100-240VAC, 50-60Hz (auto switching). All units are shipped with standard US power cord. Other power cords are available on a custom basis.

SupelPRO 3-Column or 6-Column Selector**Ordering information**

Prod No.	Description
	SupelPRO™ 3-Column or 6-Column Selector
	3-Column
53140-U	Stainless Steel
53142-U	PEEK
	6-Column
53141-U	Stainless Steel
53143-U	PEEK
	SupelPRO 2-Channel Selector with Bypass Valve
53146-U	Stainless Steel
53147-U	PEEK
	SupelPRO 11-Port, 10-Position Valve
53152-U	Stainless Steel
53153-U	PEEK
	SupelPRO 2-Position Valves
	6-Port
53148-U	Stainless Steel
53149-U	PEEK
	10-Port
53150-U	Stainless Steel
53151-U	PEEK
	SupelPRO Solvent Selector Valve
53144-U	1/16"
53145-U	1/8"

TRADEMARKS: Ascentis, Discovery, Supelguard, SupelPRO-Sigma-Aldrich Co. RheoTool, RheoBuild - Rheodyne Corp.

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VersaFlash™

Flash Purification System



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VersaFlash?

Because of its
Versatility!

Versatility *and* simplicity that get results!

With VersaFlash Flash Purification System, one system covers a wide range of sample sizes without the need of additional, expensive equipment. While competitive systems make you change hardware when scaling up or down, VersaPak pre-packed cartridges from 11g to 1900g can be used on the same, easy-to-use system. And VersaFlash has a feature that is not available in competitive products: the symmetrical cartridge design. Our VersaPak cartridges can be combined in series to preload samples, to inject larger sample masses or to combine different cartridge selectivities and increase your resolution options.


VersFlash offers:

- Versatility in sample size
- Versatility in types of samples
- Versatility in sample loading techniques
- Versatility in flow rate and flow direction

VersaFlash provides you full flexibility in your purification processes. And, it can be connected to any existing flash chromatography infrastructure you might already have in your lab.

Please request our new VersaFlash Brochure (T403110B, FWL) for further details or contact your local Sigma-Aldrich office. The complete line of VersaFlash products and accessories can be found on our website:

www.sigma-aldrich.com/versaflash

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 **SUPELCO**

PRODUCT INFORMATION

Supelpak™-2 Resins

The Supelpak-2 series of resins is purified Amberlite® XAD®-2 adsorbent, optimised for various applications, meeting or exceeding the cleanliness needed for the high recovery of specific analytes.

Amberlite XAD-2 resin is a macroreticular, styrene-divinylbenzene copolymer, nonionic bead. Each bead is an agglomeration of microspheres (Figure A) with a sponge-like structure that offers excellent physical and chemical stability. The discrete pores allow rapid mass transfer of analytes, and the 20-60 mesh particle size ensures low back pressure during use. The hydrophobic chemical nature of the resin makes the XAD-2 an excellent adsorbent under reversed phase conditions, such as for the removal of aromatic compounds (non-polar solute) from water (polar solvent).

The Supelpak-2 series of resins has evolved to provide our customers with adsorbents that have lower background levels and increased recovery of specific analytes.

Table 1. Characteristics of Amberlite XAD-2 Resin

chemical nature:	polyaromatic
approximate pore volume:	0.65 ml/g
true wet density:	1.02 g/ml
skeletal density:	1.08 g/ml
mean surface area:	300 m ² /g
mean pore diameter:	90 Å
mesh size:	20-60

Table 2. Application/Analytes for Supelpak-2 Media

Media	Application/Analytes
Supelpak-2	polynuclear aromatic hydrocarbons (PAHs) and dioxins
Supelpak-2SV	phenols such as pentachlorophenol and dinitrophenols
Supelpak-2B	polychlorinated biphenyls (PCBs) in water

50% OFF

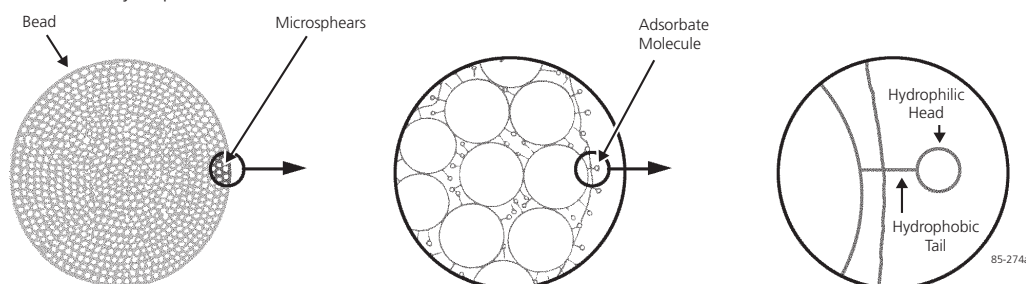
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Figure A. Structure of Hydrophobic Macroreticular Resin Bead



Supelpak-2

A dry, purified form of XAD-2, this adsorbent is cleaned in accordance with, and meeting the purity criteria of US Environmental Protection Agency SW-846, Method 0010. Supelpak-2 is suitable for airborne semivolatile analysis, particularly polynuclear aromatic hydrocarbons (PAHs) and dioxins, stack gases from incinerator stationary sources, as well as ambient and indoor air monitoring. Supelpak-2 is recommended for many air analysis methods, including:

- US EPA SW-846, 0010 (modified Method 5 Sampling Train)
- US EPA SW-846, 0020 (Source Assessment Sampling System)
- TO13 (PAHs in ambient air)
- IP-7 (PAHs in indoor air)

Supelpak-2SV

A dry, more highly purified version of XAD-2, this adsorbent has been specially cleaned for the extraction and recovery of semivolatile compounds from environmental samples. This improvement results in enhanced recoveries of difficult compounds, such as pentachlorophenol and dinitrophenols, leading to increased detection limits. For some laboratory situations, the Supelpak-2SV background levels are low enough to eliminate additional cleaning steps, saving time and money.

Supelpak-2B

A wet, high purity version of XAD-2, this adsorbent meets EPA requirements for the collection of hydrophobic organic compounds (HOCs) from water, and is highly recommended for

determining polychlorinated biphenyls (PCBs) in water according to the Great Lakes National Program Office (GLNPO).

Custom Resin Capabilities

As a leading supplier of specialized adsorbent resins, Supelco has technologies to process resins to almost any specifications. We are able and willing to fulfill your custom adsorbent and resin purification needs. Simply contact our Technical Service chemists with your questions at EurTechServ@europe.sial.com.

Ordering information

Prod No.	Description	Pack Size
20275	Amberlite XAD-2	100 g
10357		500 g
20279	Supelpak-2	100 g
21130-U		1000 g
13670	Supelpak-2B	100g
13673-U	Supelpak-2SV	100 g
13682-U		250 g
13674-U		1000 g

Trademarks

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SPE ARTICLE

NEW Custom “Dispersive” SPE Products for Pesticide and Acrylamide Analysis An Trinh atrinh@sial.com

The multi-residue surveillance of pesticides in agricultural products (fruits, vegetables, meat, shellfish, grains, and dairy products) is an ongoing project for regulatory agencies, contract laboratories, and industrial laboratories worldwide. Hundreds of thousands of samples are analyzed annually to meet a variety of purposes including regulatory enforcement and surveillance monitoring (1).

In previous issues of the Supelco Reporter we discussed the use of dual-layer SPE technology (ENVI-Carb™-II/PSA SPE) to provide the necessary sample cleanup for analysing pesticides in agricultural matrices for subsequent GC-MS analysis.

In this report, we discuss the utility of a new custom service Supelco offers to support an emerging sample prep technique called “Dispersive SPE.”

Dispersive SPE for Multi Residue Pesticide Analysis

In recent years, a number of SPE procedures for multi-residue pesticide analysis have been published. Most of them involve an initial liquid-liquid extraction step, and/or solid-liquid extraction step using a water miscible solvent such as acetonitrile or acetone. Subsequent analysis is conducted via GC-MS, GC-fluorescence, GC-ESD, and other detection techniques. Further cleanup using SPE technology prior to GC analysis is necessary to decrease background levels for trace level pesticide detection, reduce matrix-induced signal enhancement, and relieve stress and reduce downtime on the GC system (2).

Most of the published SPE procedures for multi-residue monitoring (MRM) involved the use of SPE tubes to conduct sample cleanup. In 2003, Anastasiades et al. introduced a novel approach to SPE sample prep called “dispersive SPE” (3). In this method 10-20 g of a food sample is initially extracted with acetonitrile. Gram levels of salt (magnesium sulfate, sodium chloride, and/or sodium sulfate) are then added to drive partitioning between the aqueous residues and the acetonitrile layer. A small aliquot of the acetonitrile layer is removed for further SPE cleanup. Unlike most methods using conventional SPE tubes, in dispersive SPE, removal of residual water and cleanup are performed simultaneously by mixing bulk SPE and magnesium sulfate with the acetonitrile extract. The bulk SPE sorbent adsorbs matrix interferences, and after a simple vortex and centrifugation step, the supernatant is ready for further analysis using GC-MS or LC-MS.

In a follow-up study, Lehotay et al. demonstrated the method's effectiveness by extracting over 200 pesticides in a variety of matrices including lettuce and oranges for both GC-MS and LC-MS-MS analysis (4), and the use of buffering during the initial pre-SPE extraction process to stabilise base-sensitive pesticides (5). The final method (Table 1) was further validated in an inter-laboratory trial involving 15 laboratories in 7 countries (6). The method was termed the QuEChERS method which is short for “quick, easy, cheap, effective, rugged, and safe”.

Table 1. Inter-Laboratory Dispersive SPE Procedure for Pesticide Residues in Fruits and Vegetables**Solid-Liquid Extraction**

1. Transfer 15 homogenized food sample to 50 ml PTFE tube
2. Add 15 ml 1% acetic acid in acetonitrile + 1.5 g anh. NaAc + 6 g anh. magnesium sulfate + 75 µl I.S. solution
3. Shake vigorously 1 min.; Centrifuge > 1500 rcf 1 min.

Dispersive SPE

1. Transfer 1-8 ml of acetonitrile layer to clean tube with 150 mg anh. magnesium sulfate + 50 mg PSA per ml extract and shake for 30 sec.
2. Centrifuge > 1500 rcf for 1 min.
3. Transfer supernatant to GC vial or LC vial for concurrent LC-MS and GC-MS analysis. Note that further processing may be necessary prior to chromatographic analysis (e.g. addition of formic acid for LC-MS analysis; or evaporation of supernatant and reconstitute with toluene for GC-MS analysis).

Dispersive SPE-Acrylamide Analysis in Starchy Foods

Numerous reports have been published for the extraction and analysis of acrylamide in starchy foods (e.g. potato chips). Most of which require a combination of SPE tubes of varying chemistries to provide adequate sample cleanup, and/or sample bromination to decrease acrylamide's polarity for improved chromatographic retention. In 2004, Mastovska et al. (7) introduced a fast and easy dispersive SPE method for the analysis of acrylamide in foods. In this protocol, food samples are initially extracted with a combination of hexane, water, acetonitrile, and high levels of salts (magnesium sulfate + sodium chloride). Most of the problematic fats in fried foods migrate to the hexane layer which is discarded. The high salt environment aids separation between the acetonitrile and water layers, and also drives acrylamide to the acetonitrile layer. An aliquot of the acetonitrile layer is further cleaned up with PSA (primary secondary amine) SPE under dispersive conditions. PSA removes residual fatty acids and other interferences remaining in the extract. The detailed protocol is described in Table 2.

Table 2. Dispersive SPE Procedure for Acrylamide Analysis**Solid-Liquid Extraction**

1. Add d3-acrylamide at 500 ng/g to 1 g of homogenized sample
2. Add 5 ml hexane and vortex
3. Add 10 ml DI water + 10 ml acetonitrile + 4 g magnesium sulfate + 0.5 g sodium chloride
4. Vortex 1 min. and centrifuge at 3450 rcf for 5 min.
5. Discard hexane layer

Dispersive SPE

1. Combine 1 ml of acetonitrile layer with 50 mg PSA + 150 mg magnesium sulfate
2. Vortex for 30 sec.
3. Centrifuge at 3450 rcf for 1 min. 4. Analyze supernatant via LC-MS-MS or GC-MS analysis

Custom SPE Products for Dispersive SPE

We have described just a few application examples of dispersive SPE. Although the technique offers some advantages over conventional methods, it requires the analyst to weigh a pre-determined amount of salts and SPE sorbent(s) for each sample. The weighing process could be a time consuming bottleneck for larger studies when greater throughput is required.

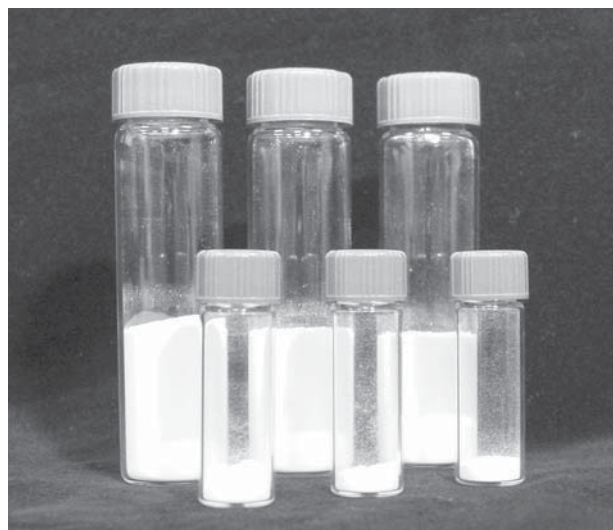
To address this issue, Supelco now offers a new custom product service to support this emerging sample prep technique. In this service, we can provide the necessary pre-weighed SPE sorbents and salts for a given method in a convenient screw top vial format(s). The analyst can either perform the extraction in the vials itself, or combine the contents with the sample at the appropriate step during the procedure. For example, to support the method described in Table 1, we can provide two vials (e.g. 10-50/pk) containing the following contents:

Vial 1: 6 g anhydrous magnesium sulfate + 1.5 g anhydrous sodium acetate

Vial 2: 150 mg anhydrous magnesium sulfate + 50 mg PSA

Table 3 lists the available sorbents and salts commonly used in dispersive SPE. Figure 1 is a visual depiction of custom pre-weighed sorbents and salts for dispersive SPE.

Figure 1. Example of Custom Pre-Weighed Salt/Sorbent Vials for Dispersive SPE



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Table 3. Available SPE Sorbents and Salts Commonly used in Dispersive SPE¹

Florisil (57209)	C18 SPE (52600-U)
NH2 SPE (57212-U)	PSA SPE (52738-U)
SAX SPE (57214-U)	ENVI-Carb (graphitized carbon black) (57210-U)
Sodium acetate (24,124-5)	Magnesium sulfate (23,039-1)
Sodium sulfate (23,859-7)	Sodium chloride (S 9888)

¹ Catalogue numbers in parentheses are for bulk quantities of 100 g or greater.

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- S.J. Lehotay, Interlaboratory Validation of the QuEChERS Method to Analyze Pesticide Residues in Fruits and Vegetables, Proceedings AOAC Annual Meeting, St. Louis, MO USA (2004).
- K. Mastovska, S.J. Lehotay, Development of a Fast and Easy Method for Analysis of Acrylamide in Various Food Matrices, Proceedings AOAC Annual Meeting, St. Louis, MO USA (2004)

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SPME ARTICLE

New Developments in Dioxin Sampling and Analysis Daniel S. Vitkuske dvitkuske@sial.com

Dioxins and furans are by-products of many industrial processes, including incinerator stack emissions, flyash, and paper bleaching. Polychlorinated dibenzo-p-dioxins (PCDD's), polychlorinated dibenzofurans (PCDF's) and associated dioxin-like polychlorinated biphenyls (PCB's), which are collectively called dioxins, represent classes of compounds that exhibit potential risks for human health. The extreme toxicity of these compounds has made their analysis in environmental samples increasingly important.

Supelco has a wide range of innovative products ideal for the sampling and analysis of dioxins and PCB's including:

- Dioxin Prep system for sample cleanup and isolation
- Dioxin SPME fibers for sample preparation
- GC columns for dioxin analysis

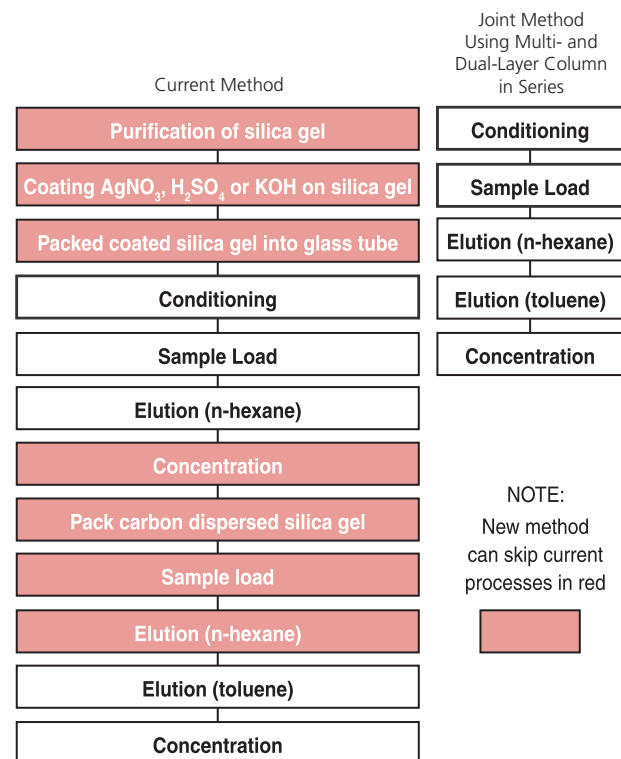
Dioxin Prep System

Currently there are several methods (USA EPA Method 23 or 1613B; European Method EN-1948) for the quantitative analysis of dioxin isomers, which involve successive clean-up steps on various chromatographic adsorbents (multi-layer silica, Florisil®, alumina, activated carbon) that are very time consuming and labor intensive. The Supelco Dioxin Prep system provides a much more efficient means of extracting and isolating dioxins, furans, and coplanar PCB's from stack gases, wastewater, soil, food, blood, and milk. The Dioxin Prep system design reduces the number of steps in the cleanup process thereby decreasing prep time by 1 day, reducing solvent usage, and resulting in extraction recoveries greater than 85%.

The convenient multi-layer silica gel column is key to the extraction process. Seven layers of treated silica oxidize and reduce polar interferences. The analytes (coplanar PCB/PCDD/PCDF's) in the extract will pass through the multi-layer column with minimal retention while interferences and non-target contamination from the extraction will be trapped and retained on the column. The analytes can then be collected in the n-hexane eluate for additional processing by a rotary evaporator or Kuderna-Danish concentrator, or another suitable concentration method.

Alternatively, a unique dual-layer carbon reversible tube can be used to isolate the PCB's, dioxins, and furan groups. Isolation and separation is based on the two layers of carbon having different affinities for such compounds. Figure 1 shows how the multi-layer column and dual-layer column reduces the number of steps for the extraction from 12 to 5.

Figure 1. Comparison of the Current Method versus the New Joint Method Using Multi- and Dual-Layer Column



Dioxin SPME Fiber

Traditionally, dioxin analysis has been conducted using ELISA analysis. More recently Supelco together with the Japan Quality Assurance Organisation have developed a new rapid, inexpensive and accurate dioxin extraction and analysis procedure using Solid Phase Micro Extraction (SPME) coupled with HRGC-HRMS. The method uses carbon coated SPME fibers without the complicated sample cleanup steps that are currently used for conventional dioxin analytical methods. Sample preparation in the analysis of co-planar PCB's/ PCDD's/ PCDF's often includes a classification procedure to separate similar compounds, which the SPME Dioxin fibers are also able to achieve.

SPME fibers coated with carbon material can selectively extract PCDD's/PCDF's and co-planar PCB's from organic solvents such as n-nonane and can introduce a large amount of PCDD's/PCDF's and co-planar PCB's into the GC without introducing a large amount of solvent.

Supelco SP-2331 GC Columns

There are 75 polychlorinated dibenzo-p-dioxin (PCDD) and 135 polychlorinated dibenzofuran (PCDF) compounds, each with a varying degree of toxicity. Of these 210 different compounds, the 17 compounds with chlorine substitution in the 2,3,7,8-positions have the highest degree of toxicity. The compound 2,3,7,8-Tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) is considered the most toxic. Therefore other 16 toxic compounds have their toxicity compared to 2,3,7,8-TCDD using toxic equivalency factors (TEFs).

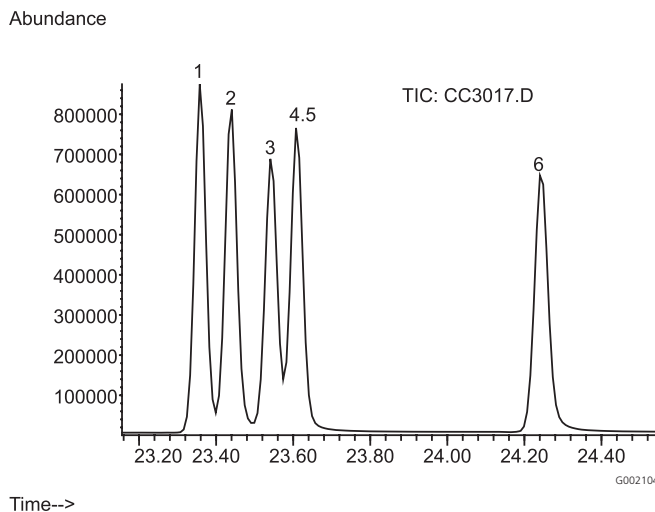
There are several US EPA test methods (1613, 8280, 8290, DLM02.0) that exist for the detection and quantification of the 17 PCDD and PCDF compounds with chlorine substitution in the 2,3,7,8-positions. Our SP-2331 capillary GC column has been specifically designed for the separation of these 17 toxic compounds from each other as well as from the other 193 compounds. Figure 2 shows a chromatogram of TCDD congeners on a SP-2331 column.

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Figure 2. Separation of TCDD Isomers on SP-2331

column: 60 m x 0.25 mm I.D., 0.20 µm (24104-U)
 oven: 170 °C (1 min.), 8 °C/min. to 265 °C
 inj.: 250 °C
 MSD interface: 265 °C
 scan range: SIM
 carrier gas: helium, 37 cm/sec constant
 injection: 1 µl, splitless (1 min.)
 liner: 4 mm I.D., single taper
 sample: 1.5 µg/ml TCDD standard in dodecane

1. 1,4,7,8-TCDD
2. 2,3,7,8-TCDD
3. 1,2,3,4-TCDD
4. 1,2,3,7-TCDD
5. 1,2,3,8-TCDD
6. 1,2,7,8-TCDD



SPME

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Solid Phase Microextraction (SPME) Application Guide CD

The 5th Edition CD includes new references, literature and applications. The searchable CD format includes a large number of application references organized by analyte and matrix, and helps you choose the right SPME fiber for your particular sample preparation need. All applications give complete SPME and chromatographic conditions. The CD also contains all current Supelco SPME literature, including the Troubleshooting Guide and Guide to Quantification with SPME.

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Solvents for residue analysis of dioxins, furans and PCB's

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1,2,3,7,8-penta-CDD	2,3,4,7,8-penta-CDF
1,2,3,4,7,8-hexa-CDD	1,2,3,7,8/1,2,3,4,8-penta-CDF
1,2,3,6,7,8-hexa-CDD	1,2,3,4,7,8/1,2,3,4,7,9-hexa-CDF
1,2,3,7,8,9-hexa-CDD	1,2,3,6,7,8-hexa-CDF
1,2,3,4,6,7,8-hepta-CDD	1,2,3,7,8,9-hexa-CDF
Octa-CDD	2,3,4,6,7,8-hexa-CDF
-	11,2,3,4,6,7,8-hepta-CDF
-	1,2,3,4,7,8,9-hepta-CDF
-	Octa-CDF

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Prod No.	Prod. Name	Description	Pack Size
34410	Acetone	ENVISOLV™ for analysis of dioxins, furans and PCB, ≥99.8% (GC)	2.5 l / 7 l
34411	Dichloromethane	ENVISOLV™ for analysis of dioxins, furans and PCB, ≥99.8% (GC)	2.5 l / 7 l
34412	Hexane	ENVISOLV™ for analysis of dioxins, furans and PCB, ≥95% (GC)	2.5 l / 7 l
34413	Toluene	ENVISOLV™ for analysis of dioxins, furans and PCB, ≥99.7% (GC)	2.5 l / 7 l



SIGMA-ALDRICH

GC ARTICLE

New Supelco Low Bleed Supelco SLB-5ms Columns for US EPA Semivolatile Methods

Katherine Stenerson kstenerson@sial.com

US EPA Methods 525, 625, 8270, and CLP use GC-MS to determine the levels of a wide variety of organic contaminants in various matrices. The low detection levels and varying functionality of the compounds of interest can make these extremely challenging applications. In addition, the method requires the use of a mass spectrometric detector (MSD). This type of detector, while providing invaluable information with regards to compound structure, is susceptible to fouling from dirty samples and column bleed. If the fouling is severe enough, the detector must be dismantled and cleaned, a procedure that

usually removes an instrument from service for one or more days. For this reason, the capillary column used, in addition to being inert towards the various analytes in the method, must exhibit a very low level of bleed. Column bleed cannot only foul an MS source, it can raise detection levels and interfere with spectral identification.

The new SLB-5ms capillary column was evaluated for use in performing US EPA Method 8270. A mixture of 74 analytes, eight surrogates, and six internal standards was analyzed.

Figure 1. 5 ng Semivolatiles Standard (with Internal Standards at 50 ng) on the new Supelco SLB-5ms Capillary Column

column: SLB-5ms 30 m x 0.25 mm I.D. x 0.25 μm (28471-U)
 oven: 40 °C (3 min.), 20 °C/min. to 100 °C, 10 °C/min. to 200 °C, 30 °C/min. to 325 °C (5 min.)
 inj.: 250 °C
 det.: MSD, 325 °C, scan range 45-450 amu
 carrier: He, 20 psi (0 min.), ramp to 80 psi (0 min.), ramp to 16.5 psi (3 min.), ramp to 25 psi
 injection: 1 μl, splitless
 liner: 4 mm I.D. single taper
 sample: 5 ng on-column of a 74-component semivolatile standard plus 8 surrogates, and 6 internal standards at 50 ng

1. 2,4-Dinitrophenol
2. Pentachlorophenol
3. Benzidine

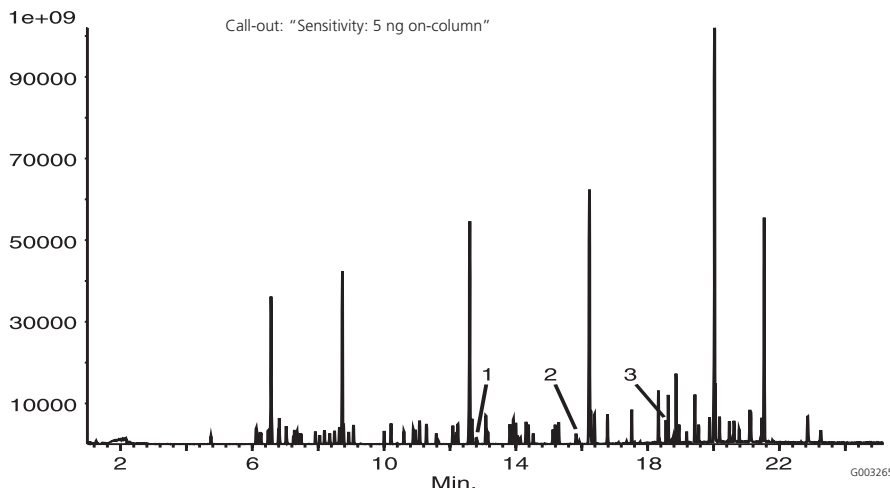
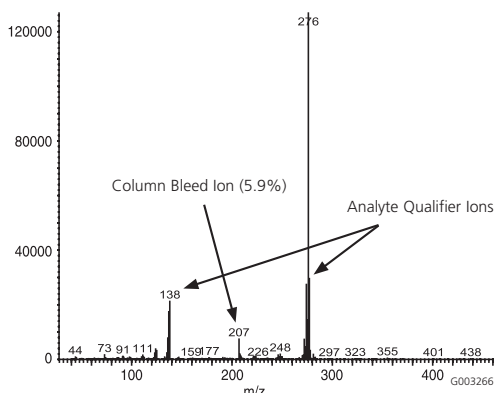


Figure 2. MS Spectra of benzo(g,h,i)perylene, 5 ng, 325 °C; Supelco SLB-5ms column



Column bleed was low enough to facilitate detection and good spectral quality at 5 ng on-column. A chromatogram of the 5 ng standard is presented in Figure 1. Using the run conditions described in Figure 1, analysis was completed in just under 24 minutes. Good response was observed for all analytes, including the difficult to analyze pentachlorophenol, 2,4-dinitrophenol, and benzidine.

As a measure of bleed, the spectrum of the last eluting peak, benzo(g,h,i)perylene, was examined at the 5 ng level (Figure 2). This peak elutes during the isothermal hold at the maximum oven temperature of the analysis. The major bleed ion, $m/z=207$, resulting from the formation of cyclic hexamethylcyclotrisiloxane (D3), is present at a level lower than $m/z=138$ and $m/z=277$, two qualifier ions (intensity of $m/z=207$ indicated as % of $m/z=276$) used in helping to confirm the

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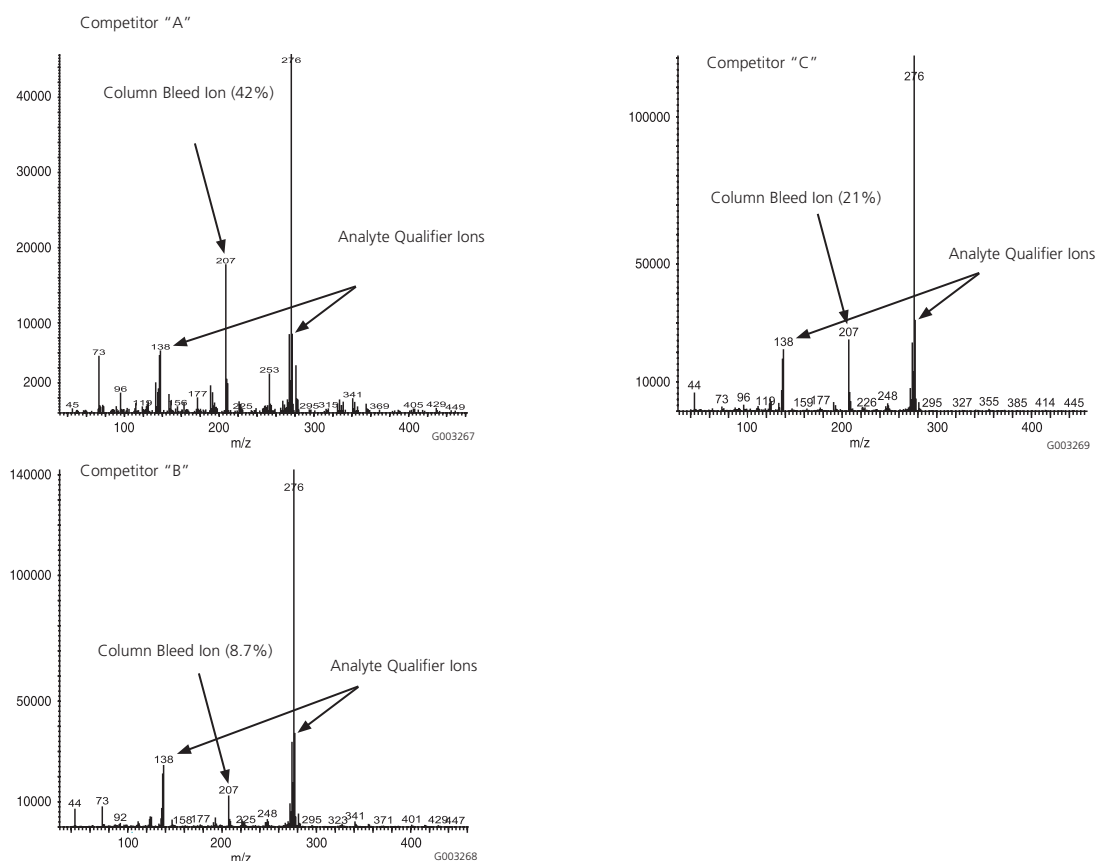
identity of the peak. Bleed was measured in this manner on three additional competitors' modern 5-type GC/MS columns. The results are presented in Figure 3. The bleed from the Competitor "B" column was the closest to the SLB-5ms, however it was not as low. The bleed from the Competitor "A" column was the highest, with the bleed ion actually larger in size than the two qualifier ions.

Unique advances in Supelco's polymer chemistry and column manufacturing procedures have allowed us to offer the SLB-5ms, a column with consistently low MS bleed and good analyte response resulting in lower detection levels and minimising contamination of the MS detector. These attributes make it the top selection for the analyses of semivolatiles by US EPA Method 8270.

Ordering information

Prod No.	Description	Length (m)	D _i (μm)	BETA
0.20 mm ID				
28513-U		30	0.2	250
0.25 mm ID				
28469-U		15	0.25	250
28471-U		30	0.25	250
28472-U		60	0.25	250
28473-U		30	0.5	125
28476-U		30	1	63
0.32 mm ID				
28557-U		15	0.25	320
28482-U		30	0.25	320
28484-U		30	0.5	160
28487-U		30	1	80

Figure 3. MS Spectra of benzo(g,h,i) perylene, 5 ng, 325 °C; 5-Type GC/MS Columns from Competitors "A", "B", and "C"



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Accessory items are for Agilent Technologies instrument models: 4890, 5880, 5890, 6890.

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1. INLET LINERS	Agilent X-Ref	Pk/5	Pk/25
Split Injection			
78.5x6.3 mm, 4 mm ID, wool packed	19251-60540	2048605	2048625
78.5x6.3 mm, cup design	18740-80190	2051005	2051025
78.5x6.3 mm, cup design, wool packed	18740-80190	2048205	2048225
Splitless			
78.5x6.5 mm, tapered	5781-3316	2046605	2046625
78.5x6.5 mm, tapered, wool packed	5062-3587	2047805	2047825
78.5x6.5 mm, 2 mm ID	5181-8818	2051305	2051325
78.5x6.5 mm, dual tapered	5181-3315	2048505	2048525
Direct/Wide-Bore			
78.5x6.5 mm, 1.5 mm ID	18740-80200	2051705	2051725
78.5x6.3 mm, 0.75 mm ID for SPME*		2637505	2637525
2. SEPTA		Pk/50	
Thermogreen LB-2, 11.0 mm (7/16")	5183-4757	20654	
Thermogreen LB-2, 9.5 mm (3/8")	5181-1283	20652	
3. FERRULES		Pk/10	Pk/50
GC Inlet - Supeltext M-4 Graphite			
0.20-0.25 mm Column ID	5080-8853	24811-U	24819-U
0.32 mm Column ID	5080-8853	24809-U	24813-U
0.53 mm Column ID	5080-8773	24808-U	24812-U
GC Inlet - Supeltext M-2A Vespel-Graphite			
0.20-0.25 mm Column ID	5181-3323	24803-U	24807-U
0.32 mm Column ID	5062-3514	24802-U	24806-U
0.53 mm Column ID	5062-3512	24801-U	24804-U
GC/MS Interface - Supeltext M-2A Vespel-Graphite			
0.20-0.25 mm Column ID	5062-3508	24826-U	28022-U
0.32 mm Column ID	5062-3506	24824-U	28023-U
0.53 mm Column ID	5062-3538	24823-U	28024-U
4. INLET SEALS		Pk/2	Pk/10
Gold-Plated	18740-20885	23318-U	23319-U
Stainless-Steel	18740-80220	23316-U	23317-U
5. O-RING SEALS			Pk/25
Therm-O-Ring	5180-4182		21004-U
6. COLUMN NUTS		Pk/2	Pk/5
Column Nut for 1/16" Ferrules	5181-8830	24833-U	
Brass Nut for GC/MS Interface			28034-U
7. MSD NUTS		Pk/2	
MSD Source Nuts for Agilent Technologies		22517	
CAP KIT CASE			
GC Accessory Case for Agilent Technologies		28035-U	

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20638	5.0		50
20651	6.0	1/4	50
28021-U	9.0	11/32	50
20652	9.5 ¹	3/8	50
20666	9.5 ¹	3/8	250
20677	9.5 ¹	3/8	1000
20653-U	10.0	13/32	50
23156	10.0	13/32	250
23157	10.0	13/32	1000
20654	11.0	7/16	50
23163	11.0	7/16	250
23164	11.0	7/16	1000
23154	11.5	11/24	50

Ordering information

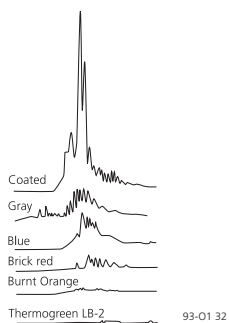
Thermogreen LB-2 Septa

Prod No.	Diameter (mm)	Diameter (In.)	Qty.
20660-U	12.5	1/2	50
20678	12.5	1/2	250
20662-U	14.0	9/16	50
20663	16.0	5/8	50
23159	17.0	21/32	50
Cylindrical, for Shimadzu instruments, for Shimadzu instruments			
20608	Plug Type		10
20633	Plug Type		50
Drilled, for Solid Phase Microextraction			
23161	9.5 ¹	3/8	25
23162-U	9.5 ¹	3/8	50
23167	11.0	7/16	25
23168	11.0	7/16	50

¹ We recommend a 9.5mm (3/8") septum to those who previously used the 9mm size.

Bleed after 25 punctures

Temperature: 40 °C to 250 °C
Injector Set Point: 350 °C



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- Easier needle penetration and high puncture tolerance, ideal for autosamplers
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STANDARDS ARTICLE

Detection, Measurement and Regulation of NOGE (Novolac Glycidyl Ethers) and Other Packaging-Derived Epoxy Compounds In Food Products By Rainer Walz**Analytical chemistry in food science**

One of the most important areas of analytical chemistry is in food preparation, packaging, safety, regulatory compliance and related industries. Sigma-Aldrich has been a consistent and innovative supplier of standards, reagents, sample prep and chromatographic products for food analysis. This article discusses the background behind the recent addition of NOGE standards to our product portfolio.

Epoxy compound residues in food packaging

Many foods and food preservatives are acidic or have high salt concentration, naturally or to inhibit microbial growth. Both conditions are harsh on metal containers. To prevent corrosion, metal cans are usually coated on their interior surfaces. The so-called "organosol" coatings, of which PVC polymer forms the backbone, were developed to provide a flexible internal coating to prevent corrosion of metal cans. However, upon curing PVC produces HCl which can degrade the polymer coating. Epoxy compounds are added to the PVC polymer to act as HCl scavengers. One of the first such compounds used for this purpose is bisphenol-A-diglycidyl ether (BADGE). When analysts turned their attention to the fate of BADGE in the food, they found that the majority of the BADGE migrated from the coating and disappeared, presumably reacting with components of the food, a great cause for concern. Food packaging chemists, looking for alternatives to BADGE, began using the Novolac glycidyl ethers (NOGE). These, too, were found to migrate into the foods, but to different degrees in different foods compared to BADGE.

NOGE chemistry: A complex mixture of isomers and oligomers

Novolac is the technical name for complex mixtures obtained by reaction of phenol with formaldehyde under acidic conditions. When Novolac is reacted with epichlorohydrin, HCl is eliminated forming Novolac glycidyl ethers (NOGE). An important difference between NOGE and BADGE is the number of reaction products that are formed during the reaction between phenol and acetone or formaldehyde. Acetone reacts with phenol only at the para-position forming bisphenol-A, a single compound that is the precursor to BADGE. However, formaldehyde can react with phenol at the para and the two ortho sites creating three bisphenol-F isomers, the precursors to NOGE. Besides the three possible 2-ring bisphenol-F isomers, up to three phenols can be bonded to each phenol enabling oligomerization through the methylene groups. The result is that NOGE is a complex mixture of 2-, 3-, 4-, 5- and 6-ringed (and greater) compounds with various mean molecular weights. The three 2-ring NOGE compounds are called BFDGE (bisphenol-F-diglycidyl ether). An example of the formation of a 2-ring NOGE compound from bisphenol-F and epichlorohydrin appears in Figure 1.

NOGE regulations

Although BADGE and NOGE (which includes BFDGE) have been used as additives for organosol coatings of metal cans, neither has been approved for this application. The European Food Safety Authority (EFSA) concluded that BADGE and its reaction products do not raise carcinogenicity and genotoxicity

Figure 1. Example of the reaction of epichlorohydrin with a bisphenol-F isomer to form a 2-ring NOGE compound. NOGE compounds are a complex mixture containing 2-, 3-, 4-, 5- and 6-ring (and higher) structures. (Note: BFDGE cannot be directly produced as a pure compound.)

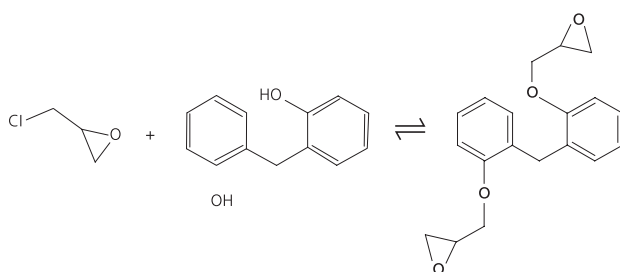
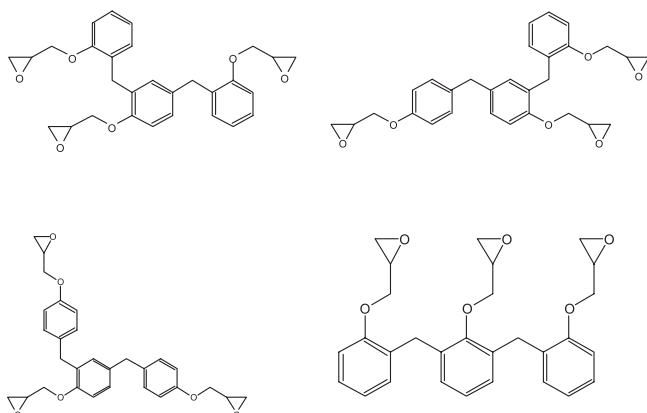


Figure 2. 3-Ring NOGE Structures

4 of 7 NOGE 3-Ring Isomers



concerns, and has allocated a Tolerable Daily Intake for BADGE of 15 mg/kg per day. On the basis of the EFSA conclusions, draft Commission regulations would permit the continued use of BADGE subject to a specific migration limit of 9mg/kg of food or food simulants. However, the verdicts for BFDGE and NOGE were not as good. Because of structural indicators for toxic effects (epoxy groups and potential chlorohydrin formation) and the lack of both toxicological data and reliable analytical methods for their identification and quantification, the regulations banned the use of these substances in all but large food containers beginning January 1, 2005.

HPLC: addressing the analytical challenge of NOGE measurement

Two of the analytical challenges of NOGE analysis are (1) the extreme complexity of NOGE samples, and (2) previous lack of reliable analytical standards. Regarding the sample

complexity, for 3-ring NOGES alone there are 135 possible NOGE compounds that are derived from the seven positional phenolic isomers. Considering there are 27 isomers of the 4-ring systems and the many more with the 5- and 6-ring systems, one can see how quickly the mixtures become extremely complex. Examples of some of the possible structures of a 3-ring NOGE compounds appear in Figure 2.

Chromatography, especially HPLC, plays an important role in describing and measuring NOGE levels. When faced with the need to determine what NOGE compounds are present in a food sample and at what levels by HPLC, an analytical approach is to run calibration curves versus NOGE standards of known composition. Fluka brand NOGE standards are ideal for this application. The three modes of HPLC commonly used in NOGE analysis are SEC, RP- and NP-HPLC. Data on the sample from each technique, because of the different selectivity they provide, can be triangulated to give a clearer picture on the NOGE composition of the sample. SEC (size exclusion chromatography) provides group separation based on number of rings. It is often used as a preparative step to collect fractions relatively homogenous in ring number. These purified fractions are then reacted with water, HCl or acetic acid followed by reversed phase HPLC analysis, or acetylation followed by normal phase HPLC analysis. Both types of reactions open and modify the epoxy ring, and give more descriptive elution patterns. The acetylated compounds can also be analysed by GC or GC-MS. In all cases, it is critical to have reliable analytical NOGE standards

to compare elution patterns, and an HPLC method that provides the maximum resolution of the complex mixture.

Fluka brand standards and Supelco HPLC columns for reliable NOGE analysis

To help food analysts create the calibration curves necessary to characterize their samples, we offer unique BADGE, BFGDE and 3-, 4-, 5- and 6-ring NOGE analytical standards. Also, our Discovery and Ascentis lines of HPLC columns provide the high efficiency and selectivity required to resolve the complex mixtures of NOGE isomers and oligomers. An example of the HPLC analysis of our 4-ring NOGE standard (04976) on a Discovery® C18 HPLC column is shown in Figure 3.

Turn to Sigma-Aldrich when you need reliable, high quality standards, reagents, sample prep and chromatography products, tailored to meet the most current regulations in food, environmental, pharmaceutical and other areas of analytical chemistry. Our expert technical service is at your disposal to help you choose the right products and get the best results from your analysis.

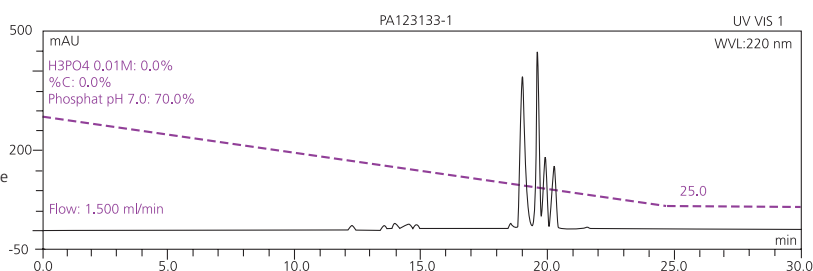
For more information, please call or visit our website:
www.sigma-aldrich.com/noge

For further reading:

Wagner, C.; Grob, K.; Biedermann, M.; Migration of Novolac Glycidyl Ether (NOGE) into Foods: Analytical Problems. *Mitt. Lebensm. Hyg.* 2000, 91, 146–157.
Biedermann, M.; Wagner, C.; Grob, K.; Imhof, D.; Beuggert, H.; Bisphenol-A-Diglycidyl Ether (BADGE) and Novolac Glycidyl Ether (NOGE) as Additives in Can Coatings. *Mitt. Lebensm. Hyg.* 2000, 91, 274–286.

Figure 3. QA chromatogram of Fluka brand 4-ring NOGE standard (04976) on a Discovery C18 column

column: Discovery C18, 15 cm x 4.6 mm I.D., 5 µm particles
mobile phase: (A) CH₃CN, (B) Potassium phosphate, pH 7.0 (both with 0.01M H₃PO₄)
gradient: 30% to 75% A in 25 min, hold at 75% A for 5 min
flow rate: 1.5 ml/min
temp: 35 °C
detection: UV at 220 nm
injection: 10 µl, 4-ring NOGE (04976), 500 mg/ml in mobile phase



Prod No.	Description	Pack Size
NOGE (Novolac glycidyl ether) analytical standards*		
68931	3-Ring NOGE (Mixed isomers) Purity: ≥90% (HPLC) ** New! **	50 mg
04976	4-Ring NOGE (Mixed isomers, aliphatic or branched chains) Purity: ≥90% (HPLC) ** New! **	50 mg
12109	5-Ring NOGE (Mixed isomers, aliphatic or branched chains) Purity: ≥90% (HPLC) ** New! **	50 mg
30977	6-Ring NOGE (Mixed isomers, aliphatic or branched chains) Purity: ≥80% (HPLC) ** New! **	50 mg

* ¹H-NMR, ¹³C-NMR and MS-spectra are available for all NOGE standards on request.

Prod No.	Description	Pack Size
BFDGE (Bisphenol-F-diglycidyl ether) analytical standards (2-ring NOGE compounds)		
15144	BFDGE Bisphenol-F-diglycidyl ether, purum p.a., qualitative standard, mixture of 3 isomers o-,o-, o-,p-, p-,p-, ≥95.0% (total assay of the 3 isomers, GC)	500 mg
15139	Bisphenol-F-bis(3-chloro-2-hydroxypropyl) ether p.a., qualitative standard, mixture of 3 isomers o-,o-, o-,p-, p-,p-, ~95% (total assay of the 3 isomers, HPLC)	250 mg
15142	Bisphenol-F-bis(2,3-dihydroxypropyl) ether purum p.a., qualitative standard, mixture of 3 isomers o-,o-, o-,p-, p-,p-, ≥95.0% (total assay of the 3 isomers, HPLC)	250 mg

Prod No.	Description	Pack Size
BADGE (Bisphenol-A-diglycidyl ether) analytical standards		
15138	BADGE Bisphenol-A-diglycidyl ether, purum p.a., qualitative standard, ≥97.0% (GC)	500 mg
15136	BADGE-2HCl Bisphenol-A-bis(3-chloro-2-hydroxypropyl) ether, purum p.a., qualitative standard, ≥97.0% (HPLC)	250 mg, 1 g
15137	Bisphenol-A-bis(2,3-dihydroxypropyl) ether, purum p.a., qualitative standard, ≥97.0% (HPLC)	250 mg
92427	BADGE-HCl-H ₂ O Bisphenol-A-(3-chloro-2-hydroxypropyl) (2,3-dihydroxypropyl) ether, purum p.a., qualitative standard, ≥95.0% (HPLC)	25 mg, 100 mg
73124	BADGE-HCl Bisphenol-A-(3-chloro-2-hydroxypropyl) glycidyl ether, p.a., qualitative standard, ≥90% (HPLC)	25 mg, 100 mg
73417	BADGE-H ₂ O Bisphenol-A-(2,3-dihydroxypropyl) glycidyl ether, purum p.a., qualitative standard, ≥95.0% (HPLC)	25 mg, 100mg

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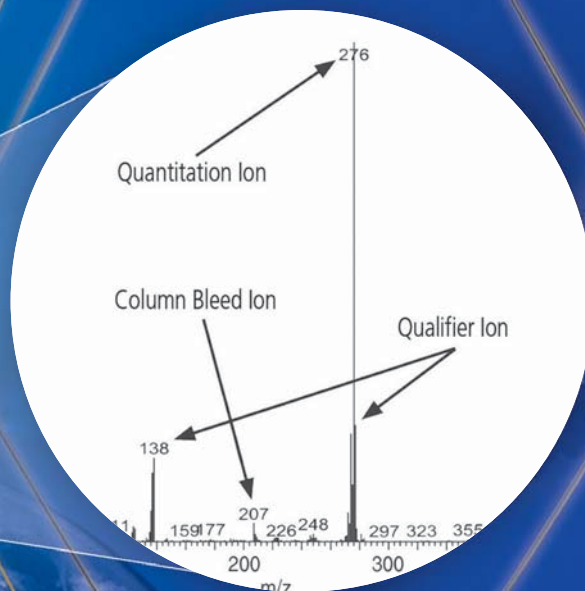
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- Less instrument downtime
- Less preventative maintenance
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- Greater reproducibility, column-to-column

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