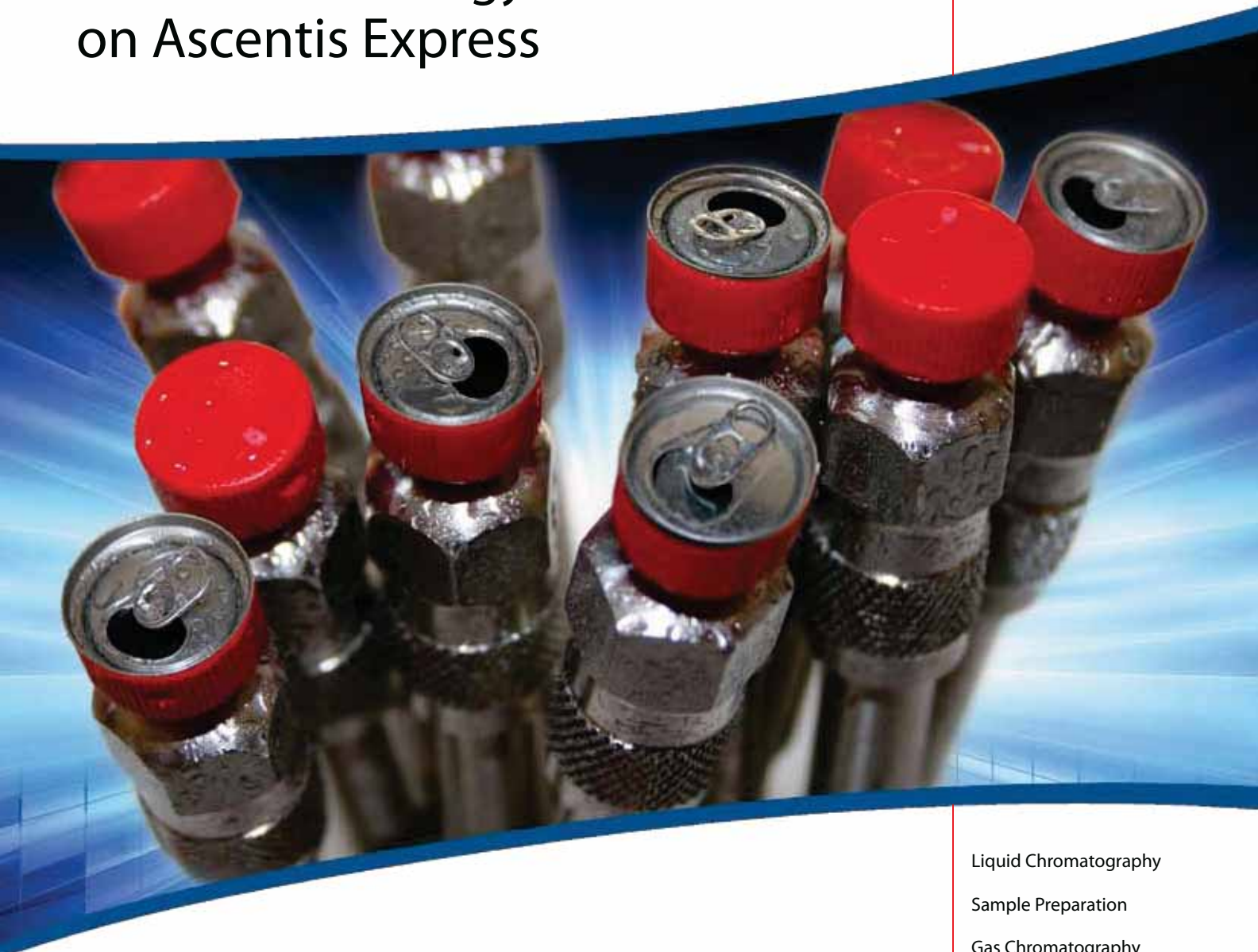


# Reporter

Volume 28.4

 **SUPELCO**  
Analytical

## Caffeinated Energy Drinks on Ascentis Express



*Ascentis Express HPLC columns are an excellent tool  
for analyses in the food industry.*

Liquid Chromatography

Sample Preparation

Gas Chromatography

Standards

Accessories

Chiral Chromatography

**SIGMA-ALDRICH**

Visit us on the web at  
[sigma-aldrich.com/thereporter](http://sigma-aldrich.com/thereporter)

## Table of Contents

### Liquid Chromatography

Rapid Analysis of Caffeinated Energy Drinks by HPLC on Ascentis Express.....3

Ascentis® Express Guards: Efficient and Effective Protection for all HPLC and UHPLC Columns.....5

LC-MS CHROMASOLV Pre-blended Solvents and Water.....20

### Sample Preparation

High Efficiency Extractions Using Empore SPE Products.....14

Supel-Select HLB SPE.....15

BPE-DNPH: A More Effective Device for Sampling Airborne Carbonyls Under High Humidity Conditions.....16

SPME Fast Fit Assemblies for Automated Fiber Exchange.....18

New SPME Devices for Air/Field Sampling.....18

### Gas Chromatography

Broad Range of Selective Cyclodextrin Phases for Chiral GC.....7

Help, I'm Trapped in Alphabet Soup!.....8

New Adapter for On-the-Fly Sampling During Distillation.....12

Detailed Hydrocarbon Analysis (DHA) Using Gas Chromatography (GC).....13

### Standards

New Analytical Standards for R-(+)-Lipoic Acid.....22

New Neat and Single-component Solutions.....22

Premixed Sterol Reference Standards.....23

C18:1 cis/trans (FAMES).....23

### Accessories

Maximum Sample Recovery with MRQ30 CD™ Vials.....19

6-Unit Multipacks of Hamilton Autosampler Syringes.....19

## Finding the eNeedle in the eHaystack



Michael Buchanan  
Product Manager,  
Gas Separations

Dear Colleague:

The World Wide Web is a very powerful and effective means of disseminating a vast amount of information to a wide audience. Unfortunately, the enormous volume of information may make it difficult to find the 'good' content in the midst of all the 'clutter' that exists, making the task seem like the proverbial 'finding the needle in the haystack' in an electronic sense. Two areas recently developed for the Sigma-Aldrich web site are designed to make 'finding the needle' a little bit easier.

### Environmental Web Node ([sigma-aldrich.com/environmental](http://sigma-aldrich.com/environmental))

This large application-based node is conveniently arranged by environmental laboratory area to assist the user in quickly locating appropriate products, information, and references for their specific application. Sections are included for:



- Organics (volatiles by GC-MS, volatiles by GC, semivolatiles by GC-MS, semivolatiles by GC, high resolution GC-MS, HPLC, and extraction)
- Inorganics (metals by ICP or AA, mercury by CVAA, and digestion)
- Wet/conventional chemistry (physical analyses, chemical analyses, and microbiology)
- Sample collection and preservation

This node will be rolled out over the next several months, so bookmark your browser and check back often.

### Oil Spill Landing Page ([sigma-aldrich.com/oilspill](http://sigma-aldrich.com/oilspill))

This new landing page relates to the many sample collection and analytical challenges faced by field workers and laboratory analysts who respond to fuel/oil spill incidents. Various aspects of a fuel/oil release are covered, including:



- Health and safety: collection/analysis of air samples
- Environmental protection: collection/analysis of water and soil samples
- Food safety: analysis of seafood products
- Litigation: fingerprinting analysis for source identification
- Environmental fate: microbiological monitoring

Appropriate products can be located by type, application, or regulated methodologies. Several great technical resources are also included.

Much more application-based content is available at the Sigma-Aldrich web site. We hope you find what you need. Let us know your needs so that we can continue to provide the information sought by analytical scientists around the world.

Regards,

Michael D. Buchanan  
Product Manager, Gas Separations  
[mike.buchanan@sial.com](mailto:mike.buchanan@sial.com)

Reporter is published 5 times a year by Supelco Marketing, 595 North Harrison Road, Bellefonte, PA 16823-0048.

# Rapid Analysis of Caffeinated Energy Drinks by HPLC on Ascentis Express

Hillel Brandes and Hugh Cramer  
hillel.brandes@sial.com

Already very popular for separations in the pharmaceutical industry, Ascentis® Express has applicability to separations in many other industries, such as food and beverage. This brief report shows an example of how Ascentis Express provides the high speed and high efficiency of sub-2 µm particles, but at half the backpressure. This lower pressure means that Ascentis Express can be run on all HPLC systems, including conventional as well as UHPLC systems.

## HPLC and Food Analysis: Caffeinated Energy Drinks

Caffeinated energy drinks contain a variety of ingredients that usually includes a sweetener (sugars, synthetic sugar substitutes, zero-calorie natural sweeteners), vitamin B supplements, and, of course, caffeine. They may also include amino acids, organic acids, and various plant extracts. The sample complexity makes it important to use highly-efficient, highly-selective phases and columns that are compatible with different detection systems to maximize the information from HPLC experiments. Ascentis Express Fused-Core® columns meet these requirements.

## Efficient, Robust Columns for Long Lifetime, Even with Real Samples

Our applications group received a request from a customer to help them transfer their 1980's-era HPLC method onto new technology Ascentis Express columns, to take advantage of the speed and sensitivity benefits the columns offer. The sample was a diet cola beverage. After we completed this work, we applied the approach to today's popular energy drinks, first looking at methods to resolve the vitamin B group. These compounds are widely used as additives, though drinks vary as to which ones are present. We also wanted to be sure the method was applicable to caffeine, sugar, and organic acids, since these are also common ingredients. We chose ROCKSTAR® Energy Drink for our initial evaluation. Although by no means a comprehensive study, we were able to resolve the major components of the drink, leveraging detection method (UV and ELSD) and phase chemistry to alter elution pattern and sensitivity.

## Using Phase Chemistry to Alter Retention and Elution Patterns

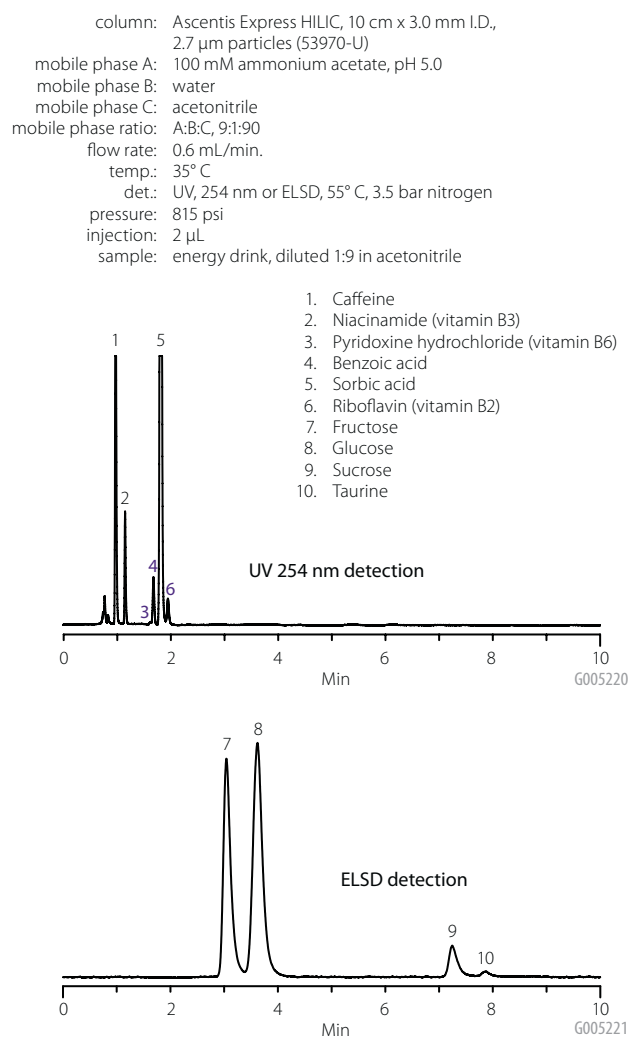
During our method development we explored different Ascentis Express phase chemistries, in particular the HILIC and RP-Amide. These phases were chosen because of their enhanced performance toward polar compounds compared to C18. HILIC (hydrophilic interaction chromatography) is complementary to reversed-phase separations, providing different selectivity for polar compounds and using high organic mobile phases. Important benefits of HILIC are retention of highly polar analytes (like metabolites) and increased MS sensitivity.

We also chose 3 mm I.D. columns to improve sensitivity by reducing peak volume over 4.6 mm I.D. columns. All separations were run on conventional HPLC systems; unlike columns packed with sub-2 µm particles, Ascentis Express does not require ultra-high pressure instruments to achieve the rapid, efficient separations.

The results on the Ascentis Express HILIC column are found in Figure 1. Here, UV and ELSD detection was used to detect different types of compounds; ELSD allowed us to see the non-UV absorbing sugars. The HILIC conditions yielded extremely rapid analysis (under 2 minutes) and MS-friendly mobile phase. The low backpressure of HILIC mobile phases also permits high flow rates for fast analysis.

(continued on page 4)

Figure 1. Energy Drink on Ascentis Express HILIC



(continued from page 3)

**Figure 2** shows the results on the Ascentis Express RP-Amide. Although technically operating by a reversed-phase mechanism and operating in the same types of mobile phases, the amide functional groups confer different selectivity than alkyl-only phases, like C8 or C18. The selectivity differences are most noticeable with polar compounds, like those in this application.

### Selectivity, Peak Order, and Trace Analysis Implications

Comparing the separations on the HILIC and RP-Amide columns, a few points are notable. Both showed good retention but different elution patterns of these very polar compounds. Of particular importance is the relative elution of caffeine. The RP-Amide column gave greater separation and caused it to elute after the lower-concentration B3 and B6 peaks. For trace analysis, it is recommended that the component in large excess elute after the components at trace levels. Using different phase chemistries is the most effective means to alter elution order. We have provided this ability by offering a wide variety of Ascentis Express phase chemistries: C18, C8, Phenyl-Hexyl, HILIC, RP-Amide, and ES-C18 for peptides, with others currently in development.

### Conclusion

This very brief example demonstrates three important features of Ascentis Express columns as they apply to analyses in the food and beverage industry:

- they provide very rapid separations on any HPLC or UHPLC system
- the robust columns stand up to long-term use, even with "real" samples
- they are available in a variety of phase chemistries for different selectivity

These benefits can be applied to analysis in any industry. The products used in this application are listed in Featured Products. The complete list of Ascentis Express columns can be found at [sigma-aldrich.com/express](http://sigma-aldrich.com/express)

Visit Supelco at the following tradeshow:

**2010 FIP PSWC/AAPS -  
American Association of  
Pharmaceutical Scientists**

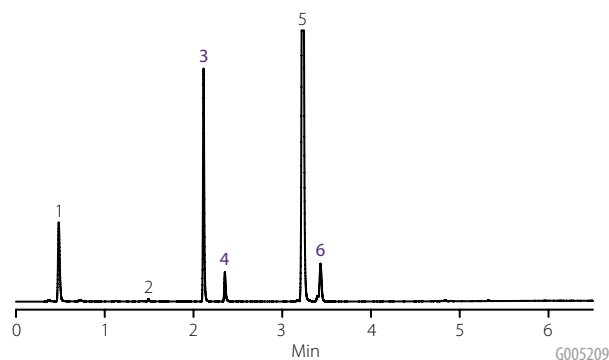
**Booth # 454**

November 15-17  
Morial Convention Center  
New Orleans, Louisiana

**Figure 2. Energy Drink on Ascentis Express RP-Amide**

column: Ascentis Express RP-Amide, 5 cm x 3.0 mm I.D.,  
2.7  $\mu$ m particles (53916-U)  
mobile phase A: 0.1% TFA (v/v) in water  
mobile phase B: 0.1% TFA (v/v) in acetonitrile  
gradient: Min. % A % B  
0.0 100 0  
0.5 100 0  
5.5 40 60  
6.5 40 60  
flow rate: 0.6 mL/min.  
temp.: 35° C  
det.: UV at 254 nm  
pressure: 1140 psi  
injection: 2  $\mu$ L  
sample: energy drink, undiluted

1. Niacinamide (vitamin B3)
2. Pyridoxine hydrochloride (vitamin B6)
3. Caffeine
4. Riboflavin (vitamin B2)
5. Sorbic acid
6. Benzoic acid



### + Featured Product

Description	Cat. No.
Ascentis Express HILIC, 10 cm x 3.0 mm, 2.7 $\mu$ m particles	<b>53970-U</b>
Ascentis Express RP-Amide, 5 cm x 3.0 mm, 2.7 $\mu$ m particles	<b>53916-U</b>

### + Related Products

Description	Qty. (mg)	Cat. No.
Caffeine	250	<b>C1778</b>
Niacinamide	1000	<b>47865-U</b>
Pyridoxine hydrochloride	1000	<b>47862</b>
Benzoic acid	1000	<b>47849</b>
Sorbic acid	1000	<b>47845</b>
Riboflavin	1000	<b>47861</b>
Fructose	10	<b>F0127</b>
Glucose	1000	<b>47829</b>
Sucrose	500	<b>47289</b>
Taurine	10	<b>T0625</b>

# Ascentis Express Guards: Efficient and Effective Protection for all HPLC and UHPLC Columns

Craig Aurand  
craig.aurand@sial.com

The new Ascentis Express Guard Cartridge system provides significant improvements in column lifetime of any HPLC column, including classic particles, sub-2 µm UHPLC, and newer technology superficially porous particles, without sacrificing resolution or throughput. The efficiency permits their use as an analytical column, directly connecting to MS interfaces.

## Protecting any HPLC Column is Important

Using guard columns is recommended to protect your HPLC column investment. Guards extend column life by providing chemical and mechanical protection from contaminants entering the system from the sample, the mobile phase, and instrument wear. The ideal guard column design provides this protection without sacrificing performance or throughput.

## Why sub-2 µm HPLC Columns Foul Easily

Recent significant advances in HPLC have evolved around new particle technologies and enabling instrumentation. Sub-2 µm or UHPLC particles have garnered attention in the past decade because of the high column efficiencies and rapid separations they can provide. Although the advantages of sub-2 µm particles have been largely supplanted by the Fused-Core® particle technology behind Ascentis Express, they are still quite common. Many users have found that a significant drawback to sub-2 µm particles is their tendency to foul, which leads to high pressure and lost resolution. The broad particle size distribution of sub-2 µm particles requires 0.5 µm frits; this is the reason UHPLC columns foul so readily.

## New Ascentis Express HPLC Guard Column

Users of any HPLC columns, whether packed with conventional HPLC particles, the sub-2 µm UHPLC particles discussed above, or the Ascentis Express Fused-Core or other superficially-porous particles, can realize significant improvements in column lifetime without sacrificing resolution or throughput by using the new Ascentis Express guards. In terms of theoretical plates per meter (N/m), these guards provide values comparable to Ascentis Express analytical columns, but add only seconds to the analysis time.

## Effective for Column Protection and Stand-alone Performance

A demonstration of the performance of the Ascentis Express guards is shown in **Figure 1**. Methadone and two metabolites were separated on a 5 cm x 2.1 mm I.D. Ascentis Express analytical column with and without a 0.5 cm x 2.1 mm I.D. Ascentis Express guard column. Comparison of the top and middle chromatograms show that the Ascentis Express guard

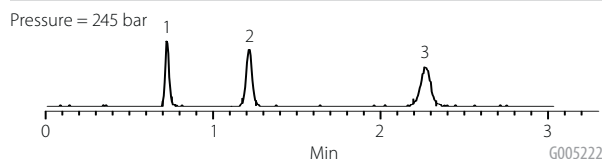
(continued on page 6)

**Figure 1. Efficient, Effective Column Protection with Ascentis Express Guard Columns**

column: Ascentis Express RP-Amide, 5 cm x 2.1 mm ID., 2.7 µm particles (53911-U)  
 guard column: Ascentis Express RP-Amide Guard, 0.5 cm x 2.1 mm ID., 2.7 µm particles (53514-U)  
 mobile phase A: 10 mM ammonium formate, pH 3.6  
 mobile phase B: acetonitrile  
 mobile phase ratio: 70:30 A:B  
 flow rate: 0.6 mL/min.  
 temp.: ambient  
 det.: MS ESI(+)-TOF, 3.0 spectra/second, 3089 transients/scan  
 injection: 1 µL  
 sample: 200 ng/mL each in mobile phase A  
 (EDDP is 2-ethylidene-1,5-dimethyl-3,3-diphenylpyrrolidine;  
 EMDP is 2-ethyl-5-methyl-3,3-diphenylpyraline)

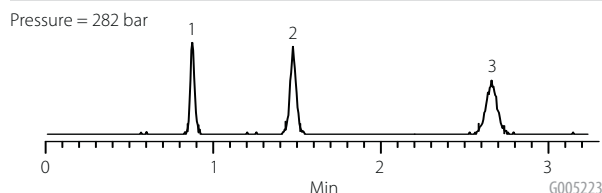
### Analytical Column Alone

Peak	Name	Ret. Time (mins.)	N (plates)
1	EDDP	0.721	5,037
2	Methadone	1.218	5,580
3	EDMP	2.267	7,337



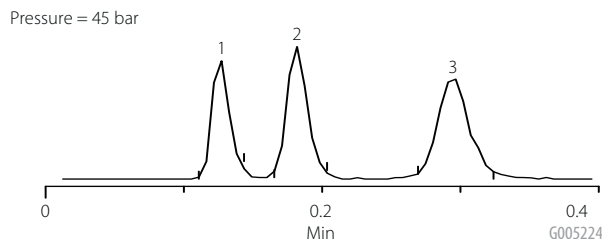
### Analytical Column + Ascentis Express Guard Column

Peak	Name	Ret. Time (mins.)	N (plates)
1	EDDP	0.875	5,117
2	Methadone	1.475	8,187
3	EDMP	2.660	10,110



### Ascentis Express Guard Column Alone

Peak	Name	Ret. time (mins.)	N (plates)
1	EDDP	0.127	243
2	Methadone	0.182	497
3	EDMP	0.297	848



Liquid Chromatography



column actually increased the total number of plates in the system by nearly 3,000, but added only 20 seconds and 37 bar. The Ascentis Express guard column actually has enough plates to serve as an analytical column. The bottom chromatogram shows the same separation with the guard column directly inserted into the MS interface as in **Figure 2**. Note the extremely rapid analysis (<20 seconds) and baseline resolution.

### Features and Benefits of Ascentis Express Guards

Four features lead to the efficiency benefits of Ascentis Express guards:

- design provides zero-dead volume connections
- auto-adjusting ferrule seating depth (prevents gaps)
- low (<1.5  $\mu\text{L}$ ) total swept volume flow path (excluding the cartridge)
- Ascentis Express Fused-Core (2.7  $\mu\text{m}$ ) packing material

### Other important attributes:

- finger-tight design for tool-free installation
- rated to 15,000 psi (1,030 bar)
- direct connection to any HPLC column or compatible threaded interface
- economical, disposable cartridges for cost-effective protection

Ascentis Express guards can be used to protect any HPLC or UHPLC column, and also can serve on their own as a short analytical column for extremely rapid analysis. The product listing below contains the various phases currently available packed into the Ascentis Express guards, along with the hardware

### + Featured Products

Description	What Phases it Protects	I.D. (mm)	Qty.	Cat. No.
<b>Ascentis Express Guard Cartridges</b> (All particles 2.7 $\mu\text{m}$ , 0.5 cm length. Requires holder 53500-U)				
Ascentis Express C18 guard cartridge	C18	2.1	3	<b>53501-U</b>
	C18	3.0	3	<b>53504-U</b>
	C18	4.6	3	<b>53508-U</b>
Ascentis Express C8 guard cartridge	C8	2.1	3	<b>53509-U</b>
	C8	3.0	3	<b>53511-U</b>
	C8	4.6	3	<b>53512-U</b>
Ascentis Express RP-Amide guard cartridge	Amide	2.1	3	<b>53514-U</b>
	Amide	3.0	3	<b>53516-U</b>
	Amide	4.6	3	<b>53519-U</b>
Ascentis Express HILIC guard cartridge	HILIC silica	2.1	3	<b>53520-U</b>
	HILIC silica	3.0	3	<b>53521-U</b>
	HILIC silica	4.6	3	<b>53523-U</b>
Ascentis Express Phenyl-Hexyl guard cartridge	Phenyl or Phenyl-hexyl	2.1	3	<b>53524-U</b>
	Phenyl or Phenyl-hexyl	3.0	3	<b>53526-U</b>
	Phenyl or Phenyl-hexyl	4.6	3	<b>53531-U</b>
Ascentis Express Peptide ES-C18 guard cartridge	ES-C18, any C18 phase for peptides or low pH work	2.1	3	<b>53536-U</b>
	ES-C18, any C18 phase for peptides or low pH work	3.0	3	<b>53537-U</b>
	ES-C18, any C18 phase for peptides or low pH work	4.6	3	<b>53542-U</b>
<b>Holder for Ascentis Express Cartridges</b>				
Contains everything needed to connect guard cartridges to an analytical column or to a compatible 10/32 threaded interface.				<b>53500-U</b>

**Figure 2. Ascentis Express Guard Connected Directly to MS Interface**



**Note:** Guard cartridge shown connected with our new Supelco high performance fittings.

housing. The guards are supplied in packs of three cartridges. We also recommend using our high performance HPLC fittings with these and any HPLC columns.

### + Related Products

Description	Qty. (mg)	Cat. No.
EDDP	10	<b>E5264</b>
Methadone	50	<b>M0267</b>

# Broad Range of Selective Cyclodextrin Phases for Chiral GC

Tracy Ascah  
tracy.ascah@sial.com

Supelco carries the widest range of CSPs for chiral GC in the industry, and offers the technical expertise to guide you through simple or complex chiral separations.

In the realm of chiral GC separations, cyclodextrins are king because they have the combined power of architecture and selective surface chemistry. As with chiral HPLC, it is difficult if not impossible to predict elution when facing a new racemate. Usually multiple columns are screened for the desired enantioselectivity. Screening protocols benefit when phases with widely different selectivity are employed. That is where the Astec CHIRALDEX® and Supelco DEX™ lines come into play. Together, these two lines comprise twenty different chemistries – the largest selection in the industry.

**Table 1** below presents the different CHIRALDEX and Supelco DEX chemistries available. If you can't have one of each chemistry in your lab, we highly recommend including at least these three as part of all chiral GC screening protocols:

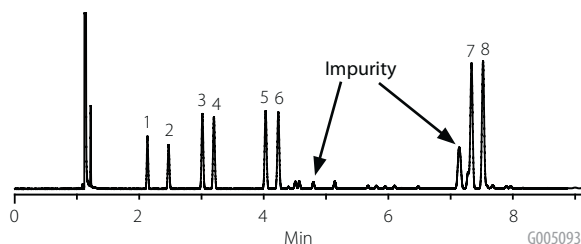
- Astec CHIRALDEX G-TA (Trifluoroacetyl, 2,6-di-O-pentyl-3-trifluoroacetyl) (See **Figure 1**)
- Supelco β-DEX 120 (Permethyl, 2,3,6-tri-O-methyl)
- Astec CHIRALDEX B-DM (Dimethyl, 2,3-di-O-methyl-6-t-butyl silyl)

These columns are included in the cost-effective screening kits described in **Table 2**. To see our complete line of capillary chiral GC columns, along with our complete line for chiral chromatography and chiral chemistry, please visit [sigma-aldrich.com/chiral](http://sigma-aldrich.com/chiral).

**Figure 1. Example of Chiral GC Separation on Astec CHIRALDEX G-TA: 1,2-Alkyldiol Enantiomers (O-Trifluoroacetyl Derivatives)**

column: Astec CHIRALDEX G-TA, 30 m x 0.25 mm I.D., 0.12 μm (73033AST)  
oven: 80 °C, 5 °C/min. to 130 °C  
carrier gas: helium, 30 psi  
det.: FID, 250 °C  
injection: 1 μL, 80:1 split

peaks 1 & 2: 1,2-propanediol enantiomers  
peaks 3 & 4: 1,2-pentenediol enantiomers  
peaks 5 & 6: 1,2-hexanediol enantiomers  
peaks 7 & 8: 1,2-octanediol enantiomers



**Table 2. Chiral Capillary GC Screening Kits**

Kit 1: Astec CHIRALDEX	Kit 2: Supelco DEX	Kit 3: Supelco DEX
Cat. No. 71030AST	Cat. No. 24340	Cat. No. 24328-U
All 30 m x 0.25 mm, 0.12 μm	All 30 m x 0.25 mm, 0.25 μm	All 30 m x 0.25 mm, 0.25 μm
CHIRALDEX G-TA	α-DEX 120	β-DEX 120
CHIRALDEX B-DM	β-DEX 120	β-DEX 225
CHIRALDEX B-DA	γ-DEX 120	γ-DEX 225
		β-DEX 325
<b>Cost Savings Over Columns Sold Separately:</b>		
14%	13%	13%

**Table 1. Cyclodextrin Chemistries Available in the CHIRALDEX and Supelco DEX Product Lines**

Phase	Derivative	Cyclodextrin type		
		α (alpha)	β (beta)	γ (gamma)
CHIRALDEX BP	Butyryl			G-BP
Supelco DEX 225	Diacetyl	α-DEX 225	β-DEX 225	γ-DEX 225
CHIRALDEX DA	Dialkyl	A-DA	B-DA	G-DA
CHIRALDEX DM	Dimethyl		B-DM	G-DM
Supelco DEX 325	Dimethyl	α-DEX 325	β-DEX 325	γ-DEX 325
CHIRALDEX DP	Dipropionyl		B-DP	G-DP
CHIRALDEX PM	Permethylated		B-PM	
Supelco DEX 110	Permethylated		β-DEX 110	
Supelco DEX 120	Permethylated	α-DEX 120	β-DEX 120	γ-DEX 120
CHIRALDEX PN	Propionyl			G-PN
CHIRALDEX PH	S-Hydroxypropyl		B-PH	
CHIRALDEX TA	Trifluoroacetyl	A-TA	B-TA	G-TA



# DRO, EPH, ETPH, GRO, PRO, PVOC, STARS, TPH, VPH? Help, I'm Trapped in Alphabet Soup!



Katherine K. Stenerson and Michael D. Buchanan  
mike.buchanan@sial.com

## Introduction

The analysis of samples resulting from fuel and oil spills is different from the analyses routinely performed by either a petroleum or an environmental testing laboratory.

- Many petroleum methods require identification and quantitation using pattern recognition against known reference fuel/oil standards. This is possible when the fuel/oil is relatively pure (such as when contained/protected in a drum or tank).
- Most environmental methods require individual analytes to be identified and quantified. The complex nature of many fuels/oil tends to mask the detection of individual analytes at low levels.

Once a fuel/oil is released into the environment, two factors may contribute to a decrease in its integrity:

- 1) If more than one fuel/oil type is present, it may be impossible to use pattern recognition because the analyte ratios may not match any reference fuel/oil standards.
- 2) As the fuel/oil is weathered (evaporation of lower molecular weight compounds and/or degradation from UV rays or oxygen of some or all of the compounds), it becomes very hard to identify fuel/oil type because the analyte ratios may no longer match reference fuel/oil standards. Also, the quantitation of weathered patterns in the samples to non-weathered reference standards is inaccurate.

For soil and water samples collected due to off-shore oil spills, leaking railroad tanker cars and highway tanker trucks after accidents, and underground storage tank (UST) remediation at service stations, it may be preferred to measure contamination by carbon range, instead of by fuel/oil type or individual analytes. The United States Environmental Protection Agency (US EPA), United States Coast Guard (USCG), American Society for Testing and Materials (ASTM), and several States have published methodologies for these purposes.

## Terms and Definitions

Several terms associated with the sampling and analyses of environmental samples for fuels/oils are currently used. Because there are many agencies with methods, there are many terms, resulting in 'alphabet soup' due to the vast number of acronyms. Also note that the same term may be used by multiple agencies but with slightly different meanings. While not inclusive, **Table 1** represents an attempt to define some of the most common acronyms. While there are many methods, they can be grouped into three categories:

- Gasoline Range Organics (GRO): The GRO-like methods are for volatile analytes, and require purge and trap or direct injection
- Diesel Range Organics (DRO): The DRO-like methods are for semivolatile analytes, and require solvent extraction
- Total Petroleum Hydrocarbons (TPH): The TPH methods are for the heavier volatile, the semivolatile, and some heavier analytes, and require solvent extraction

---

*...there are many terms, resulting in 'alphabet soup'  
due to the vast number of acronyms...*

---

## GRO-Like Analysis by Purge and Trap

Analysis of the volatile portion of fuels/oils typically requires calibration with several n-alkane and aromatic compounds. Some UST programs also require testing for lower molecular weight n-alkanes such as pentane, and for oxygenates such as methyl tert-butyl ether (MTBE). **Figure 1** shows a GRO-like chromatogram that includes an analyte list combined from several methods.

The analysis of soil samples often involves a methanol extraction, resulting in a large amount of methanol being transferred to the purge vessel. If retained by the purge trap, methanol can interfere with the detection of early eluting compounds when the trap is desorbed to the GC. The "M" purge trap is specially designed to retain the lighter analytes (pentane and MTBE), but not a significant amount of methanol. A VOCOL™ column was selected due to its ability to provide a short run time while maintaining resolution of critical pairs.

## GRO-Like Analysis by Direct Injection

Most GRO-like methods require a purge and trap technique for sample introduction into the GC. US EPA Method 8015 also allows direct injection to be used in some instances (such as for high level samples). **Figure 2** shows a chromatogram of our EPA GRO Mix. An SLB™-5ms column with a 1.0 μm film was used to help focus and retain 2-methylpentane.



Table 1. Terms and Definitions (see References 1-12 on page 10)

Term	Agency	n-Alkane Range	Aromatic Range	Boiling Point Range	Sample Preparation	Analysis
DRO (Diesel Range Organics)	US EPA	C10-C28	n/a	170-430 °C	solvent extraction	GC-FID
	Alaska	C10-C25	n/a	170-400 °C	solvent extraction	GC-FID
	Wisconsin	C10-C28	n/a	170-430 °C	solvent extraction	GC-FID
EPH (Extractable Petroleum Hydrocarbons)	Massachusetts	C9-C36	C11-C22	150-265 °C	solvent extraction + SPE fractionation	GC-FID
ETPH (Extractable Total Petroleum Hydrocarbons)	Connecticut	C9-C36	n/a	n/a	solvent extraction	GC-FID
GRO (Gasoline Range Organics)	US EPA	C6-C10	n/a	60-170 °C	purge/trap or direct injection	GC-FID
	Alaska	C6-C10	n/a	60-170 °C	purge/trap	GC-FID
	Wisconsin	C6-C10	n/a	60-220 °C	purge/trap	GC-FID
PRO (Petroleum Range Organics)	Florida	C8-C40	n/a	n/a	solvent extraction	GC-FID
PVOC (Petroleum Volatile Organic Compounds)	Wisconsin	▲	n/a	60-220 °C	purge/trap	GC-PID
STARS (Spill Technology and Remediation Series)	New York	◆	◆	◆	◆	GC-MS
TPH (Total Petroleum Hydrocarbons)	Texas	C6-C35	n/a	n/a	solvent extraction	GC-FID
	Washington	C7-C30	n/a	n/a	solvent extraction	GC-FID
VPH (Volatile Petroleum Hydrocarbons)	Massachusetts	C5-C12	C9-C10	36-220 °C	purge/trap	GC-PID-FID

▲ Wisconsin defines the PVOC analyte list as the Wisconsin GRO list minus naphthalene.

◆ STARS is a State of New York document that provides guidance on the handling, disposal, and/or reuse of excavated petroleum-contaminated soil, with analysis by GC-MS per US EPA Methods 8260 and 8270, modified with short, specific analyte lists.

Figure 1. GRO-Like Analysis (Purge and Trap)

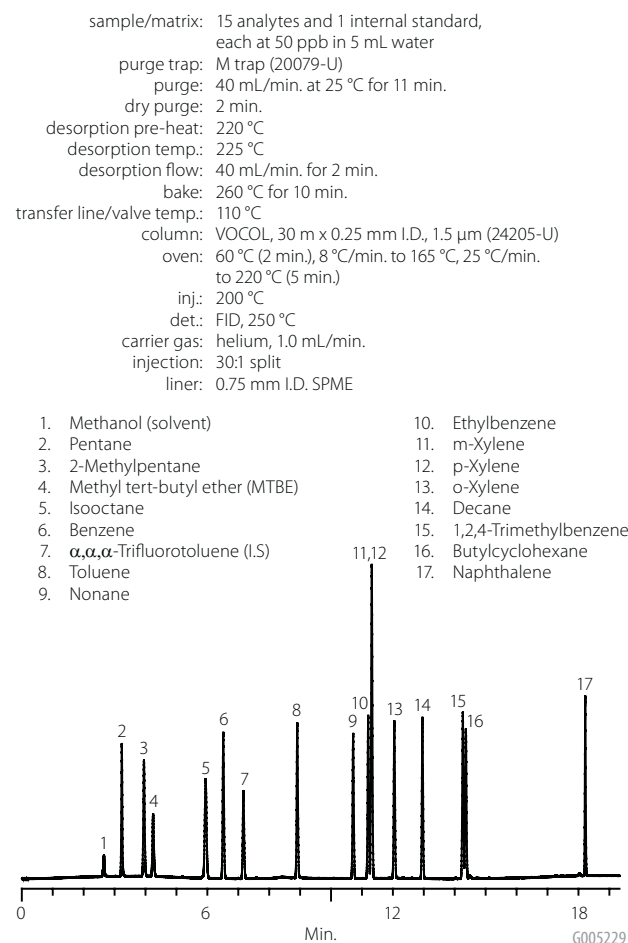


Figure 2. GRO-Like Analysis (Direct Injection)

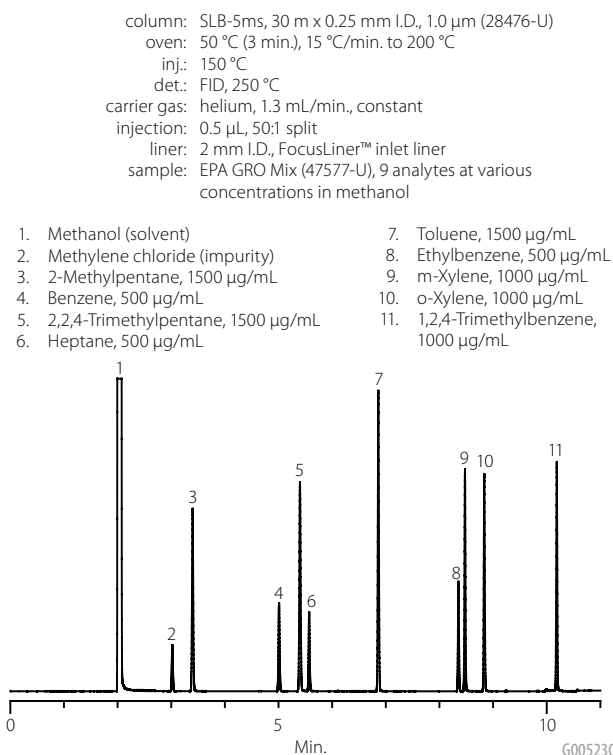


Figure 3. DRO Analysis

column: SLB-5ms, 20 m x 0.18 mm I.D., 0.18  $\mu$ m (28564-U)  
 oven: 50 °C (2 min.), 20 °C/min. to 325 °C (1 min.)  
 inj.: 250 °C  
 det.: FID, 350 °C  
 carrier gas: helium, 0.9 mL/min., constant  
 injection: 1  $\mu$ L, splitless (splitter open at 0.5 min.)  
 liner: 2 mm I.D., FocusLiner inlet liner  
 sample: 10 analytes, 1 surrogate, and 1 internal standard,  
 each at 50  $\mu$ g/mL in methylene chloride

- |                                 |                                   |
|---------------------------------|-----------------------------------|
| 1. Methylene chloride (solvent) | 8. Eicosane                       |
| 2. Decane                       | 9. 5- $\alpha$ -Androstane (I.S.) |
| 3. Dodecane                     | 10. Docosane                      |
| 4. Tetradecane                  | 11. Tetracosane                   |
| 5. Hexadecane                   | 12. Hexacosane                    |
| 6. Octadecane                   | 13. Octacosane                    |
| 7. o-Terphenyl (surr.)          |                                   |

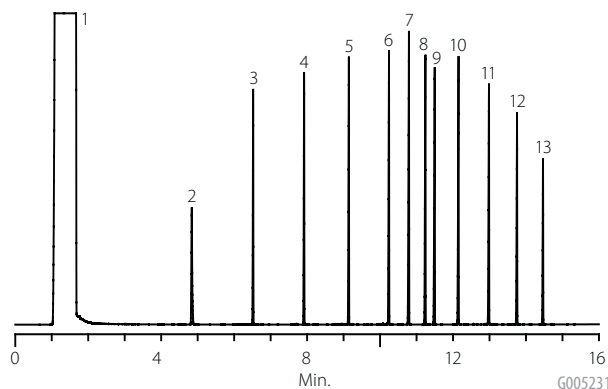
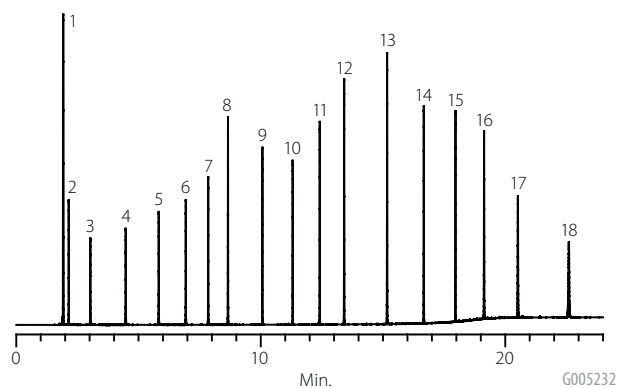


Figure 4. TPH Analysis

column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25  $\mu$ m (28471-U)  
 oven: 45 °C (3 min.), 20 °C/min. to 360 °C (10 min.)  
 inj.: 275 °C  
 det.: FID, 365 °C  
 carrier gas: helium, 1.3 mL/min. constant  
 injection: 1.0  $\mu$ L, 100:1 split  
 liner: 2 mm I.D. straight  
 sample: 17 analytes, each at 1000  $\mu$ g/mL in carbon disulfide

- |                               |                       |
|-------------------------------|-----------------------|
| 1. Carbon disulfide (solvent) | 10. Hexadecane        |
| 2. Hexane                     | 11. Octadecane        |
| 3. Heptane                    | 12. Eicosane          |
| 4. Octane                     | 13. Tetracosane       |
| 5. Nonane                     | 14. Octacosane        |
| 6. Decane                     | 15. Dotriacontane     |
| 7. Undecane                   | 16. Hexatriacontane   |
| 8. Dodecane                   | 17. Tetracontane      |
| 9. Tetradecane                | 18. Tetratetracontane |



### DRO-like Analysis

Methods for DRO-like are not too different from other work typically performed by environmental laboratories, requiring a solvent extraction and a concentration step prior to GC analysis. The main difference is in the data processing. **Figure 3** shows a DRO-like chromatogram with 10 analytes, a surrogate, and an internal standard. A short, narrow bore SLB-5ms column was used to reduce analysis time to less than 15 minutes.

### TPH Analysis

**Figure 4** shows the analysis of C<sub>6</sub>-C<sub>44</sub> n-alkanes on the SLB-5ms. The composition of the standard represents a carbon range that meets/exceeds the range typically used for TPH analysis. The stability of the SLB-5ms allowed for the use of a high final oven temperature of 360 °C, reducing the elution time of the final C<sub>44</sub> hydrocarbon.

### Conclusion

The analytical methods required to process samples from a fuel/oil spill incident differ from the methods typical performed in either petroleum or environmental laboratories. Once released into the environment, the fuel/oil is no longer pure enough for pattern recognition, but is too complex for identification and quantitation of individual analytes. A multitude of methods exist to handle these unique sample types.

As confusing as it seems to untangle the many acronyms, there are just three types of analyses: GRO, DRO, and TPH. The published methods are simply variations of these to meet the

different analytical needs of the individual agencies. Of utmost importance is to follow the exact methodology required by the regulatory group that will be validating the data reports.

### References

1. US EPA Method 8015C, "Nonhalogenated Organics by Gas Chromatography" Revision 3, February 2007.
2. Alaska DEC Method AK101, "For the Determination of Gasoline Range Organics" Version 04/08/02
3. Alaska DEC Method AK102, "For Determination of Diesel Range Organics" Version 04/08/02
4. Connecticut DEP Method, "Analysis of Extractable Total Petroleum Hydrocarbons (ETPH) Using Methylene Chloride Gas Chromatograph/Flame Ionization Detection" March 1999.
5. Florida DEP Method FL-PRO, "Method for Determination of Petroleum Range Organics" Revision 1, November 1995.
6. Massachusetts DEP Method MADEP-VPH-04-1.1, "Method for the Determination of Volatile Petroleum Hydrocarbons (VPH)" Revision 1.1, May 2004.
7. Massachusetts DEP Method MADEP-EPH-04, "Method for the Determination of Extractable Petroleum Hydrocarbons (EPH)" Revision 1.1, May 2004.
8. New York DEC STARS ([www.dec.ny.gov/regulations/30902.html](http://www.dec.ny.gov/regulations/30902.html)), accessed 10-Aug-2010.
9. Texas NRCC Method 1005, "Total Petroleum Hydrocarbons" Revision 03, June 2001.
10. Washington DEQ Method NWTPH-HCID, "Hydrocarbon Identification Method for Soil and Water" June 1997.
11. Wisconsin DNR Method PUBL-SW-140, "Modified GRO Method for Determining Gasoline Range Organics" September 1995.
12. Wisconsin DNR Method PUBL-SW-141, "Modified DRO Method for Determining Diesel Range Organics" September 1995.

## + Featured Products

Description	Cat. No.
<b>"M" Purge Traps</b>	
For Tekmar 2000, 4000, and Velocity XPT	<b>20077-U</b>
For Tekmar 3000 and 3100	<b>20079-U</b>
<b>Capillary GC Columns</b>	
VOCOL, 30 m x 0.25 mm I.D., 1.5 µm	<b>24205-U</b>
SLB-5ms, 20 m x 0.18 mm I.D., 0.18 µm	<b>28564-U</b>
SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm	<b>28471-U</b>
SLB-5ms, 30 m x 0.25 mm I.D., 1.0 µm	<b>28476-U</b>
<b>Calibration, Surrogate, and Internal Standard Mixes for GRO Analyses</b>	
Underground Storage Tank (UST) BTEX Mix <i>6 analytes, each at 200 µg/mL in methanol, 1 mL ampul</i>	<b>48026</b>
Benzene	o-Xylene
Ethylbenzene	m-Xylene
Toluene	p-Xylene
PVOC Mix <i>8 analytes, each at 2000 µg/mL in methanol, 1 mL ampul</i>	<b>47916</b>
Benzene	1,2,4-Trimethylbenzene
Ethylbenzene	1,3,5-Trimethylbenzene
Methyl tert-butyl ether (MTBE)	o-Xylene
Toluene	m-Xylene
GRO Mix <i>9 analytes, each at 2000 µg/mL in methanol, 1 mL ampul</i>	<b>47576-U</b>
Benzene	1,2,4-Trimethylbenzene
Ethylbenzene	2,2,4-Trimethylpentane
3-Methylpentane	o-Xylene
Naphthalene	m-Xylene
Toluene	
EPA GRO Mix <i>9 analytes, at varied concentrations in methanol, 1 mL ampul</i>	<b>47577-U</b>
Benzene, 500 µg/mL	1,2,4-Trimethylbenzene, 1000 µg/mL
Ethylbenzene, 500 µg/mL	2,2,4-Trimethylpentane, 1500 µg/mL
Heptane, 500 µg/mL	o-Xylene, 1000 µg/mL
2-Methylpentane, 1500 µg/mL	m-Xylene, 1000 µg/mL
Toluene, 1500 µg/mL	
UST Modified GRO <i>10 analytes, each at 1000 µg/mL in methanol, 1 mL ampul</i>	<b>48167</b>
Benzene	1,2,4-Trimethylbenzene
Ethylbenzene	1,3,5-Trimethylbenzene
Methyl tert-butyl ether (MTBE)	o-Xylene
Naphthalene	m-Xylene
Toluene	p-Xylene
$\alpha,\alpha,\alpha$ -Trifluorotoluene at 10,000 µg/mL in methanol, 1 mL ampul	<b>47582-U</b>
1-Chloro-4-fluorobenzene at 5000 µg/mL in methanol, 1 mL ampul	<b>48194</b>

## + Related Information

Additional information about this, or other collection and analytical methodologies used for environmental applications, can be found at our environmental web node at [sigma-aldrich.com/environmental](http://sigma-aldrich.com/environmental)

Description	Cat. No.
<b>Calibration, Surrogate, and Internal Standard Mixes for DRO Analyses</b>	
UST Modified DRO <i>10 analytes, each at 1000 µg/mL in hexane, 1 mL ampul</i>	<b>48166</b>
Decane	Hexadecane
Docosane	Octacosane
Dodecane	Octadecane
Eicosane	Tetracosane
Hexacosane	Tetradecane
DRO Calibration Mix <i>12 analytes, each at 500 µg/mL in hexane, 1 mL ampul</i>	<b>861287</b>
Chlorobenzene	Hexadecane
Decane	Octacosane
Docosane	Octadecane
Dodecane	o-Terphenyl
Eicosane	Tetracosane
Hexacosane	Tetradecane
5- $\alpha$ -Androstane at 2000 µg/mL in methylene chloride, 1 mL ampul	<b>48168</b>
o-Terphenyl at 2000 µg/mL in acetone, 1 mL ampul	<b>48169</b>
o-Terphenyl at 10,000 µg/mL in methylene chloride, 1 mL ampul	<b>47580-U</b>
<b>Calibration Standard Mix for TPH Analyses</b>	
TPH Mixture 3 <i>17 analytes, each at 1000 µg/mL in carbon disulfide, 1 mL ampul</i>	<b>861394-U</b>
Decane	Octacosane
Dodecane	Octadecane
Dotriacontane	Octane
Eicosane	Tetracontane
Heptane	Tetracosane
Hexadecane	Tetradecane
Hexane	Tetratetracontane
Hexatriacontane	Undecane
Nonane	
<b>Calibration Standard Mix for Aromatic Analyses</b>	
EPA TCL Polynuclear Aromatic Hydrocarbons Mix <i>16 analytes, each at 2000 µg/mL in methylene chloride:benzene (1:1), 1 mL ampul</i>	<b>48905-U</b>
Acenaphthene	Chrysene
Acenaphthylene	Dibenz[a,h]anthracene
Anthracene	Fluoranthene
Benz[a]anthracene	Fluorene
Benzo[b]fluoranthene	Indeno[1,2,3-cd]pyrene
Benzo[k]fluoranthene	Naphthalene
Benzo[g,h,i]perylene	Phenanthrene
Benzo[a]pyrene	Pyrene

### Did you know...?

A new oil spill landing page ([sigma-aldrich.com/oilspill](http://sigma-aldrich.com/oilspill)) on the Sigma-Aldrich web site contains a substantial collection of numerous products and information related to the many sample collection and analytical challenges faced by field workers and laboratory analysts.



Gas Chromatography

# New Adapter for On-the-Fly Sampling During Distillation

## Contributed Article

The following was generated with the assistance of an outside source using Sigma-Aldrich products. Technical content was generated and provided by:

Thomas J. Bruno<sup>1</sup> and James J. Brien<sup>2</sup>

1. National Institute of Standards and Technology (NIST), Thermophysical Properties Division, Boulder, Colorado, USA
2. Sigma-Aldrich, Milwaukee, Wisconsin, USA

*mike.buchanan@sial.com*

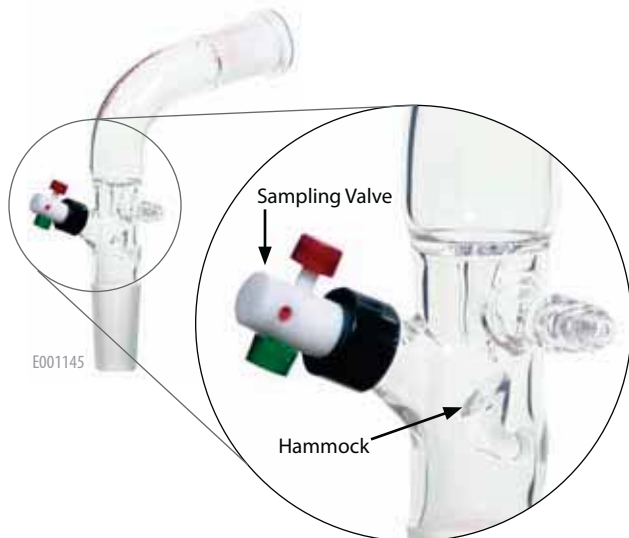
## Introduction

Distillation is the most widely used bulk separation method used in the laboratory as well as industry. Beyond purification, it is widely used to characterize complex fluids (such as fuels) through measurement of the distillation curve, a plot of the boiling temperature against volume distilled. A common theme in both of these applications is the desire to understand the composition. In purification, the goal is to monitor the distillation progress, and in fluid characterization, one seeks to relate the composition to the temperature data.

## Features and Benefits

A distillate sampling adapter (**Figure 1**) installed following a condenser or distillation column can provide this important capability without the need for cumbersome, expensive, and often unreliable fraction collectors. The flow of the distillate is focused to drop into a 0.05 mL “hammock” that is positioned directly below the flow path. The sampling port, equipped with a vacuum tight valve, allows access to the hammock with a

**Figure 1. Distillate Sampling Adapter**



standard chromatographic syringe, through a septum. To sample the distillate, one simply positions the chromatographic syringe, preferably equipped with a blunt tipped needle, in the well of the hammock. It is a simple matter to withdraw samples as the distillation progresses. The sample can then be directly injected into the gas chromatograph or spectrometer, or injected into an autosampler vial for analysis later. Indeed, any analytical technique that is applicable for liquid samples ranging in volume from 1 to 50 microliters can be used to characterize the distillate.

## Application Examples

This adapter has been used for many complex fluid analyses, including gasolines (including oxygenates and bio-derived alternatives), diesel fuels (including oxygenates and biodiesel fuels), rocket kerosenes, jet fuels, crude oils (including bio-crudes), transformer fluids, waste oils, and arson accelerants. Some of the analytical techniques applied to distillate fraction analysis have included gas chromatography (with mass selective, flame ionization, and chemiluminescence detection), FTIR spectroscopy, Karl Fischer coulombic titrimetry, and refractometry. The ability to easily and quickly couple quantitative analysis with the distillation opens the door to thermochemical determinations such as the enthalpy of combustion of fuels, as a function of distillate cut. The adapter has also been used to measure corrosivity of crude oil fractions, with a copper coupon test performed at various distillate cuts.

## Conclusion

The distillate sampling adapter is offered with several joint options and is designed to work with existing modular distillation glassware. It is simply a matter of replacing the existing vacuum adapter with a distillate sampling adapter. No further modification to the set-up is required. The simple concept eliminates the need for cumbersome, expensive, and often unreliable fraction collectors.

## References

1. T.J. Bruno, L.S. Ott, B.L. Smith, and T.M. Lovestead, “Complex Fluid Analysis with the Advanced Distillation Curve Approach” *Anal. Chem.* 82 (2010) p. 777.
2. T.J. Bruno, L.S. Ott, T.M. Lovestead, and M.L. Huber, “The Composition Explicit Distillation Curve Technique: Relating Chemical Analysis and Physical Properties of Complex Fluids” *J. Chromatogr. A* 1217 (2010) p. 2703.
3. T.J. Bruno, L.S. Ott, T.M. Lovestead, and M.L. Huber, “Relating Complex Fluid Composition and Thermophysical Properties with the Advanced Distillation Curve Approach” *Chemical Eng. Tech.* 33 (2010) p. 363.

## + Featured Products

Description	Cat. No.
Distillate sampling adapter, with vacuum connection and PTFE valve, 14/20 joint	<b>Z569895</b>
Distillate sampling adapter, with vacuum connection and PTFE valve, 24/40 joint	<b>Z569909</b>
Distillate sampling adapter, with vacuum connection and PTFE valve, 29/32 joint	<b>Z569917</b>

## + Related Products

Description	Cat. No.
Replacement septa for Mininert® valves, pack of 50	<b>33310-U</b>
Septum inserter for Mininert valve	<b>33311</b>
Hamilton® 701SNR syringe, 10 µL, 22s gauge blunt tip needle	<b>58380-U</b>

## Did you know...?



Sigma-Aldrich, through its Aldrich brand, manufactures a very large selection of distillation glassware, including adapters, apparatus, condensers, columns, flasks, heads, kits, and traps. For complete product and ordering information, see the Labware catalog, or visit [sigma-aldrich.com/glass](http://sigma-aldrich.com/glass)

Gas Chromatography

# Detailed Hydrocarbon Analysis (DHA) Using Gas Chromatography (GC)

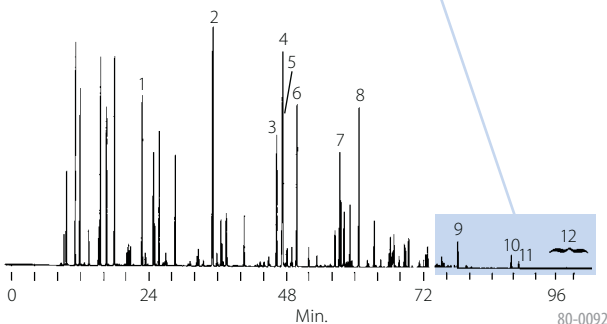
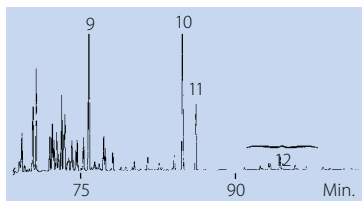
Michael D. Buchanan  
mike.buchanan@sial.com

Detailed hydrocarbon analysis (DHA) of petroleum samples is commonly performed by gas chromatography (GC) using a Petrocol™ capillary GC column. This family of columns is characterized by high efficiency and great reproducibility. The chromatogram shown in **Figure 1** was obtained by analyzing a petroleum reformat on a 100 m Petrocol DH column.

**Figure 1. Petroleum Reformat**

column: Petrocol DH, 100 m x 0.25 mm I.D., 0.50 µm (24160-U)  
oven: 35 °C (15 min.), 2 °C/min. to 200 °C (30 min.)  
det.: FID  
injection: 0.10 µL, 100:1 split

1. Benzene
2. Toluene
3. Ethylbenzene
4. m-Xylene
5. p-Xylene
6. o-Xylene
7. 1-Methyl-3-ethylbenzene
8. 1,2,4-Trimethylbenzene
9. Naphthalene
10. 2-Methylnaphthalene
11. 1-Methylnaphthalene
12. Dimethylnaphthalenes



## + Featured Product

Description	Cat. No.
Petrocol DH, 100 m x 0.25 mm I.D., 0.50 µm	<b>24160-U</b>

## + Related Products

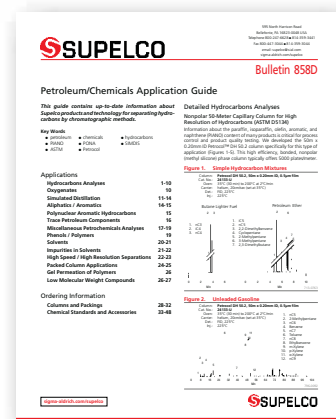
Description	Cat. No.
Petrocol DH 50.2, 50 m x 0.20 mm I.D., 0.50 µm	<b>24133-U</b>
Petrocol DH 150, 150 m x 0.25 mm I.D., 1.0 µm	<b>24155</b>

## + Related Information

To learn more about the use of GC for the analysis of petroleum samples, request **Bulletin 858 *Petroleum/Chemical Application Guide***, T109858 (AYD).

To obtain a no-charge copy of this 48-page bulletin, contact Technical Service by telephone at 800-359-3041 (US and Canada only) or 814-359-3041, or by email at [techservice@sial.com](mailto:techservice@sial.com).

Alternatively, it can be downloaded from our web site at [sigma-aldrich.com/gc](http://sigma-aldrich.com/gc)



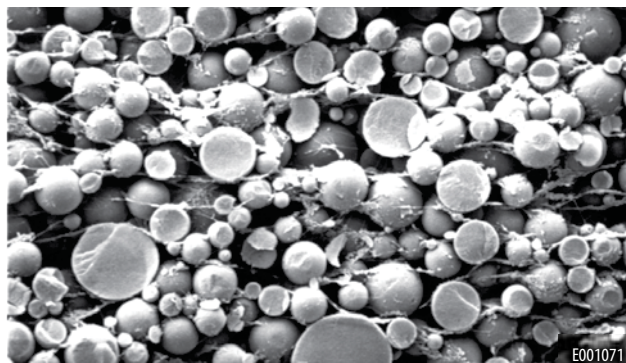
# High Efficiency Extractions Using Empore Solid Phase Extraction Products

Michael Monko  
michael.monko@sial.com

Empore® membrane Solid Phase Extraction (SPE) technology is made up of SPE particles tightly enmeshed within a network of inert PTFE fibrils. This fabrication process results in a highly dense and uniform extraction medium that offers distinct advantages over traditional sorbent/packed-bed SPE products. These advantages include smaller bed weights, shorter analyte to pore diffusion paths, and more efficient extractions.

Reduced SPE bed mass directly affects the volume of elution solvent necessary to efficiently desorb your analytes of interest. The lower the bed weight, the less solvent required. This presents some advantages; such as minimizing eluate evaporation time or allowing the direct injection of the SPE eluate. The dense and uniform extraction medium also minimizes channeling and voiding in the sorbent bed. This results in efficient mass-transfer kinetics and faster flow rates, and also eliminates SPE fines, thereby improving HPLC column and instrument life. These features and advantages allow a user to save both time and money, using Empore products for routine SPE cleanup.

Figure 1. Microscopic View of Empore SPE Disks



Empore SPE disks made up of 90% SPE particles and 10% PTFE, (by weight)

## Did you know...?

Empore SPE products are available in standard solid phase extraction formats. Many phase chemistries can be purchased in disk, cartridge, or 96-well formats. Please visit [sigma-aldrich.com/empore](http://sigma-aldrich.com/empore) for a complete listing of products and literature on Empore SPE.



## + Featured Products

Description	Cat. No.
Empore C18-SD cartridge 4 mm, 1 mL, pk. 100	<b>66871-U</b>
Empore C18-SD cartridge 7 mm, 3 mL, pk. 50	<b>66872-U</b>
Empore UR-SD (universal resin) cartridge 7 mm, 3 mL, pk. 50	<b>66874-U</b>
Empore C18 96-well plate 5.5 mm, 1.2 mL	<b>66875-U</b>
Empore UR 96-well plate 5.5 mm, 1.2 mL	<b>66877-U</b>
Empore C18 disks, 47 mm, pk. 20	<b>66883-U</b>
Empore oil and grease disks, 47 mm, pk. 20	<b>66887-U</b>
Empore cation disks, 47 mm, pk. 20	<b>66889-U</b>

TRADEMARKS: 3M, Empore – 3M Company; Aldrich, Ascendis, Astec, Carboxen, CHIRALDEX, CHIROBIOTIC, CHROMASOLV, DEX, Petrocol, SLB, Supel, Supelco, VOCOL – Sigma-Aldrich Biotechnology LP; FocusLiner – SGE Internationals Pty Ltd.; Fused-Core – Advanced Materials Technology, Inc.; Gastight, Hamilton, Microliter – Hamilton Co.; Mininert – Valco Instruments Co., Inc.; CD Vial, QuikCap – QIS, Inc.; ROCKSTAR – ROCKSTAR, Inc.;

Visit Supelco at the following tradeshow:

## 2010 Gulf Coast Conference

Booth # 1004

October 12-13

Moody Gardens Convention Center  
Galveston, Texas

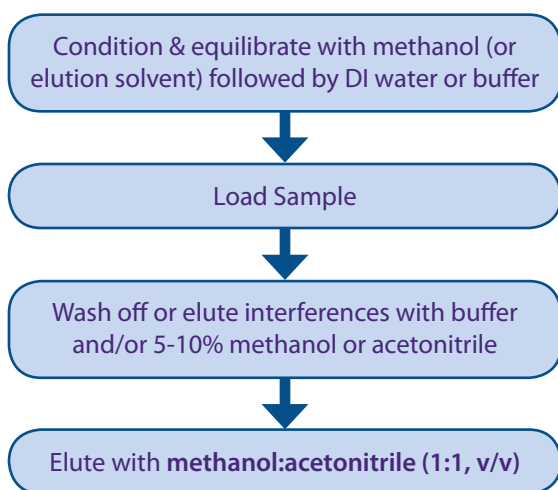
# Supel-Select HLB Solid Phase Extraction

## Increased Savings Without Sacrificing Performance or Quality

Michael Monko  
michael.monko@sial.com

Supelco Supel™-Select HLB SPE is a hydrophilic modified styrene-based polymer developed for the solid phase extraction of a broad range of compounds from aqueous samples. The polymer is available in standard SPE cartridge format as well as 96-well plates. The retention mechanism is based primarily on reversed-phase; however, because the polymer is hydrophilic modified it is also appropriate for more polar compounds. This dual selectivity creates a Hydrophobic Lipophilic Balance (HLB). It's this broad selectivity that allows Supel-Select HLB to be used in generic methods for a wide variety of analytes. For most applications, Supel-Select HLB is amenable to generic methodology with minimal effort required to optimize recovery, selectivity, and reproducibility (Figure 1).

Figure 1. Generic Method for Supel-Select HLB



As more assays require greater sensitivity, the need for cleaner SPE sorbents becomes a much higher priority. Supel-Select HLB is also quality controlled for both LC-UV and LC-MS extractables. This stringent quality control ensures optimum performance year-to-year, lot-to-lot, and tube-to-tube. We test each and every lot of Supel-Select HLB for analyte recovery, LC-MS & LC-UV cleanliness, particle size, density, pore size, and pore volume. These and other features of Supel-Select HLB result in many benefits; including saving time, money, and headache during method development and routine analysis (Table 1). These benefits ultimately allow for greater savings without sacrificing the performance or quality of your SPE methods.

Table 1. Features and Benefits of Supel-Select HLB

Feature	Advantage/Benefit
Hydrophilic modified styrene polymer	Extract a broad range of analytes using a single sorbent
Generic methodology	Saves time, money, and headache during method development
High capacity	Saves time by using smaller bed weights and smaller elution volumes
Low UV and MS extractables	Better sensitivity in analytical methods
Stringent QC	Better accuracy and precision because of low variability from lot-to-lot

### + Featured Products

Description	Cat. No.
Supel-Select HLB 30 mg, 1 mL, pk 100	54181-U
Supel-Select HLB 60 mg, 3 mL, pk 100	54182-U
Supel-Select HLB 96-well plate, 30 mg/well	575661-U
Supel-Select HLB 96-well plate, 60 mg/well	575662-U

### + Related Information

The Supelco Guide to SPE Methodology is a useful tool whether you're just getting started with Solid Phase Extraction or would like a quick reference guide for your lab. This colorful poster outlines the steps in the SPE process, phase selection, and unique tips for reversed phase, ion-exchange, and normal phase SPE.

To request your free Guide to SPE Methodology poster, please visit us at: [sigma-aldrich.com/posters-cds](http://sigma-aldrich.com/posters-cds)



### Did you know...?

Free sample packs of the Supel-Select HLB and many other SPE phases are available to help kick-start your SPE method development. To receive free samples, please visit us and complete the request form at [sigma-aldrich.com/spe-samples](http://sigma-aldrich.com/spe-samples)



Sample Preparation

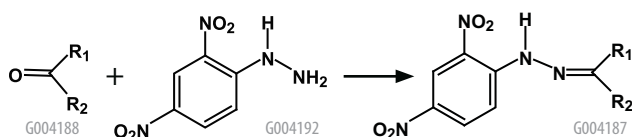
# BPE-DNPH: A More Effective Device for Sampling Airborne Carbonyls Under High Humidity Conditions

Shigehisa Uchiyama<sup>1</sup>, Jamie Desorcie, and Kristen Schultz  
 kristen.schultz@sial.com

1. National Institute of Public Health  
 2-3-6, Minami, Wako City, Saitama 351-0197, Japan

Derivatization with 2,4-dinitrophenylhydrazine (DNPH), followed by HPLC analysis, is a widely used selective and sensitive method for the measurement of carbonyl compounds in air (Figure 1). However, ozone is a known chemical interference of the method.

Figure 1. Measurement of Carbonyl Compounds in Air



Ozone interferes negatively at high concentrations by reacting with DNPH and its carbonyl derivatives (2,4-DNPhyrazones) that are formed in the sampling cartridge.

Ozone scrubbing cartridges are available that contain potassium iodide. These are placed before the DNPH cartridge in the air-sampling train and trap ozone before it can interfere with the reaction between DNPH and carbonyl compounds. However, potassium iodide can become wetted under high humidity conditions. The wetted potassium iodide can trap carbonyl compounds before they reach the DNPH sampler. In addition, wet potassium iodide can migrate into the DNPH sampler and generate unknown compounds.

An alternative ozone trap to hydroscopic potassium iodide is 1,2-bis(2-pyridyl)ethylene (2-BPE). 2-BPE is unaffected by atmospheric moisture and removes ozone via the formation of pyridine-2-aldehyde as outlined in Figure 2.

Figure 2. 2-BPE is Unaffected by Atmospheric Moisture and Removes Ozone via the Formation of Pyridine-2-aldehyde

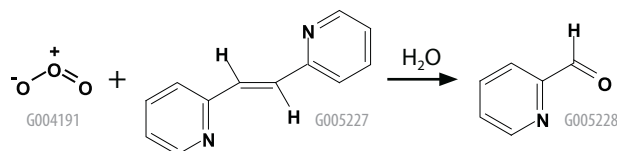


Table 1. Concentrations of Formaldehyde and Acetaldehyde Measured in Ambient Air Collected by a BPE-DNPH Cartridge and a DNPH Cartridge Coupled to a KI Ozone Scrubber

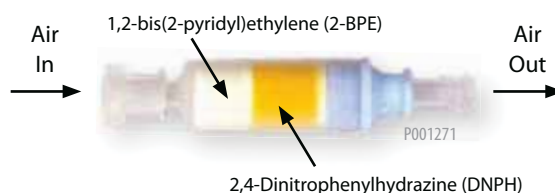
Date (2009)	RH (%)	Ozone**	Formaldehyde*		Acetaldehyde*	
			2-BPE	KI	2-BPE	KI
February 21-22	44	43	2.2	2.1	2.0	2.0
March 20-21	53	63	1.8	1.5	1.9	1.9
April 25-26	97	74	2.1	0.9	1.9	0.8
May 30-31	92	47	1.8	1.0	1.2	0.5

\*Concentration units are  $\mu\text{g}/\text{m}^3$ .

\*\*Ozone concentration is the average over the sampling period reported by a local air monitoring station.

The BPE-DNPH sampler is a 2-bed cartridge consisting of silica particles impregnated with 2-BPE and DNPH (Figure 3). Air is first drawn through the 2-BPE bed, which can trap up to 0.24 mg ozone. Airborne carbonyls pass unimpeded through the 2-BPE bed and are trapped in the second bed by the DNPH-coated silica particles.

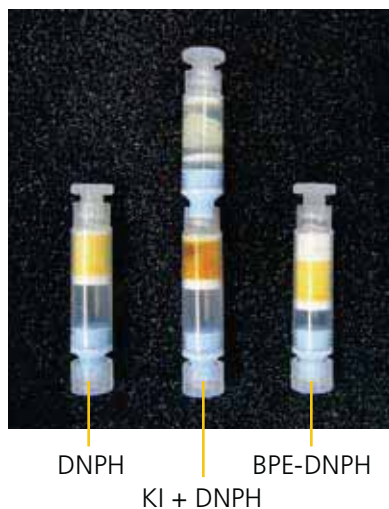
Figure 3. BPE-DNPH Cartridge



The effectiveness of 2-BPE as an ozone scrubber, particularly under high humidity conditions, is demonstrated by the data presented in Table 1 (Reference 1). Ambient air was sampled during various time periods during 2009 in Chiba City, Japan. Two sampling devices were used – a BPE-DNPH cartridge and a conventional DNPH cartridge coupled to a potassium iodide (KI) ozone scrubber. Formaldehyde and acetaldehyde concentrations were measured using DNPH protected by each ozone-scrubbing material. Average ozone concentration, reported by a local air monitoring station, is also listed.

When the relative humidity was close to 50% (February 21-22 and March 20-21), similar concentrations of formaldehyde and acetaldehyde were measured using either 2-BPE or KI to scrub ozone. Alternatively, much higher concentrations of formaldehyde and acetaldehyde are measured using the BPE-DNPH cartridge when the relative humidity is above 90% (April 25-26 and May 30-31). It is believed that wet potassium iodide acts as a trap for formaldehyde and acetaldehyde and prevents much of these materials from reaching the DNPH sampling reagent. In addition, potassium iodide was observed to migrate into the DNPH cartridge, which resulted in a color change of yellow to reddish brown. This is depicted in the photographs in Figure 4.

Figure 4. Appearance of a DNPH Cartridge, a KI Ozone Scrubber Coupled to a DNPH Cartridge and a Dual-Bed BPE-DNPH Cartridge Following Exposure to High Humidity Air (>90% RH)



## Conclusion

The dual-bed BPE-DNPH cartridge is preferred over a DNPH cartridge coupled to a potassium iodide ozone scrubber for sampling carbonyls in high humidity, ambient air. Unlike wetted potassium iodide, 2-BPE does not trap carbonyl compounds before they are able to reach the DNPH sampling medium. Also, removal of KI from the sampling train eliminates the possibility of KI migration to the DNPH bed where it may interfere with the carbonyl-DNPH derivatization reaction.

## Reference

1. Uchiyama, S., Naito, S., Matsumoto, M., Inaba, Y., Kunugita, N. *Anal. Chem.* 2009, 81, 6552.

## + Featured Products

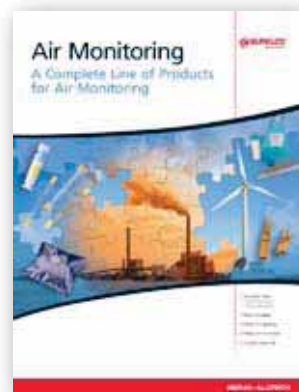
Description	Pkg. Size	Cat. No.
BPE-DNPH, 130/270 mg cartridge	10	54278-U
BPE-DNPH, 130/270 mg cartridge	50	54279-U

## + Related Products

Description	Pkg. Size	Cat. No.
<b>Analytical Columns, 15 cm x 4.6 mm I.D., 3 µm</b>		
Ascentis C18	1	581322-U
Ascentis RP-Amide	1	565322-U
<b>Calibration Standards</b>		
TO11/IP-6A Aldehyde/Ketone DNPH Mix (15 µg/mL as carbonyl)	1	47285-U
Formaldehyde-DNPH, 1 mL (100 µg/mL as aldehyde)	1	47177
Formaldehyde-DNPH, 1 mL (100 µg/mL as aldehyde)	5	4M7177
Acetaldehyde-DNPH, 1 mL (1000 µg/mL as aldehyde)	1	47340-U
Acetaldehyde-DNPH, 1 mL (1000 µg/mL as aldehyde)	5	4M7340-U

## + Related Information

For more information regarding our products and select applications, [visit sigma-aldrich.com/air\\_monitoring](http://www.sigma-aldrich.com/air_monitoring) and request our *Air Monitoring Brochure*, T408103 (KQV).



## Technical Seminar at EAS, Somerset, NJ

# Understanding Karl Fischer Titration

Date: **Tuesday, November 16, 2010**  
 Time: **1:00 pm – 3:00 pm**  
 Location: **McDivitt Room at the Garden State Convention & Exhibit Center**  
 Speaker: **Doug Clark, Sigma-Aldrich**

The Karl Fischer titration is the most versatile and generally accepted analytical method for determining water content. It is independent of the sample's state of matter and is therefore suitable for the analysis of solids, liquids, and gases. Critical to the accurate determination of water content is that the sample be soluble in the solvent mixture, the working pH of the system is not affected by the sample, and side reactions are avoided.

This seminar addresses the development and principles behind Karl Fischer analysis, the differences between the volumetric and coulometric approaches to water determination, good lab practices and proper technique, modifications to improve solubility, maintaining proper pH control, and problematic functional groups and the specialty reagents that allow for their analysis. Use of various standards to verify that the system is functioning properly and the benefits of routine maintenance will also be discussed.

Questions are always welcome and will be encouraged throughout the seminar.

To register for this seminar, visit our website: [sigma-aldrich.com/analytical-seminars](http://sigma-aldrich.com/analytical-seminars)



## SPME Fast Fit Assemblies for Automated Fiber Exchange

The SPME Fast Fit Assemblies (FFA) are a new configuration of SPME fibers allowing the automated exchange of SPME fibers by the Multi Fiber EXchanger (MFX) unit of an autosampler.

The barcoded SPME FFA's in use with the Multi Fiber EXchanger (MFX) system offer the following benefits:

- Analyte polarity range enhancement - no manual switching out of the fiber for extractions using a range of SPME phases.
- Automated screening for optimal selectivity in SPME method development by setting up different SPME phase selectivities.
- Program longer unattended GC-MS autosampler sequences.



### + Featured Products

Description	Qty.	Cat. No.
<b>SPME Fast Fit Assemblies</b>		
100 µm PDMS (fused silica), 23 ga	3	<b>FFA57341-U</b>
100 µm PDMS (fused silica), 24 ga	3	<b>FFA57301</b>
85 µm Carboxen™, 23 ga	3	<b>FFA57295-U</b>
50/30 µm DVb/CAR/PDMS, 24 ga	3	<b>FFA57329-U</b>

For a complete list of SPME Fast Fit Assemblies, visit [sigma-aldrich.com/spme](http://sigma-aldrich.com/spme)

The Multi Fiber EXchanger was developed and is produced by Chromline s.r.l. Prato/Italy. SPME Multi-Fiber stations for 3 fibers or for 25 fibers are available from GERSTEL GmbH & Co. KG and fit CTC type autosamplers.



For more MFX product information, refer to the GERSTEL website: [www.gerstel.com/en/multi-spme-fiber-exchange-mfx.htm](http://www.gerstel.com/en/multi-spme-fiber-exchange-mfx.htm)

## New SPME Devices for Air/Field Sampling

### SPME-Diffusive Fiber Holder

Useful for time-weighted average (TWA) air sampling with SPME. Using the SPME-FFA configuration, this holder operates as a passive sampling device where the fiber is retracted into the piercing needle at a defined distance and functions as a passive sampler whose sensitivity can be adjusted.

### SPME-FFA Field Sampler

This holder allows you to safely transport conditioned sample fibers to and from the lab, even under rough conditions out in the field. The built-in sealing mechanism insures the fibers integrity before and after sampling.

### SPME Storage Containers

Two new devices for safely storing conventional SPME Fibers and SPME-FFA to maintain conditioning or sample integrity.

See our web site [sigma-aldrich.com/spme](http://sigma-aldrich.com/spme) for more details on these new SPME products.



# Maximum Sample Recovery with MRQ30 CD™ Vials

The MRQ30 CD vial is a recent addition to our popular line of CD vials. These 1.2 mL vials are made in the USA of Type 1, Class A borosilicate glass. The precision-formed center well allows maximum sample recovery; with as little as 2 µL of sample remaining in the vial after sample withdrawal.

Work done in our laboratories demonstrates the utility of these vials. We reliably get three 5 µL injections from 20 µL of sample while doing LC-MS analysis. Additionally, our testing shows the MRQ30 CD vials to be comparable to other certified clean vials for LC-MS detectable bleed. Finally, these vials are excellent for use with immersion SPME. Covering a 1 cm SPME fiber in a normal 2 mL autosampler vial requires at least 1 mL of sample. In a MRQ30 CD vial only 200 µL is required.

MRQ30 CD vial kits contain 12 x 32 mm wide-mouth vials and blue QuikCap™ threaded caps with bonded TEF/silicone pre-slit septa.

## Benefits of MRQ30 CD vial kits are:

- No inserts required
- Vial design allows for maximum recovery of samples with residual dead volume as low as 2 µL
- Wide-mouth opening has large target area reducing the possibility of bent needles
- Compatible with most autosamplers
- Ideal for immersion SPME sampling
- Clean enough for LC-MS analysis



E001143

MRQ30 CD vials for microsampling compliment the already extensive line of chromatography vials and accessories being offered by Supelco Analytical. For more information on these or any other products, email our Technical Service Department at [techservice@sial.com](mailto:techservice@sial.com) or visit [sigma-aldrich.com/vials](http://sigma-aldrich.com/vials)

Description	Pkg. Size.	Cat. No.
MRQ30 Clear Glass CD Vial Kit	100	<b>23187-U</b>

# 6-Unit Multipacks of Hamilton Autosampler Syringes

Advantages of buying 6-unit Multipacks over individual syringes:

- **Cost savings:** The price per syringe is lower
- **Time savings:** Syringes need to be ordered less frequently
- **Space savings:** There is less packaging material than with six individual syringes
- **Lab efficiency:** The analyst can replace damaged or worn syringes immediately without having to wait for a new order



9100205

If you have additional questions, or require help in choosing the correct syringes, please contact Supelco Technical Service at [techservice@sial.com](mailto:techservice@sial.com) or visit [sigma-aldrich.com/syringes](http://sigma-aldrich.com/syringes)

	Volume (µL)	Description	Needle Style	Needle Length (mm)	Cat. No.
<b>For Agilent 7670, 7671, and 7672 Autosamplers</b>					
Hamilton Microliter™ Syringes - Fixed Needle	10	701N	26s gauge, bevel tip	51	<b>20779</b>
Hamilton Microliter Syringes - Removable Needle	10	701RN	26s gauge, bevel tip	51	<b>20793</b>
<b>For Agilent 7673-7683 and 6850 ALS Autosamplers</b>					
Hamilton Microliter Syringes - Fixed Needle	5	75ASN	23s gauge, cone tip	43	<b>21315</b>
	5	75ASN	26s gauge, cone tip	43	<b>21314</b>
	5	75ASN	23s-26s gauge, cone tip	43	<b>24571</b>
	10	701ASN	23s gauge, cone tip	43	<b>21317</b>
	10	75ASN	26s gauge, cone tip	43	<b>21316</b>
	10	75ASN	23s-26s gauge, cone tip	43	<b>24574</b>
Hamilton Gastight® Syringes - Fixed Needle	5	175ASN	23s gauge, cone tip	43	<b>26700-U</b>
	5	175ASN	23s-26s gauge, cone tip	43	<b>24577</b>
	10	1701ASN	23s gauge, cone tip	43	<b>26701</b>
	10	1701ASN	23s-26s gauge, cone tip	43	<b>24580</b>



# LC-MS CHROMASOLV Pre-blended Solvents and Water

## High Purity for Accurate Analysis

Shyam Verma  
shyam.verma@sial.com

In LC-MS analysis, solvent with impurities commonly result in extraneous peaks and unstable baselines. Solvent-derived impurities do not condition out over time and can interfere in the analysis in multiple ways, such as: a) collect on head of HPLC column and elute as a distinct peak or as baseline rise, b) cause general elevation in baseline, lowering sensitivity of analysis, c) foul or damage sensitive instrument components, and d) cause cluster ion formation that prevents reliable identification and quantification.

In order to minimize the background and artifacts in LC-MS analysis, highly specified solvents are spiked with ultra pure salts and acids. These additives improve the chromatographic peak shape and optimize ionization in the MS interface. The most commonly used LC-MS solvents are acetonitrile, methanol and water, and most common additives include trifluoroacetic acid (TFA), formic acid, acetic acid and ammonium acetate.

The mobile phase composition also plays a critical role in the success of an LC-MS experiment. Precise formulations provide accurate and reproducible results. Sigma-Aldrich offers pre-blended solutions of most commonly used LC-MS mobile phases, prepared with precision and unsurpassed attention to quality. These pre-blended solvents offer: 1) time savings, 2) accurate composition, and 3) minimized baseline and artifacts.

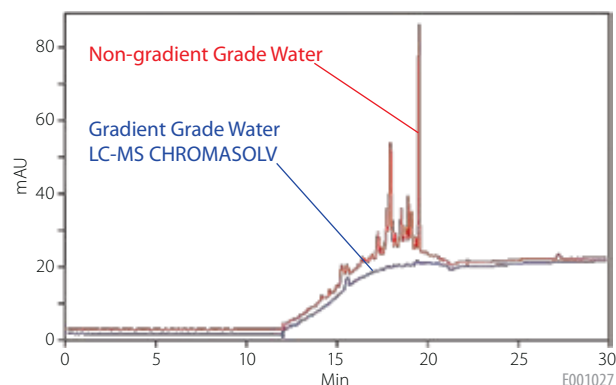
High purity CHROMASOLV® Water is suitable for both gradient HPLC and MS applications. It offers tremendous advantages over other non-gradient grade water available in the market. It can be used in both UV and MS detection methods without any compromise. **Figure 1** clearly shows differences between LC-MS CHROMASOLV Water and a non-gradient grade water.

For additional information on these products, please visit our website: [sigma-aldrich.com/lc-ms-solvents](http://sigma-aldrich.com/lc-ms-solvents)



E001104

Figure 1. UV Gradient at 205 nm, LC-MS Water (Cat. No. 39253) and Non-gradient Grade Water



### + Featured Products

Description	Cat. No.
LC-MS CHROMASOLV Water	<b>39253</b>
<b>LC-MS CHROMASOLV Solvent Blends</b>	
Acetonitrile with 0.1% TFA	<b>34976</b>
Methanol with 0.1% TFA	<b>34974</b>
Acetonitrile with 0.1% formic acid	<b>34668</b>
Acetonitrile with 0.1% ammonium acetate	<b>34670</b>
Acetonitrile with 0.1% formic acid and 0.1% TFA	<b>34676</b>
Water with 0.1% TFA	<b>34978</b>

### + Related Products

Description	Cat. No.
<b>LC-MS CHROMASOLV Solvents</b>	
Acetonitrile	<b>34967</b>
Methanol	<b>34966</b>
2-Propanol	<b>34965</b>
Ethyl acetate	<b>34972</b>
<b>LC-MS CHROMASOLV Mobile Phase Additives*</b>	
Trifluoroacetic acid	<b>40967</b>
Formic acid	<b>56302</b>
Acetic acid	<b>49199</b>
Ammonium formate	<b>55674</b>

\* puriss p.a.



- High purity – *microfiltered (0.2 μm)*
- Improved analyte recoveries
- Longer shelf life – *packed under inert gas*
- Specifications matching USP, Ph.Eur.& ICH guidelines



## GC-Headspace Solvents

Specifically developed and optimized for sensitive static GC-Headspace analysis of volatile organics

For additional information, visit us at [sigma-aldrich.com/gc-hs](http://sigma-aldrich.com/gc-hs)  
To order, call: 800-247-6628 or 814-359-3441.

Description	Size	Cat. No.
1,3-Dimethyl-2-imidazolidinone	1 L	67484
N,N-Dimethylacetamide	1 L	44901
Dimethyl sulfoxide	1 L	51779
N,N-Dimethylformamide	1 L	51781
Water	1 L	53463



- Products sorted by GC, HPLC, Chiral and TLC techniques
- Reagents also listed by “Application”
- Vials, syringes and other useful items for derivatization reactions
- Up-to-date application information and references



## Derivatization Reagents Brochure

Listing over 400 Derivatization Reagents

To order your free copy go to [sigma-aldrich.com/derivatization](http://sigma-aldrich.com/derivatization), call 800-359-3041 (US and Canada) or 814-359-3041, or email [techservice@sial.com](mailto:techservice@sial.com).



## New Analytical Standards for R-(+)-Lipoic Acid

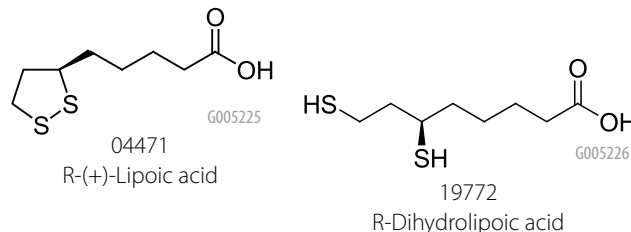
R-(+)-Lipoic acid (RLA) is an important cofactor in aerobic metabolism. It is part of several enzyme complexes including the pyruvate dehydrogenase complex (PDHC) and  $\alpha$ -keto-glutarate dehydrogenase complex (KDHC). During reactions catalysed by these enzymes, RLA is reduced to dihydrolipoic acid. Both compounds are found in many foods, especially organ meats, spinach, broccoli and yeast extracts.

RLA behaves like an antioxidant by activating cytoprotective detoxification enzyme pathways. This action also protects organs against the toxic effects of a wide range of chemicals. Because of these actions, RLA is widely used as a food supplement as well as therapeutically.

Although only the R form is bioactive, the cheaper racemic mixture (R/S-LA) has often been used in supplements and in therapeutic agents. Evidence shows the S enantiomer of lipoic acid inhibits some of the functions of the R enantiomer.

Sigma-Aldrich now offers new analytical standards for the naturally occurring R enantiomer of lipoic acid and its reduced form R-Dihydrolipoic acid.

Figure 1. Structures of R-(+)-Lipoic acid and R-Dihydrolipoic acid



Description	Qty.	Cat. No.
R-(+)-Lipoic acid	100 mg	<b>04471</b>
R-Dihydrolipoic acid	100 mg	<b>19772</b>

## New Neat and Single-component Solutions for a Wide Variety of Applications

Sigma-Aldrich actively maintains a large line of high-purity neat and single-component solutions for environmental, air monitoring, food and beverage, and petroleum/petrochemical applications. These multipurpose materials are appropriate for preparing your own calibration standards or spiking a commercially prepared standard. Many of these chemicals have a purity of at least 99+%. Neat standards are tested for purity and identity. Single-component solutions are evaluated for purity, identity and concentration.

A partial listing of the many new neats and single-component solutions recently added to support your application work is shown here. To view a complete list, please visit our website:

[sigma-aldrich.com/standards](http://sigma-aldrich.com/standards)

Visit Supelco at the following tradeshow:

### EAS 2010 – Eastern Analytical Symposium

**Booth # 318**

November 15-17

Garden State Convention Center  
Somerset, New Jersey

Description	Qty.	Cat. No.
Aflatoxin B1	5 mg	<b>32754</b>
Aflatoxin G2	5 mg	<b>32757</b>
Auramin	25 mg	<b>51362</b>
Azimsulfuron	25 mg	<b>32521</b>
Beflubutamid	100 mg	<b>32866</b>
Bifenazate	50 mg	<b>32504</b>
Carminic acid	25 mg	<b>11298</b>
Cimaterol	10 mg	<b>32568</b>
Cimaterol- <i>d7</i>	10 mg	<b>32569</b>
Citrus Red 4	25 mg	<b>89774</b>
3,5-Dihydroxyhydrocinnamic acid	10 mg	<b>56452</b>
Fumonison B3, 50 µg/mL in acetonitrile:water	1 mL	<b>32606</b>
5-Heptadecylresorcinol (n=15)	10 mg	<b>97001</b>
Isotianil	100 mg	<b>32524</b>
Malachite Green Chloride	25 mg	<b>38800</b>
Oil Orange SS	25 mg	<b>79285</b>
Patulin	5 mg	<b>32759</b>
Sterigmatocystin	5 mg	<b>32609</b>
Sunset Yellow	25 mg	<b>68775</b>
Toluidine	25 mg	<b>59659</b>
5-Tridecylresorcinol (n=11)	10 mg	<b>56453</b>
Triflumizole	100 mg	<b>32611</b>
Zoxamide	50 mg	<b>32501</b>



## Premixed Sterol Reference Standards

Sterols make up the majority of the unsaponifiable matter found in vegetable and animal fats. Animal fats contain cholesterol, whereas most vegetable oils contain only traces of this sterol. The other sterols found in plants are collectively referred to as phytosterols.

Cholesterol is an important component for the manufacture of bile acids, steroid hormones and fat soluble vitamins. Plant sterols, such as  $\beta$ -sitosterol, play an important role in the lowering of high cholesterol levels.

Sigma-Aldrich offers six individual plant and animal sterol standards for analytical use. The sterol standards are prepared in chloroform and packaged under nitrogen in small 1 mL volumes to minimize waste and disposal problems. We analyze each sterol by capillary GC to determine lot purity. A Certificate of Composition accompanies each individual standard.

Description	CAS No.	Concentration	Qty.	Cat. No.
5- $\alpha$ -Cholestane	481-21-0	10 mg/mL in chloroform	1 mL	<b>47124</b>
Cholestanol	80-97-7	10 mg/mL in chloroform	1 mL	<b>47129</b>
Cholesterol	57-88-5	10 mg/mL in chloroform	1 mL	<b>47127-U</b>
Ergosterol	57-87-4	10 mg/mL in chloroform	1 mL	<b>47130-U</b>
$\beta$ -Sitosterol	83-46-5	100 $\mu$ g/mL in chloroform	1 mL	<b>47133</b>
Stigmasterol	83-48-7	10 mg/mL in chloroform	1 mL	<b>47132</b>

## C18:1 cis/trans Fatty Acid Methyl Esters (FAMES)

Supelco offers both cis and trans FAME standard reference materials for the analysis of cis and trans monounsaturated octadecenoic (C18:1) fatty acids in food matrices. These standards have been thoroughly evaluated to establish purity

and identity. A Certificate of Analysis is supplied with each quantitative single-component solution, and a data sheet is included with each mix.

Description	CAS No.	Qty.	Cat. No.
<b>C18 FAME Individual Solutions</b>			
<i>10 mg/mL each in heptane</i>			
cis-6-Octadecenoic (Petroselinic) acid methyl ester	2777-58-4	1 mL	<b>47198</b>
trans-6-Octadecenoic (Petroselaicid) acid methyl ester	--	1 mL	<b>47199</b>
cis-9-Octadecenoic (Oleic) acid methyl ester	112-62-9	1 mL	<b>46902-U</b>
trans-9-Octadecenoic (Elaidic) acid methyl ester	1937-62-8	1 mL	<b>46903</b>
cis-11-Octadecenoic (cis-Vaccenic) acid methyl ester	1937-63-9	1 mL	<b>46904</b>
trans-11-Octadecenoic (trans-Vaccenic) acid methyl ester	6198-58-9	1 mL	<b>46905-U</b>
cis-9,12-Octadecadienoic acid methyl ester	112-63-0	1 mL	<b>46950-U</b>
trans-9,12-Octadecadienoic acid methyl ester	2566-97-4	1 mL	<b>46951-U</b>
cis-5,8,11,14,17-Eicosapentaenoic acid methyl ester	2734-47-6	1 mL	<b>47571-U</b>
cis-4,7,10,13,16,19-Docosahexaenoic acid methyl ester	2566-90-7	1 mL	<b>47570-U</b>
cis-7,10,13,16,19-Docosapentaenoic acid methyl ester	108698-02-8	1 mL	<b>47563-U</b>

Description	Qty.	Cat. No.
<b>C18 FAME Isomer Mixes</b>		
Linolenic Acid Methyl Ester Isomer Mix <i>10 mg/mL in methylene chloride</i>	1 mL	<b>47792</b>
cis-9, cis-12, cis-15-Octadecatrienoic acid methyl ester, 3 Wt%		
cis-9, cis-12, trans-15-Octadecatrienoic acid methyl ester, 7 Wt%		
cis-9, trans-12, cis-15-Octadecatrienoic acid methyl ester, 7 Wt%		
cis-9, trans-12, trans-15-Octadecatrienoic acid methyl ester, 15 Wt%		
trans-9, cis-12, cis-15-Octadecatrienoic acid methyl ester, 7 Wt%		
trans-9, cis-12, trans-15-Octadecatrienoic acid methyl ester, 15 Wt%		
trans-9, trans-12, cis-15-Octadecatrienoic acid methyl ester, 15 Wt%		
trans-9, trans-12, trans-15-Octadecatrienoic acid methyl ester, 30 Wt%		
Total percent of the three isomers		

Description	Qty.	Cat. No.
<b>C18 FAME Isomer Mixes</b>		
Linoleic Acid Methyl Ester cis/trans Isomer Mix <i>10 mg/mL in methylene chloride</i>	1 mL	<b>47791</b>
trans-9, trans-12-Octadecadienoic acid methyl ester, 50 Wt%		
cis-9, trans-12-Octadecadienoic acid methyl ester, 20 Wt%		
trans-9, cis-12-Octadecadienoic acid methyl ester, 20 Wt%		
cis-9, cis-12-Octadecadienoic acid methyl ester, 10 Wt%		

### + Related Information

For more information on FAME analyses, request a copy of the *Fatty Acid/FAME Application Guide*, T408126 (KUK).

## Sigma-Aldrich® Worldwide Offices

### Argentina

Free Tel: 0810 888 7446  
Tel: (+54) 11 4556 1472  
Fax: (+54) 11 4552 1698

### Australia

Free Tel: 1800 800 097  
Free Fax: 1800 800 096  
Tel: (+61) 2 9841 0555  
Fax: (+61) 2 9841 0500

### Austria

Tel: (+43) 1 605 81 10  
Fax: (+43) 1 605 81 20

### Belgium

Free Tel: 0800 14747  
Free Fax: 0800 14745  
Tel: (+32) 3 899 13 01  
Fax: (+32) 3 899 13 11

### Brazil

Free Tel: 0800 701 7425  
Tel: (+55) 11 3732 3100  
Fax: (+55) 11 5522 9895

### Canada

Free Tel: 1800 565 1400  
Free Fax: 1800 265 3858  
Tel: (+1) 905 829 9500  
Fax: (+1) 905 829 9292

### Chile

Tel: (+56) 2 495 7395  
Fax: (+56) 2 495 7396

### China

Free Tel: 800 819 3336  
Tel: (+86) 21 6141 5566  
Fax: (+86) 21 6141 5567

### Czech Republic

Tel: (+420) 246 003 200  
Fax: (+420) 246 003 291

### Denmark

Tel: (+45) 43 56 59 00  
Fax: (+45) 43 56 59 05

### Finland

Tel: (+358) 9 350 9250  
Fax: (+358) 9 350 92555

### France

Free Tel: 0800 211 408  
Free Fax: 0800 031 052  
Tel: (+33) 474 82 28 88  
Fax: (+33) 474 95 68 08

### Germany

Free Tel: 0800 51 55 000  
Free Fax: 0800 64 90 000  
Tel: (+49) 89 6513 0  
Fax: (+49) 89 6513 1160

### Hungary

Ingyenes telefonszám: 06 80 355 355  
Ingyenes fax szám: 06 80 344 344  
Tel: (+36) 1 235 9063  
Fax: (+36) 1 269 6470

### India

Telephone  
Bangalore: (+91) 80 6621 9400  
New Delhi: (+91) 11 4358 8000  
Mumbai: (+91) 22 2570 2364  
Hyderabad: (+91) 40 4015 5488  
Kolkata: (+91) 33 4013 8003  
Fax  
Bangalore: (+91) 80 6621 9550  
New Delhi: (+91) 11 4358 8001  
Mumbai: (+91) 22 4087 2364  
Hyderabad: (+91) 40 4015 5488  
Kolkata: (+91) 33 4013 8000

### Ireland

Free Tel: 1800 200 888  
Free Fax: 1800 600 222  
Tel: (+353) 402 20370  
Fax: (+353) 402 20375

### Israel

Free Tel: 1 800 70 2222  
Tel: (+972) 8 948 4100  
Fax: (+972) 8 948 4200

### Italy

Free Tel: 800 827 018  
Tel: (+39) 02 3341 7310  
Fax: (+39) 02 3801 0737

### Japan

Tel: (+81) 3 5796 7300  
Fax: (+81) 3 5796 7315

### Korea

Free Tel: (+82) 80 023 7111  
Free Fax: (+82) 80 023 8111  
Tel: (+82) 31 329 9000  
Fax: (+82) 31 329 9090

### Malaysia

Tel: (+60) 3 5635 3321  
Fax: (+60) 3 5635 4116

### Mexico

Free Tel: 01 800 007 5300  
Free Fax: 01 800 712 9920  
Tel: (+52) 722 276 1600  
Fax: (+52) 722 276 1601

### The Netherlands

Free Tel: 0800 022 9088  
Free Fax: 0800 022 9089  
Tel: (+31) 78 620 5411  
Fax: (+31) 78 620 5421

### New Zealand

Free Tel: 0800 936 666  
Free Fax: 0800 937 777  
Tel: (+61) 2 9841 0555  
Fax: (+61) 2 9841 0500

### Norway

Tel: (+47) 23 17 60 00  
Fax: (+47) 23 17 60 10

### Poland

Tel: (+48) 61 829 01 00  
Fax: (+48) 61 829 01 20

### Portugal

Free Tel: 800 202 180  
Free Fax: 800 202 178  
Tel: (+351) 21 924 2555  
Fax: (+351) 21 924 2610

### Russia

Tel: (+7) 495 621 5828  
Fax: (+7) 495 621 6037

### Singapore

Tel: (+65) 6779 1200  
Fax: (+65) 6779 1822

### Slovakia

Tel: (+421) 255 571 562  
Fax: (+421) 255 571 564

### South Africa

Free Tel: 0800 1100 75  
Free Fax: 0800 1100 79  
Tel: (+27) 11 979 1188  
Fax: (+27) 11 979 1119

### Spain

Free Tel: 900 101 376  
Free Fax: 900 102 028  
Tel: (+34) 91 661 99 77  
Fax: (+34) 91 661 96 42

### Sweden

Tel: (+46) 8 742 4200  
Fax: (+46) 8 742 4243

### Switzerland

Free Tel: 0800 80 00 80  
Free Fax: 0800 80 00 81  
Tel: (+41) 81 755 2828  
Fax: (+41) 81 755 2815

### United Kingdom

Free Tel: 0800 717 181  
Free Fax: 0800 378 785  
Tel: (+44) 1747 833 000  
Fax: (+44) 1747 833 313

### United States

Toll-Free: 800 325 3010  
Toll-Free Fax: 800 325 5052  
Tel: (+1) 314 771 5765  
Fax: (+1) 314 771 5757

### Vietnam

Tel: (+84) 3516 2810  
Fax: (+84) 6258 4238

### Internet

[sigma-aldrich.com](http://sigma-aldrich.com)



### Mixed Sources

Product group from well-managed forests, controlled sources and recycled wood or fiber  
[www.fsc.org](http://www.fsc.org) Cert no. SGS-COC-003338  
© 1996 Forest Stewardship Council



*Accelerating Customers'  
Success through Innovation and  
Leadership in Life Science,  
High Technology and Service*

Order/Customer Service (800) 325-3010 • Fax (800) 325-5052  
Technical Service (800) 325-5832 • [sigma-aldrich.com/techservice](http://sigma-aldrich.com/techservice)  
Development/Custom Manufacturing Inquiries **SAFC**® (800) 244-1173  
Safety-related Information [sigma-aldrich.com/safetycenter](http://sigma-aldrich.com/safetycenter)

World Headquarters  
3050 Spruce St.  
St. Louis, MO 63103  
(314) 771-5765  
[sigma-aldrich.com](http://sigma-aldrich.com)