

Hi Tech Sigma-Aldrich Carbons in Space

Chromatography

- Lewatit® MonoPlus® Technology
- LC-MS Mobile Phase Additives

Microbiology

- Carbohydrate Differentiation Discs

Standards

- Mycotoxins
- LC/GC Test Mixes
- Ion Chromatography

Sample Preparation

- Derivatization Kits
- Silver Ion SPE

Titration

- HYDRANAL® Medium K
- New AQUANAL® Spectro 3

New Product Corner

Events



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Dear Reader,



Picture Rainer Walz, PhD, Product Manager Analytical, Sigma-Aldrich

A decade or so ago, if anyone suggested that the internet was just a passing fad, I'm sure they are eating their words today. It doesn't seem like that long ago when I first noticed a web address on an advertisement. Now, the web is often the first place I look for information and news, and I'm sure I'm not in the minority.

Today, successful companies keep pace with their customers' use of the internet, especially in the way they search for information. At Sigma-Aldrich, improvements to our website and its navigation have been ongoing and regular Analytix readers might recall mention of some of its new features. Today, I'd like to present the improvements we've made to the navigation and content of one of the most important analytical product areas.

Analytical Standards Explorer

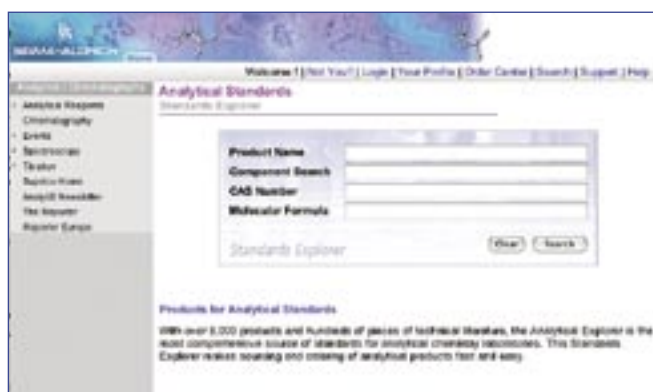
www.sigma-aldrich.com/standards_explorer

Few companies can match our breadth of chemical and reference standards. To make searching our standards faster, easier and more accurate, we've created the Standards Explorer. Using it, as opposed to the general Sigma-Aldrich search engine, you can target a search on a standard and not have to sift through multiple listings that include bulk chemicals. In the near future, we'll be adding capability to perform multiple CAS# searches, and search for standards by agency and method number (e.g. EPA 535).

We know your time is precious, and through our website improvements, and the Analytix newsletter, we are working hard to bring you relevant and interesting product information at your fingertips. Please let us know how we may serve you better. We look forward to hearing from you!

Kind regards,

Rainer Walz, PhD
Product Manager Analytical
Sigma-Aldrich



Picture

New Analytical Standards Explorer
www.sigma-aldrich.com/standards_explorer

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New Product Corner

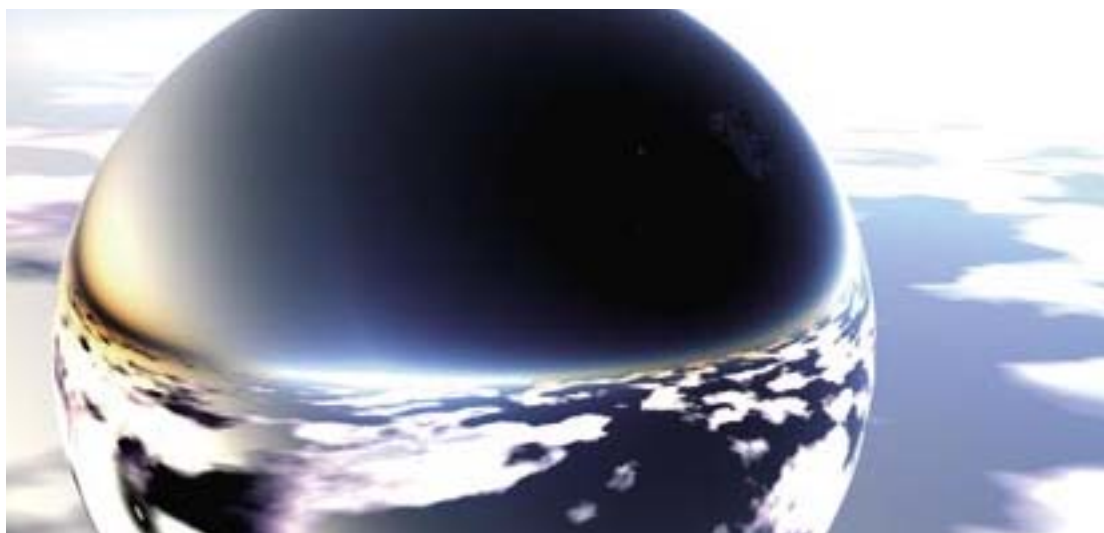
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Carbon Adsorbent Kits from Supelco A cost-effective tool for the selection of the right adsorbent

By Michael D. Buchanan, Product Manager, Gas Separations ... mbuchanan@sial.com



Carbon Adsorbent Research

Supelco's commitment to carbon adsorbent research and product development spans more than two decades. With the aim being the understanding of how the physical and chemical properties of the adsorbents affect performance characteristics, our efforts have been broad in scope, ranging from purification process development to research focusing on the thermodynamic and kinetic properties of both existing and novel adsorbents. The knowledge gained from this fundamental research has led to innovative new carbons and subsequent advances in the performance of many important tools for chromatography, from GC columns to sample preparation products.

Today, Supelco offers nearly 90 different carbons that represent ranges in chemical selectivity and physical properties to match the nearly limitless analytical applications with which carbons have become associated. Ranging in particle size from 1 to 1000 microns and surface area from 1 to 1500 m²/g, Supelco carbon adsorbents are found in packed and capillary GC columns (including PLOT columns), Supelco SPME (solid phase microextraction) fibers, Supelclean™ SPE tubes, Supelco Dioxin Prep System carbon tubes, chemical desorption (ORBO) tubes and thermal desorption (TDU) tubes, and others. We also supply these materials in bulk quantities for customer-specific devices and applications.

Carbon Adsorbent Sampler Kits

The large choice in carbon chemistries and physical properties poses a new challenge: choosing the right one for a new application, especially considering they are often used in combination. The goal in selecting the right carbon adsorbent is to find a system (one or a combination of carbons or other adsorbents), that can retain a specific analyte or group of analytes for a specific sample volume during the adsorption process, and release the analyte(s) efficiently during the desorption process.

In order to facilitate the process of choosing the right adsorbent system in an economical fashion, we introduced the Supelco Carbon Adsorbent Sampler Kits. Each kit contains small quantities of several related Supelco carbon adsorbents. Once the ideal carbon has been identified, Supelco is ready to work with you to supply larger quantities to meet your specific requirements.

Two Innovative Customer-Specific Carbon Applications

The unique and valuable characteristics of Supelco carbons warranted their inclusion in experiments investigating the analysis for organic material on the 1995 Galileo Mission to Jupiter and the 2005 Cassini-Huygens Missions to Titan, Saturn's largest moon. One of the carbons, a Carboxen™ carbon molecular sieve, is used in the enrichment cell of an Ion and Neutral Mass

Spectrometer (INMS) onboard the Cassini orbiter. Gaseous components in Saturn's atmosphere, including elements, isotopes and molecules, are collected, concentrated and separated by the Carboxen. The other Carboxen, a graphitized polymeric carbon, is onboard the Huygens probe that landed on Titan. During its two and one-half hour descent and for ninety minutes after it landed on the surface, the probe transmitted data to the Cassini orbiter. This Carboxen is also contained in an enrichment cell, this time in a GC-MS instrument, where it is used for collecting, concentrating and separating light hydrocarbons, such as methane and acetylene. Scientists believe that the Titan atmosphere is composed of hydrocarbons and data provided by this instrument will further their understanding. Supelco carbons will make their next venture into space with the 2007 Mars Mission.

In another example, Dr. Michael Davis and colleagues at Oklahoma State University have employed Supelco carbons to monitor biomarkers in exhaled breath from sled dogs for depletion of muscle glycogen during cold weather distance races. Data from this experiment is slated for publication and will be cited in a future issue of Analytix.

Technical Service: A Valuable Resource

If you need assistance in choosing the right kit for your application, simply contact our Technical Service Department at techservice@sial.com for expert answers to your adsorbent questions.

Related Information

For more information, request the following literature:

"Carbon Adsorbent Kits" (T406044)

... a thorough description of the kits and the properties of the adsorbents they contain.

"A Tool for Selecting an Adsorbent for Thermal Desorption Applications" (T402025)

... presents adsorption/desorption data on 43 common air pollutants on 24 different adsorbents.

"Characterization of Adsorbents for Sample Preparation Process" (T402026)

... describes the past, present and future of carbon adsorbent research at Supelco.



Figure 1 Carbon Molecular Sieve Kit

Product List Supelco Carbon Adsorbent Sampler Kits

Cat. No	Brand	Description	Components	Packaging Size
13027-U	Supelco	Graphitized Carbon Black Kit (20/40)	20/40 Carbotrap B 20/40 Carbotrap C 20/40 Carbotrap F 20/40 Carbotrap X 20/40 Carbotrap Y	5g 5g 5g 5g 5g
13026-U	Supelco	Graphitized Carbon Black Kit (60/80)	60/80 Carbopack B 60/80 Carbopack C 60/80 Carbopack F 60/80 Carbopack X 60/80 Carbopack Y 60/80 Carbopack Z	5g 5g 5g 5g 5g 5g
13028-U	Supelco	Carbon Molecular Sieve Kit	60/80 Carbosieve G 60/80 Carbosieve S-III 20/45 Carboxen 569 60/80 Carboxen 1000 40/60 Carboxen 1003 80/120 Carboxen 1012 60/80 Carboxen 1016 60/80 Carboxen 1018 60/80 Carboxen 1021	5g 5g 5g 5g 5g 5g 5g 5g 5g

Lewatit ion exchange resins: Setting the pace with MonoPlus technology

New applications for the tiny beads with big benefits

By Dipl.-Ing. Stefan Hilger, Ion Exchange Resins Business Unit, LANXESS Deutschland GmbH ... lewatit@lanxess.com

Figure 1

The Lewatit® plant in Bitterfeld, Germany with production and logistics optimized for maximum flexibility to benefit the customer.

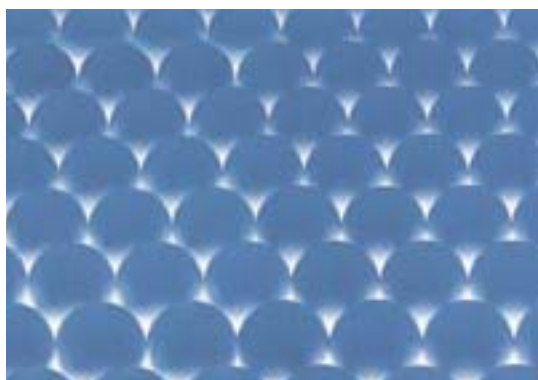


No matter whether the requirement is for potable water treatment or the extraction of valuable materials in hydrometallurgy, Lewatit® ion exchange resins from LANXESS and offered through Sigma-Aldrich give the right solution in more than 500 fields of application. The innovative, state-of-the-art MonoPlus™ process now allows the production of resin beads with a defined uniform size and homogeneous internal structure.

As is true for any analytical technology, the performance and specifications of the product must keep pace with the demands of the application. Selection criteria are becoming stricter in all industries, in all sectors, and as the range of applications expands, there is an increasing need for specialized, higher-performance ion exchange resins and adsorbents. The Lewatit team of LANXESS has come up with an innovative answer: The current generation of selective monodisperse resins is now available for an even wider range of applications. The Lewatit MonoPlus line represents the state-of-the-art in research and development engineering, and gives end-users the assurance that they have opted for top product performance with unmatched efficiency.

Figure 2

Lewatit® MonoPlus® beads are available in monodisperse diameters for many applications. The bead size can be adjusted between 0.30 mm and 0.90 mm.



Lewatit particles: Shaped, not screened

The vast majority of what the ion exchange resin market claims as “constant” bead size pales in comparison with Lewatit MonoPlus beads. The difference lies in the manufacturing process and is apparent in the resulting performance of the resin. Resins that are manufactured by conventional processes and then turned into a supposedly „constant size” by screening cannot offer the monodisperse particle size uniformity of MonoPlus, which is characterized by extremely narrow tolerances. With the foundation of the original monodisperse process, the Lewatit team continually improved their processes and products, underscoring the claim of Lewatit to be the leader in its field in production of monodisperse resins. Their diligent efforts have certainly proven worthwhile.

The performance attained by today’s MonoPlus products is quite simply outstanding. The range of products in the MonoPlus line has been consistently oriented to the varied individual needs of the customers. Equal diameter of the resin beads not only means equal size, it also means consistency in both chemical and physical properties.

Innovations for all applications!

Lewatit is available in the optimized MonoPlus grade for virtually every application. Whether it involves industrial water, drinking water or ultra-pure water, whether the application is in the food industry or chemical industry for catalysis or chemical processing, there likely is a MonoPlus resin with the right properties. Choose from monodisperse anion and cation exchange resins, strongly or weakly basic, in perfectly blended mixtures, and chelating resins. Compared to conventional resins, MonoPlus has a number of clear advantages: greater mechanical and osmotic stability, higher kinetics, 10-20 % higher capacity and much lower leakage.

Here are just a few examples that illustrate the scope of the current MonoPlus applications:

MonoPlus MP 500 has proved ideal for the demineralization of water for industrial steam production by modern counter-current processes. It is a monodisperse, strongly basic, macroporous anion exchange resin based on a styrene divinylbenzene

copolymer. Its favorable kinetic properties result in better capacity utilization than with comparable ion exchange resins that have a heterodisperse bead-size distribution.

MonoPlus M 800 KR is used for the decontamination of circuits in nuclear power plants. This grade is highly regenerated and purified to meet the nuclear industry

specifications. It is generally combined with MonoPlus S 200 KR, a highly effective cation resin.

MonoPlus TP 214 is the ideal candidate for removing mercury from flue gas water or groundwater, and for removing or extracting metals (gold, silver, platinum group elements) in hydrometallurgy.

Product List Lewatit MonoPlus Ion-exchange resins from Sigma-Aldrich

Cat. No.	Brand	Description	Remarks	Application
03953	Fluka	Lewatit MonoPlus S-100	Monodisperse, extremely high chemical and mechanical stability, high osmotic resistance and exchange kinetics, almost linear pressure drop over total bed height, excellent separability of components in mixed bed filters	Deminerlization or ultra-purification by mixed bed with Lewatit MonoPlus M-800
62102	Fluka	Lewatit MonoPlus SP-112	Pronouncedly high chemical stability and high osmotic resistance to oxidizing agents	Deminerlization, condensate polishing in combination with Lewatit MonoPlus MP-500, removal of metal ions and heavy metals
62096	Fluka	Lewatit MonoPlus M-500	Monodisperse, extremely high chemical and mechanical stability, high osmotic resistance and exchange kinetics	Deminerlization
84186	Fluka	Lewatit MonoPlus MP-64	Medium basicity (bears also quaternary ammonium groups)	Removal of halides, heavy metal complexes, humic acids, lignin sulfonates, anionic detergents and other organics, deminerlization in combination with Lewatit MonoPlus M-500, removal of acids
62107	Fluka	Lewatit MonoPlus TP-214	Partially regenerable Hg-exhausted resins with HCl. Complete regeneration only with 2M Na2S solution	Removal of Hg/Ag/Au/Pt from aqueous solutions

Product List Lewatit MonoPlus Ion-exchange resins from Sigma-Aldrich

Cat. No.	03953	62102	62096	88757	84186	62107
Brand	Fluka	Fluka	Fluka	Fluka	Fluka	Fluka
Description	Lewatit MonoPlus S-100	Lewatit MonoPlus SP-112	Lewatit MonoPlus M-500	Lewatit MonoPlus MP-500	Lewatit MonoPlus MP-64	Lewatit MonoPlus TP-214
Matrix	Styrene-DVB, gel-type	Styrene-DVB, macroporous	Styrene-DVB, gel-type	Styrene-DVB, macroporous	Styrene-DVB, macroporous	Styrene-DVB, porous
Mode	Strongly acidic cation exchange	Strongly acidic cation exchange	Strongly basic anion exchange	Strongly basic anion exchange	Weakly (medium) basic anion exchange	Chelate-forming resin
Functional group	Sulfonic acid	Sulfonic acid	Quaternary amine	Quaternary amine	Tertiary/quaternary amine	Thiourea
Ionic form	Sodium (Na ⁺)	Sodium (Na ⁺)	Chloride (Cl ⁻)	Chloride (Cl ⁻)	Free base / Cl ⁻	activated, regenerated
Particle size (mm)	0.60 ± 0.05	0.65 ± 0.07	0.62 ± 0.05	0.62 ± 0.05	0.59 ± 0.05	0.50 - 0.60
Water retention (%)	42 - 48	52 - 57	48 - 55	60 - 65	61 - 66	43 - 48
pH range	0 - 14	0 - 14	0 - 14	0 - 14	0 - 14	0 - 14
Max. operating temp (°C)	120	120	70	70	70	80
Total exchange capacity (meq/mL)	>2.0	>1.7	>1.3	>1.1	>1.3	>2.0
Swelling (%)	8 (Na ⁺ → H ⁺)	8 (Na ⁺ → H ⁺)	22 (Cl ⁻ → OH ⁻)	22 (Cl ⁻ → OH ⁻)	20 (FB → Cl ⁻)	5
Density (g/mL)	1.28	1.24	1.08	1.06	1.04	1.1
Shipping weight (g/L)	830	740	700	640	620	700

Mobile Phase Additives for LC-MS. Part 1: Acids – The Most Common Choice

This is the first in a five-part series on mobile phase additives for LC-MS to appear in each issue of *Analytix* in 2006

By Joachim Emmert, Senior Scientist R&D, Fluka / Riedel-de Haën ... jemmert@sial.com

In LC-MS certain chemicals are often added to the mobile phase or introduced post-column prior to the interface to influence analyte ionization. Small organic acids like formic and acetic acid are among the most commonly used additives (see **Table 1**). Their widespread use is derived from two fundamental reasons. First, many chromatographic separations benefit in terms of

retention and/or peak shape under acidic conditions. Second, most mass spectrometric measurements are done in positive ion mode, which is accomplished by the addition of a proton to form the molecular ion $[M+H]^+$. The above mentioned organic acids have necessary acidity and volatility to provide an excess of cations for this purpose.

Table 1 Product List of LC-MS additives

Cat. No.	Brand	Description*	Package Size
40967	Fluka	Trifluoroacetic acid, puriss p.a., eluent additive for LC-MS	50 mL
40967	Fluka	Trifluoroacetic acid, puriss p.a., eluent additive for LC-MS	10 x 1 mL
56302	Fluka	Formic acid, puriss p.a., eluent additive for LC-MS	50 mL
49199	Fluka	Acetic acid, puriss p.a., eluent additive for LC-MS	50 mL
49916	Fluka	Propionic acid, puriss p.a., eluent additive for LC-MS	50 mL
55674	Fluka	Ammonium formate, puriss p.a., eluent additive for LC-MS	50 g
49638	Fluka	Ammonium acetate, puriss p.a., eluent additive for LC-MS	50 g
61333	Fluka	Sodium citrate tribasic dihydrate, puriss p.a., eluent additive for LC-MS	50 g
40867	Fluka	Ammonium bicarbonate, puriss p.a., eluent additive for LC-MS	50 g
44273	Fluka	Ammonium hydroxide solution 25%, puriss p.a., eluent additive for LC-MS	100 mL
65897	Fluka	Triethylamine, puriss p.a., eluent additive for LC-MS	50 mL

* "puriss" quality grade is defined as >98.5% assay, <0.1% ash, and specification n + 0.001, d + 0.001 with no extraneous color and a homogeneous appearance. "p.a." or pro analysi denotes a product with guaranteed trace impurity levels and/or suitability for the indicated analytical application.

Test conditions

In this article, our aim is to demonstrate the effect on ionization, chromatographic and mass spectral behavior of some common acidic LC-MS mobile phase additives with five test analytes. **Table 2** lists the test compounds, their sum formula and mass in addition to the observed mass and its explanation. The additives were dissolved in both aqueous and organic mobile phase components at a concentration of 0.1%. The alternative method of adding them prior to the interface was not tested in this case. The MS was an ion trap (Bruker Esquire 3000+) operated in positive ion mode. Reliable MS-identification and quantification depends upon using MS-compatible HPLC columns and solvents to minimize background, reduce instrument fouling and maximize sensitivity. Riedel-de Haën LC-MS **CHROMASOLV**[®] solvents and Supelco's Ascentis[™] and Discovery[™] HS columns meet these requirements.

HPLC Conditions

LC-MS column: Supelco Discovery HS C18, 15 cm x 2.1 mm, 5 µm particles (Cat. No. 568502-U)
 Mobile phase: A: Water (LC-MS **CHROMASOLV**[®], Cat. No. 39253), B: Acetonitrile (LC-MS **CHROMASOLV**[®], Cat. No. 34967)

Gradient profile:	Time (min.)	%A	%B
	0	100	0
	10	100	0
	20	0	100
	30	0	100

Flow rate: 0.4 mL/min
 Sample: Raffinose, bradykinin, digoxin, propazine each 10 ng/mL, reserpine, 5 ng/mL
 Injection volume: 5 µL

Table 2 Test compounds

Cat. No.	Brand	Compound	Formula	Molecular Mass (monoisotopic)	Observed Mass	Explanation
83400	Fluka	Raffinose	C ₁₈ H ₃₂ O ₁₆	504.2	527.2	[M+Na] ⁺
15859	Fluka	Bradykinin	C ₅₀ H ₇₃ N ₁₅ O ₁₁	1059.6	530.8	[M+2H] ²⁺
37100	Fluka	Digoxin	C ₄₁ H ₆₄ O ₁₄	780.4	803.4 651.3	[M+Na] ⁺ [M-Digitoxose] ⁺
R0875	Sigma	Reserpine	C ₃₃ H ₄₀ N ₂ O ₉	608.3	609.3	[M+H] ⁺
45640	Fluka	Propazine	C ₉ H ₁₆ N ₅ Cl	229.1	230.1	[M+H] ⁺

Figure 1 EIC without mobile phase additives

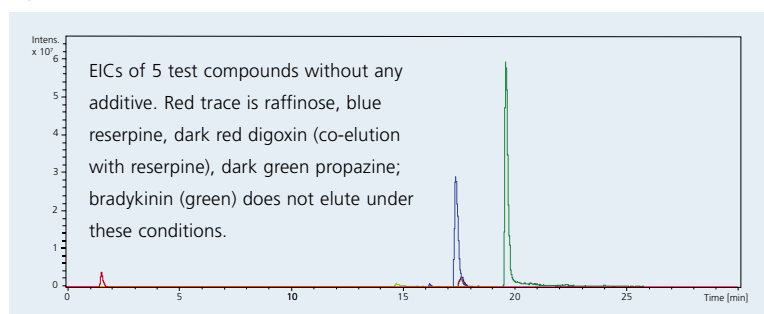


Figure 2 EIC with acetic acid additive

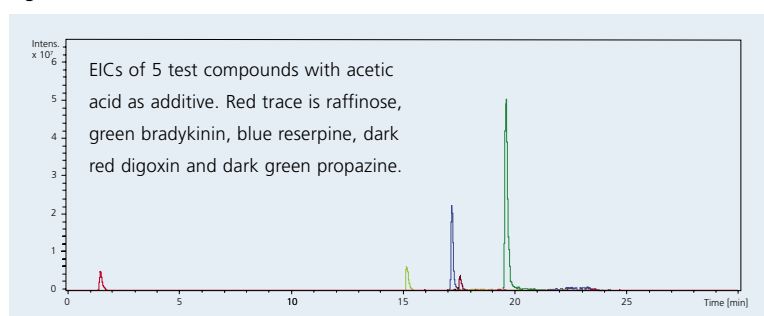


Figure 3 EIC with formic acid additive

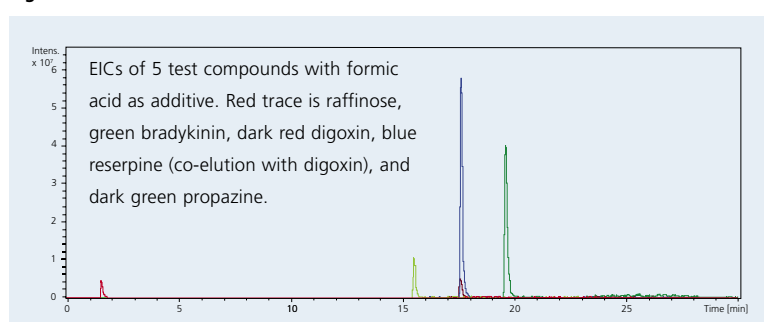


Figure 4 Mass spectrum of propazine with and without acidic additives

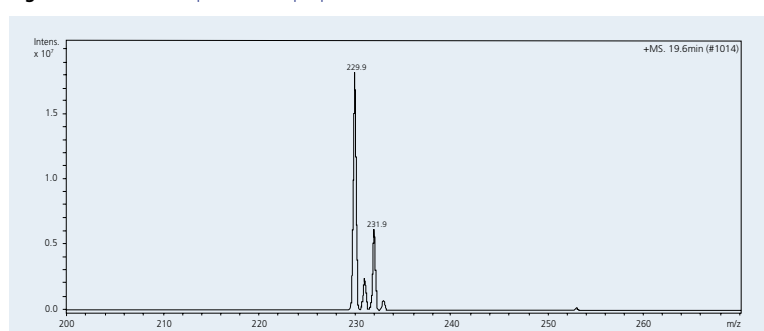
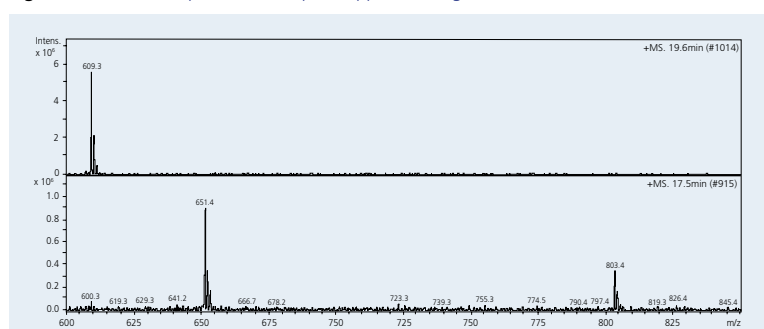


Figure 5 Mass spectra of reserpine (upper) and digoxin (lower) with acetic acid as additive



Effect of acidic additives on chromatography and sensitivity

When used as a mobile phase additive, acids impact the retention behavior of pH-sensitive compounds, especially reserpine where slight pH changes shift its position relative to digoxin and even cause co-elution. The chromatograms always consist of the extracted ion chromatograms (EIC) of the observed mass of each compound. If the observed mass changes due to conditions, the EIC is adjusted accordingly. The y-axis is a measure of the extent of ionization and the achieved sensitivity, and is therefore kept constant in most examples. **Figures 1 – 3** show the effect of acidic additives on the EIC of the test compounds.

Without acidic additives in the mobile phase (**Fig. 1**) elution and ionization of bradykinin and digoxin are insufficient. However, the ionization of propazine is slightly better than under acidic conditions. It will be shown later in the series that neutral conditions are preferred for the ionization of propazine.

Fig. 4 shows the mass spectrum of propazine with the typical chlorine isotopic pattern and the addition of one H⁺-ion; the theoretical observed mass is calculated with 230.1 Da. This spectral behavior does not change with the addition of acids, only the extent of ionization.

Resolution of reserpine and digoxin is accomplished by adding acetic acid (**Fig. 2**), which gives a pH of 3.3 to the aqueous portion of the mobile phase. Improved resolution results from a slight retention time shift of reserpine, sharper peaks and a change in the ionization of digoxin. In **Fig. 1** (no additives) the observed mass was the sodium adduct $M = 803.4 [M+Na]^+$, whereas with acetic acid as additive (**Fig. 2**) $M = 651.3 [M-Dig]^+$ is the most abundant mass and is actually a fragment, originating from the removal of one digitoxose unit. Reserpine shows the typical behavior of adding one H⁺-ion. **Fig. 5** shows the mass spectra of reserpine (upper) and digoxin (lower) when acetic acid is added to the mobile phase.

Effect of formic acid addition, which gives a pH of 2.7 to the aqueous mobile phase component, is shown in **Figure 3**. Adding formic acid increases the reserpine signal and changes the relative elution of digoxin and reserpine again. The effect on raffinose and bradykinin is only small. More details on this in the next article of the series.

Conclusions

Although volatile, low molecular weight organic acids are commonly used as additives in LC-MS, their effect on ionization is usually not dramatic. Their primary advantage is that they improve ionization and resolution of a wide range of molecules

You know the power of LC-MS

You rely on your LC-MS instrument to produce more data everyday. You're asking it to look at lower and lower analyte levels. You can't afford instrument downtime. You can't afford to question your data: "Is that a real peak, or is it an artifact?"

Everything you value about your LC-MS and the data it provides can be lost by using impure HPLC mobile phase solvents

Impure HPLC solvents are the most common source of artifacts and unstable LC-MS baselines. Particles in the solvents will clog and damage your instrument components.

Can you afford any of these problems?

Of course not.

So switch to LC-MS CHROMASOLV® solvents, additives and blends that provide:

- Very low level of inorganic and metal ions
- No particles and non-volatile compounds
- Low gradient baseline even with your own optimized protocols

And they come in convenient blends that save you preparation time.

Trust your time

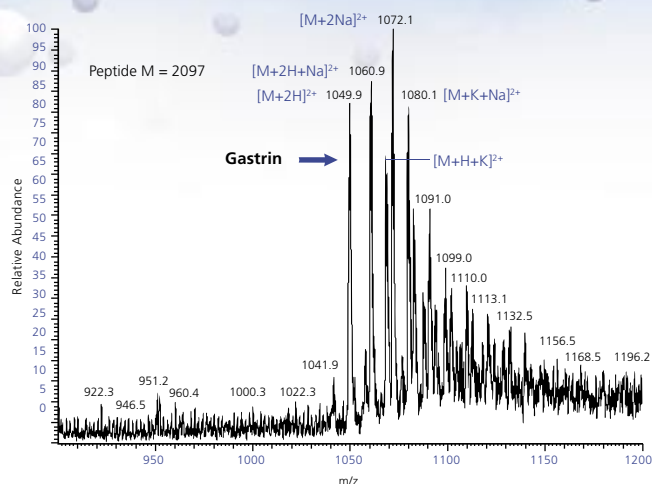
Trust your data

Trust your instrument

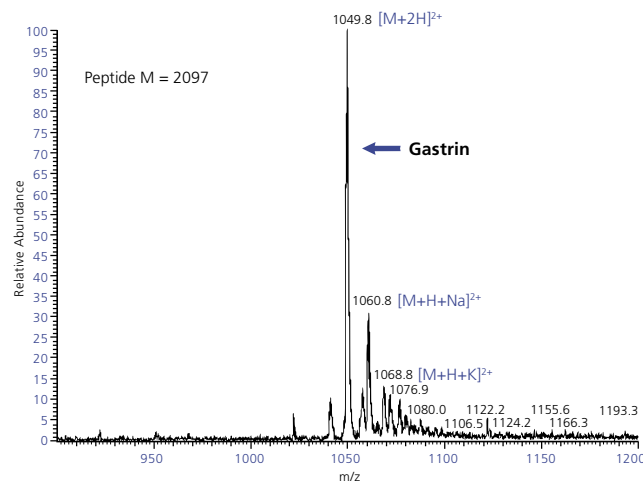
To only the best solvents, additives and blends...
LC-MS CHROMASOLV® from Sigma-Aldrich.

Your LC-MS solvents: CHROMASOLV®

...is it worth taking a chance with anything else but the best?



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.

To see the complete line of CHROMASOLV® solvents, additives and blends for LC-MS and other sensitive analytical applications, visit our website.



Carbohydrate Differentiation Discs for identification of microorganisms

Differentiate and identify bacteria based on their carbohydrate fermentation profile in liquid, solid and semi-solid growth media

By Jvo Siegrist, Product Manager Microbiology ... isiegris@sial.com

Figure 1

Salmonella typhimurium
ATCC 14028

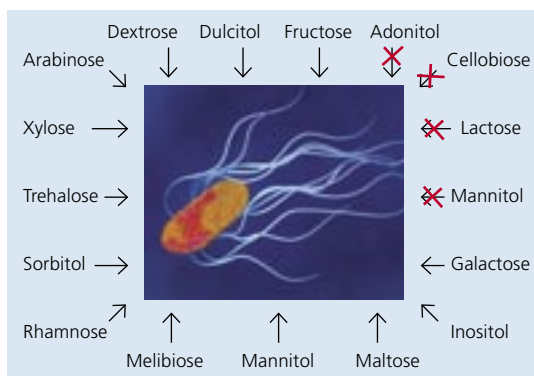


Figure 2 Test row with Carbohydrate Differentiation Discs for carbohydrate fermentation abilities in liquid media. The discs can be seen at the bottom of the tubes.



A microorganism's carbohydrate fermentation profile is often characteristic and can facilitate its identification. The differences arise because of the varying types and activity of enzymes that target specific carbohydrates (1, 2). As an example, the different carbohydrates metabolized by *Salmonella typhimurium* ATCC 14028 are shown in **Figure 1**.

The presence of carbohydrate-utilizing enzymes can be detected visually via gas (CO₂) production or as a pH-induced color change from the production of acid.

Product List Sigma-Aldrich Carbohydrate Differentiation Discs

Cat. No.	Brand	Carbohydrate	Code	Package size
55876	Fluka	Adonitol	Ad	25 or 10 x 25 discs
80372	Fluka	Arabinose	Ar	25 or 10 x 25 discs
56481	Fluka	Cellobiose	Ge	25 or 10 x 25 discs
63367	Fluka	Dextrose	De	25 or 10 x 25 discs
73044	Fluka	Dulcitol	Du	25 or 10 x 25 discs
53901	Fluka	Fructose	Fc	25 or 10 x 25 discs
89608	Fluka	Galactose	Ga	25 or 10 x 25 discs
89614	Fluka	Inositol	Is	25 or 10 x 25 discs
90058	Fluka	Inulin	In	25 or 10 x 25 discs
28816	Fluka	Lactose	La	25 or 10 x 25 discs
77653	Fluka	Maltose	Ma	25 or 10 x 25 discs
94438	Fluka	Mannitol	Mn	25 or 10 x 25 discs
94445	Fluka	Mannose	Mo	25 or 10 x 25 discs
93196	Fluka	Melibiose	Mb	25 or 10 x 25 discs
94226	Fluka	Raffinose	Rf	25 or 10 x 25 discs
93999	Fluka	Rhamnose	Rh	25 or 10 x 25 discs
92971	Fluka	Salicin	Sa	25 or 10 x 25 discs
93998	Fluka	Sorbitol	Sb	25 or 10 x 25 discs
94309	Fluka	Sucrose	Su	25 or 10 x 25 discs
92961	Fluka	Trehalose	Te	25 or 10 x 25 discs
07411	Fluka	Xylose	Xy	25 or 10 x 25 discs

The Sigma-Aldrich analytical microbiology product line includes Carbohydrate Differentiation Discs to detect microorganisms based on their ability to ferment twenty-one different carbohydrates. The discs are effective yet economical, and work in any sugar-free medium – solid, liquid or semi-solid. The detailed instructions supplied with the discs lists the recommended media

Using Discs with Liquid and Semi-solid Media

Liquid or semi-solid media are dispensed in 5 mL amounts in test tubes and sterilized. A single Carbohydrate Differentiation Disc and the inoculum, which is the sample to be analyzed for the presence of the microorganism, are aseptically added to each tube. With liquid medium, CO₂ gas produced during fermentation is collected in an inverted Durham's tube while any acid produced changes the color of the medium. **Figure 2** shows an example of some developed tubes indicating the presence (or absence) of the target microorganism based on the ability to ferment the specific sugars on the discs. The discs are visible at the bottom of the tubes.

In semi-solid medium, the disc and inoculum are pushed into the medium just below the surface. The inoculum-free medium at the bottom of the tube serves as the control, while fermentation can be detected at the upper level. Any CO₂ gas produced is trapped and seen as bubbles in the semi-solid media.

Using Discs with Solid Media

By using solid media it is possible to detect fermentation of number of carbohydrates on same plate. Sterile plates containing the agar medium of choice are surface-seeded with the test organism. Carbohydrate Differentiation Discs are pressed gently onto the surface of the plate at sufficient distance (2 cm) from each other. The plate is incubated 36 ± 1.0°C for 18 - 48 hours and results are recorded at 18 - 24 hours and again at 48 hours. The results should be monitored frequently since reversal of fermentation can occur. On agar plates fermentation is visualized by change color of pH-indicator around the discs.

References:

- [1] Bergey's Manual of Systematic Bacteriology, 1984, Vol. 1, Williams and Wilkins, Baltimore.
- [2] Bergey's Manual of Determinative Bacteriology, 1994, 9th ed., Williams and Wilkins, Baltimore, MD, USA. (Available from Sigma-Aldrich, Cat. No. B0411)

Isotopically labeled mycotoxin standards for LC-MS/MS: 15-*d*₇-deoxynivalenol (15-*d*₇-DON) and 3-*d*₃-acetyldeoxynivalenol (3-*d*₃-AcDON) New standards from Sigma-Aldrich permit reliable quantification of these important toxins associated with cereal commodities

By Rainer Walz, Product Manager Analytical ... rwalz@europe.sial.com

Fusarium moulds are a large group of phytopathogenic fungi which are able to infect cereal crops in the field and contaminate them with toxic secondary metabolites. Concerning its rate of occurrence and mean concentration, deoxynivalenol (DON) appears to be the most important *Fusarium* mycotoxin in cereal commodities. In recent years LC-MS/MS has become the method of choice for the analysis of mycotoxins as it offers high sensitivity and specificity and permits the determination of many different analytes within one run. However, LC-MS/MS is susceptible to matrix effects, which give analyte-dependent signal enhancement or, more likely, suppression. The use of stable isotope labeled analytes as internal standards can fully compensate for matrix effects, and instrument and extraction bias.

Procedures for the isotopic labeling of DON and its natural precursor, 3-acetyldeoxynivalenol (3-AcDON), to give 15-*d*₇-deoxynivalenol (15-*d*₇-DON) [1] and 3-*d*₃-acetyldeoxynivalenol (3-*d*₃-AcDON) [2] have recently been reported.

In order to minimize the spectral overlap between isotopically labeled standard and analyte, the mass difference should be at least 2 or 3 amu. However, due to the specific fragmentation of DON and 15-*d*₇-DON it is not necessary in this case (negative ion electrospray ionization, -ESI). Studies showed in detail that the spectral overlap between DON and single labeled 15-*d*₇-DON is only 1.2 % and completely compensated through the calibration curve [2]. In the case of 3-*d*₃-AcDON no spectral overlap was observed due to the mass difference of 3 amu.

Various cereal product samples were analyzed for DON and 3-AcDON by LC-MS/MS using 15-*d*₇-DON and 3-*d*₃-AcDON as internal standards. As a representative example, **Figure 2** shows the LC-MS/MS chromatogram of a cereal product sample monitoring the respective transitions for DON, 15-*d*₇-DON, 3-AcDON and 3-*d*₃-AcDON. The sample was contaminated with 216 µg/kg DON and 14 µg/kg 3-AcDON [2].

Sigma-Aldrich is pleased to offer DON, 15-*d*₇-DON, 3-AcDON and 3-*d*₃-AcDON standards to enable the reliable quantification of these substances.

In addition, we recommend LC-MS CHROMASOLV[®] solvents (www.sigma-aldrich.com/chromasolv) and Ascentis HPLC columns (www.sigma-aldrich.com/chromasolv) as perfect accompaniments to this and any sensitive LC-MS application.

Figure 1
Fusarium mycotoxins
DON and 3-AcDON

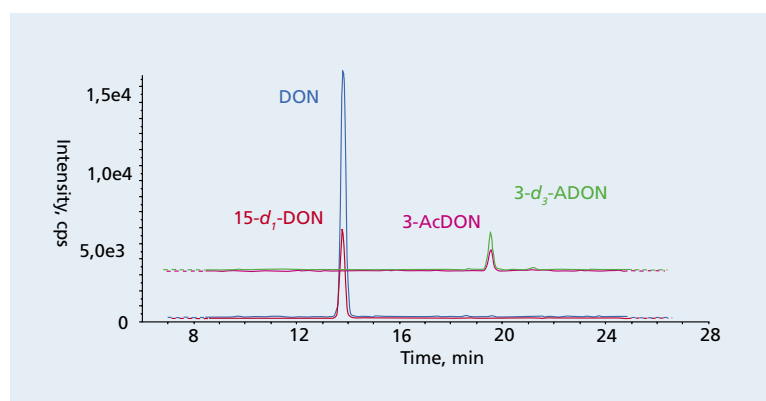
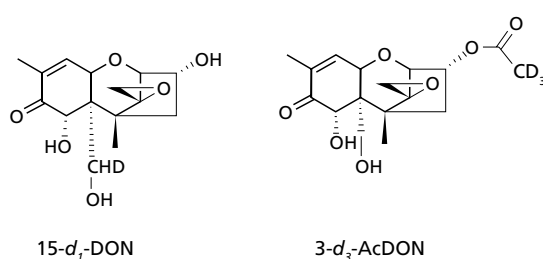


Figure 2 LC-MS/MS chromatogram of a cereal product sample contaminated with 216 µg/kg DON and 14 µg/kg 3-AcDON

References:

- [1] Bretz, M.; Beyer, M.; Cramer, B.; Humpf, H.-U.; Synthesis of stable isotope labeled 3-acetyldeoxynivalenol. *Mol. Nutr. Food Res.* **2005**, 49, 1151-1153.
- [2] Bretz, M.; Beyer, M.; Cramer, B.; Humpf, H.-U.; Stable isotope dilution analysis of the *Fusarium* mycotoxins deoxynivalenol and 3-acetyldeoxynivalenol. *Mol. Nutr. Food Res.* **2006**, 50, 251-260.

Acknowledgments: This article was developed in close collaboration with Prof. Dr. Hans-Ulrich Humpf, from the Food Chemistry Institute of the Muenster University, Germany

Product Listing Mycotoxins, Trichothecenes, Aflatoxins and LC-MS CHROMOSOLV® solvents from Sigma-Aldrich

Mycotoxins from *Fusarium* spp (Trichothecenes)

Cat. No.	Brand	Description	Concentration	Pack size
34129	Riedel-de Haën	d ₃ -Acetyl-Deoxynivalenol	100 µg/mL in acetonitrile	2 mL
34155	Riedel-de Haën	d ₁ -Deoxynivalenol	100 µg/mL in acetonitrile	2 mL

Other Trichothecenes from Sigma-Aldrich

Cat. No.	Brand	Description	Concentration	Pack size
34071	Riedel-de Haën	T-2 Toxin	100 µg/mL in acetonitrile	2 mL
34124	Riedel-de Haën	Deoxynivalenol	100 µg/mL in acetonitrile	2 mL
34128	Riedel-de Haën	¹³ C ₁₅ -Deoxynivalenol	25 µg/mL in acetonitrile	1 mL
34130	Riedel-de Haën	Fusarenon-X	100 µg/mL in acetonitrile	2 mL
34131	Riedel-de Haën	Nivalenol	100 µg/mL in acetonitrile	2 mL
34132	Riedel-de Haën	3-Acetyldeoxynivalenol	100 µg/mL in acetonitrile	2 mL

Further Mycotoxin Standards

Cat. No.	Brand	Description	Concentration	Pack size
Aflatoxins				
34029	Riedel-de Haën	Aflatoxin B1	2 µg/mL in acetonitrile	2 mL, 5 mL
34031	Riedel-de Haën	Aflatoxin M1	0.5 µg/mL in acetonitrile	2 mL, 5 mL
34032	Riedel-de Haën	Aflatoxin G1	2 µg/mL in acetonitrile	2 mL, 5 mL
34033	Riedel-de Haën	Aflatoxin G2	0.5 µg/mL in acetonitrile	2 mL, 5 mL
34034	Riedel-de Haën	Aflatoxin B2	0.5 µg/mL in acetonitrile	2 mL, 5 mL
34036	Riedel-de Haën	Aflatoxin Mix B1 and G1 B2 and G2	2 µg/mL in acetonitrile 0.5 µg/mL in acetonitrile	1 mL, 2 mL 1 mL, 2 mL
BCR-262	BRC® IRMM	Defatted Peanut Meal (Aflatoxin B1)	blank	100 g
BCR-263	BRC® IRMM	Defatted Peanut Meal (Aflatoxin B1)	medium level	100 g
BCR-264	BRC® IRMM	Defatted Peanut Meal (Aflatoxin B1)	high level	150 g
BCR-375	BRC® IRMM	Compound Feed (Aflatoxin B1)	blank	50 g
BCR-376	BRC® IRMM	Compound Feed (Aflatoxin B1)	low level	50 g
BCR-401	BRC® IRMM	Peanut Butter (Aflatoxins)	blank	50 g
ERM- BD282	ERM® IRMM	Whole Milk Powder (Aflatoxin M1)I	zero level	30 g
ERM- BD282	ERM® IRMM	Whole Milk Powder (Aflatoxin M1)	medium level	30 g
ERM- BD284	ERM® IRMM	Whole Milk Powder (Aflatoxin M1)	high level	30 g

Other Mycotoxins

34037	Riedel-de Haën	Ochratoxin A	10 µg/mL in acetonitrile	2 mL, 5 mL
34126	Riedel-de Haën	Zearalenon	100 µg/mL in acetonitrile	2 mL
34127	Riedel-de Haën	Patulin	100 µg/mL in acetonitrile	2 mL

Product Listing LC-MS CHROMASOLV® Pure Solvents

Cat. No.	Brand	Solvent	Package size
39253	Riedel de Haën	Water	1 L
34967	Riedel de Haën	Acetonitrile	1 L, 6 x 1 L, 4 x 2.5 L, 5 L
34966	Riedel de Haën	Methanol	1 L, 6 x 1 L, 4 x 2.5 L, 5 L
34965	Riedel de Haën	2-Propanol	1 L, 6 x 1 L, 4 x 2.5 L, 5 L
34972	Riedel de Haën	Ethyl acetate	1 L, 2.5 L

Test Mixes for GC and HPLC Application-relevant and easy-to-use test mixes from Supelco, complete with methods and interpretation instructions, are valuable tools for maintaining and troubleshooting HPLC and GC columns and systems

By Vicki Yearick, Environmental Market Segment Manager ... vyearick@sial.com

Well-defined GC and HPLC test mixes enable you to troubleshoot chromatographic problems, optimize efficiency and evaluate the system under conditions where results are predicable and not complicated by vagaries of the sample or method. Sigma-Aldrich offers several GC and HPLC test mixes through its Supelco brand. When used on a weekly basis, the mixes can detect small, developing problems in time for intervention.

All test mixes are prepared from raw materials and solvents that have been screened for identify and tested for purity. Instructions on the proper use and interpretation are included.

Product Listing Test mixes for Gas Chromatography from Sigma-Aldrich

Test Mixes for Gas Chromatography – Isothermal

Cat. No.	Brand	Description	Components	Package Size
47300-U	Supelco	Non-polar Column Test Mix	500 µg/mL each component in methylene chloride: 1-Octanol 2-Octanone Decane (C10) Undecane (C11) Dodecane (C12) Tridecane (C13) 2,6-Dimethylphenol 2,6-Dimethylaniline	2 mL
47301	Supelco	Intermediate Polarity Column Test Mix	500 µg/mL each component in methylene chloride: Octanol 2-Octanone Decane (C10) Undecane (C11) Dodecane (C12) Tridecane (C13) Tetradecane (C14) 2,6-Dimethylaniline 2,6-Dimethylphenol	2 mL
47302	Supelco	Polar Column Test Mix	500 µg/mL each component in methylene chloride: 2-Octanone 2,6-Dimethylphenol 1-Octanol Pentadecane (C15) Hexadecane (C16) Heptadecane (C17) Octadecane (C18) Eicosane (C20) 2,6-Dimethylaniline	2 mL

Test Mixes for Gas Chromatography – Programmed Test Mix

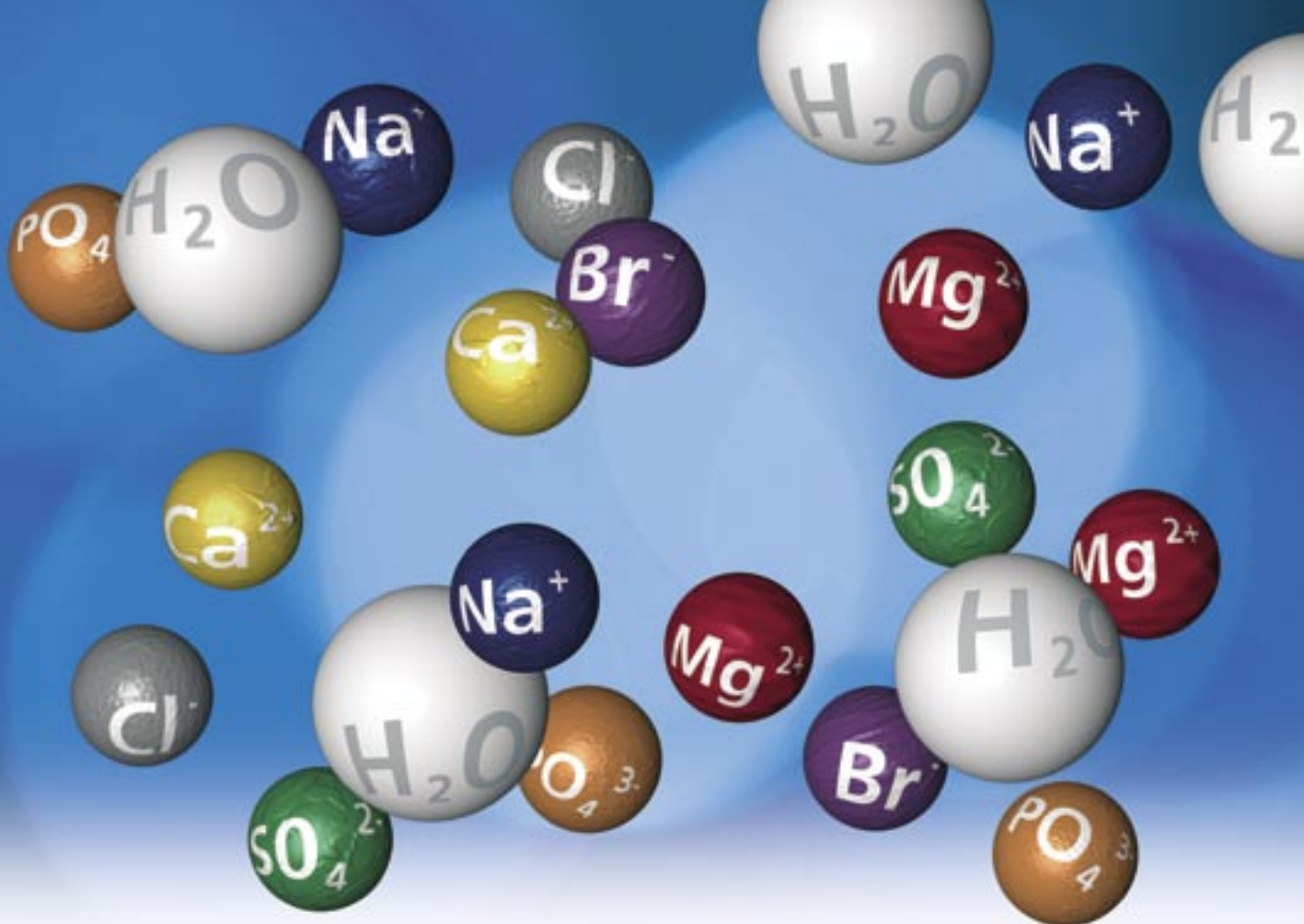
47304	Supelco	(Grob) Programmed Test Mix	Varied concentration in methylene chloride: 2,3-Butanediol, 530 µg/mL 2,6-Dimethylaniline, 320 µg/mL C10 acid methyl ester, 420 µg/mL 2,6-Dimethylphenol, 320 µg/mL C11 acid methyl ester, 420 µg/mL 2-Ethylhexanoic acid, 380 µg/mL C12 acid methyl ester, 410 µg/mL Nonal, 400 µg/mL Decane, 280 µg/mL 1-Octanol, 360 µg/mL Dicyclohexylamine, 310 µg/mL Undecane, 290 µg/mL	2 mL
48244	Supelco	Hydrocarbon Test Mix	Varied concentration in chloroform: Dodecane (C12) Tridecane (C13) Tetradecane (C14) Pentadecane (C15) Hexadecane (C16) Heptadecane (C17)	2 mL

Test Mixes for Gas Chromatography – Specialty Test Mixes

Cat. No.	Brand	Description	Components	Package Size
48476	Supelco	Omegawax Column Test	FAME compounds, ~50 mg/mL in hexane	1 mL
48473	Supelco	Partially Hydrogenated Menhaden Oil	FAME compounds, ~100 mg/mL in hexane	1 mL

HPLC Test Mixes

58278	Supelco	Reversed Phase 1 (for reversed phase columns, e.g. Ascentis C18, RP-Amide or C8, Discovery C18, etc.)	Test compounds in methanol:water (60:40): Uracil, 7 µg/mL (To marker) Acetophenone, 7 µg/mL Benzene, 750 µg/mL Toluene, 775 µg/mL	1 mL
47641-U	Supelco	Reversed Phase 2 (for reversed phase columns, e.g. Ascentis C18, RP-Amide or C8, Discovery C18, etc.)	Test compounds in acetonitrile:water (58:42): Uracil, 5 µg/mL (To marker) N,N-Diethyl-m-toluamide, 600 µg/mL Phenol, 700 µg/mL Toluene, 4 mg/mL	1 mL
58281	Supelco	Normal Phase 1 (for silica columns, e.g. SUPELCOSIL LC-Si)	Test compounds in methylene chloride: Acetanilide, 20 µg/mL Benzanilide, 20 µg/mL Benzene, 600 µg/mL	1 mL
47640-U	Supelco	Normal Phase 2 (for all normal phase columns, e.g. SUPELCOSIL LC-Si, LC-CN, LC-NH ₂)	Test compounds in ethanol:hexane (5:95), each 1 mg/mL: Diethyl phthalate Dimethyl phthalate Toluene	1 mL
58424	Supelco	Amino phase (for NH ₂ -type columns, e.g. Supelcosil LC-NH ₂)	Sugars in acetonitrile:water (25:75), each 25 mg/mL: α-D-glucose Maltose D-fructose Sucrose Lactose	1 mL
58299	Supelco	Cyano phase (for CN-type columns, e.g. Discovery Cyano, SUPELCOSIL LC-CN, LC-PCN or any weak hydrophobic phase)	Test compounds in acetonitrile:water (25:75): Acetophenone, 7 µg/mL Toluene, 775 µg/mL Benzene, 750 µg/mL Uracil, 7 µg/mL	1 mL
47310-U	Supelco	Nucleosides (e.g. SUPELCOSIL LC-18-S)	Nucleosides in water:isopropyl alcohol (1:1), each 10 – 100 µg/mL: 1-Methyladenosine 2'-O-Methylcytidine 2-Thiocytidine 3-Methylcytidine 5-Methylcytidine 7-Methylguanosine Cytidine Guanosine Inosine Pseudouridine Ribothymidine Uridine	1 mL
48743	Supelco	Polyaromatic hydrocarbons (PAH) (for PAH compounds by RP-HPLC according to EPA Method 610, e.g. SUPELCOSIL LC-PAH)	PAHs in methanol:methylene chloride (1:1): Acenaphthene, 1000 µg/mL Acenaphthylene, 2000 µg/mL Anthracene, 100 µg/mL Benzo[a]anthracene, 100 µg/mL Benzo[a]pyrene, 100 µg/mL Benzo[b]fluoranthene, 200 µg/mL Benzo[ghi]perylene, 200 µg/mL Benzo[k]fluoranthene, 100 µg/mL Chrysene, 100 µg/mL Dibenzo[a,h]anthracene, 200 µg/mL Fluoranthene, 200 µg/mL Fluorene, 200 µg/mL Indeno[1,2,3-cd]pyrene, 100 µg/mL Naphthalene, 1000 µg/mL Phenanthrene, 100 µg/mL Pyrene, 100 µg/mL	1 mL
H2016	Supelco	Peptides (for RP-HPLC of peptides, e.g. Discovery BIO Wide Pore C18 or C8, Ascentis C18, etc.)	Peptides (lyophilized), each ~0.5 mg: Gly-Tyr Leu enkephalin Met enkephalin Val-Tyr-Val Angiotensin II	Reconstitute to 1 mL



cer·tain·ty (sûr'tn-tē) noun

1. The fact, quality, or state of being certain.
2. Something that is clearly established or assured.

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Derivatization Reagent Sampler Kits An economical solution to identifying the best reagent for your derivatization needs

By Vicki Yearick, Environmental Market Segment Manager ... vyearick@sial.com

Selecting a derivatization reagent is much easier now than even just a decade ago. Today, analysts have many tools available to guide them in selecting the correct reagent for their particular application. However, there is significant overlap between reagents and applications, and often multiple reagents must be tested to optimize the derivatization. To perform this evaluation it is often necessary to purchase more reagent than is actually needed.

Supelco answered this need by introducing derivatization reagent sampler kits that are the perfect answer for determining the best reagent for a specific application, without the associated cost of purchasing, storing, and ultimately disposing of large volumes of unneeded reagents.

Each sampler kit incorporates a group of related reagents that differ in the stability and volatility of the final derivative. Because of our high purity specifications and reaction efficiencies, Supelco guarantees consistently high reactivity from every lot of each reagent.

Each derivatization reagent sampler kit includes documentation detailing the reaction chemistries and lot purity of each reagent, along with tested derivatization procedures and recommendations for handling and storing of the reagents.

Product Listing Derivatization Reagent Sampler Kits

Cat. No.	Brand	Description	Concentration and Pack Size
505862	Supelco	Acylation Sampler Kit	Heptafluorobutyric anhydride, 3 x 1 mL Trifluoroacetic anhydride, 3 x 1 mL Pentafluoropropionic anhydride, 3 x 1 mL Acetic anhydride, 3 x 2 mL
505870	Supelco	ECD Alkylation Sampler Kit	BCl ₃ -2-Chloroethanol, 11% w/w, 3 x 1 mL Hexaoxyoctadecane, 18 crown 6 (1 gram) BCl ₃ -Methanol, 12% w/w, 3 x 1 mL Pentafluorobenzylbromide, 3 x 1 mL
505854	Supelco	FID Alkylation Sampler Kit	BF ₃ -Methanol, 3 x 1 mL Methanolic HCl (3N), 3 x 1 mL Methanolic Base, 3 x 1 mL TMAH, 0.2M in Methanol, 3 x 1 mL Methanolic HCl (0.5N), 3 x 1 mL
505846	Supelco	Silylation Sampler Kit	BSA, 3 x 1 mL HMDS + TMCS, 3:1 (Sylon HT), 3 x 1 mL BSTFA, 3 x 1 mL TMSI, 3 x 1 mL BSTFA + TMCS, 99:1 (Sylon BFT), 3 x 1 mL

Fractionation of *cis/trans* FAMES using Discovery Ag-Ion SPE A powerful new tool for simplifying GC analysis and improving method accuracy

By Olga Shimelis, R&D Chemist ... oshimelis@sial.com, Craig Aurand, R&D Chemist ... caurand@sial.com and An Trinh, Liquid Separations Product Manager ... atrinh@sial.com

One measure of the nutritional and health value of a food is its fat content. However, it is not only total fat, but also the type of fat that must be considered. Two major chemical markers for the health impact of fats are the degree of unsaturation and, for unsaturated fats, the *cis/trans* orientation across the C=C bonds. *Cis* and *trans* fats differ both in conformation and in biological fate. Rare in nature, *trans* fats are typically formed by hydrogenation of vegetable oil, which increases the shelf life and flavor stability of foods containing them. However, humans lack the enzymes necessary to metabolize *trans* fats, which leads to their accumulation and deposition in the cardiovascular system and subsequent heightened risk of coronary artery disease, which has reached epidemic levels in many developed countries (1).

Because *trans* fatty acids have no known nutritional benefits, but do have adverse health consequences, the United States Food and Drug Administration (FDA) issued a regulation that requires, as of January 1, 2006, food manufacturers must list *trans* fat on the Nutrition Facts panel of foods and some dietary supplements (2). Having a reliable analytical method to measure *trans* fatty acids is an integral component of compliance to this regulation.

The Analytical Difficulty presented by *cis/trans* FAME Separation

The analytical method specified for this new regulation is AOAC 996.06, "Fat (Total, Saturated, and Unsaturated) in Foods", and is suitable for the analysis in *trans* fat in a wide range of foods of varying fat content. In this method, fats are first extracted from food samples by hydrolytic methods (acidic and/or alkaline depending on food type) and petroleum ether followed by methylation to fatty acid methyl esters (FAMES) using BF_3 . FAMES are further extracted into a small volume of hexane prior to GC analysis (3).

The most common *trans* fats in food are mono-unsaturated octadecenoic (C18:1) fatty acids (2). They are also the most difficult to resolve on capillary GC columns using polar stationary phases, like the Supelco SP-2560 which is often employed for this method. To reduce the sample complexity and improve resolution, silver-ion solid phase extraction (Ag-Ion SPE) is used to fractionate *cis/trans* isomers and other FAMES prior to capillary GC analysis.

How does Ag-Ion work?

In Ag-Ion SPE, silver ions are anchored onto SCX SPE functional group as counter-ions. As the FAME sample passes through the cartridge, the SCX-silver counter-ions act as an electron acceptor to form polar complexes with double bonds of unsaturated FAMES. Strength of the interaction increases with the number of double bonds, and saturated fatty acids, which do not contain double bonds, are only weakly retained (4). *Cis* substituted double bonds offer more steric accessibility than their *trans* counterpart, and can therefore form stronger polar complexes with the stationary phase (Figure 1). As a result, *cis* fatty acids are more strongly retained on Ag-Ion SPE than *trans* fatty acids. Differences in retention strength between classes of FAMES and silver counter-ions can be exploited allowing for FAME fractionation prior to GC analysis.

Discovery™ Ag-Ion SPE was developed specifically for the fractionation of FAMES based on degree of unsaturation and for the resolution of *cis/trans* isomers. The SPE phase is silver-loaded using a proprietary procedure to offer optimal resolution, performance and capacity.

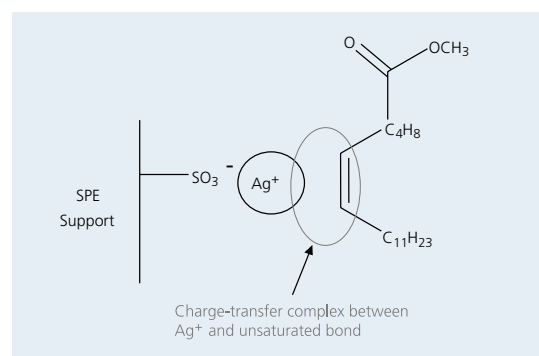


Figure 1 Schematic representation of Ag-Ion SPE interacting with *cis*-FAME

Ag-Ion Application: Extraction and Fractionation of FAMES in Microwave Popcorn Oil

To demonstrate the application, a 1.0 g sample of popcorn oil was collected from a bag of fresh microwaveable popcorn and mixed with 8 mL DI H_2O and 4 mL petroleum ether. The mixture was centrifuged to remove particulates and the ether supernatant layer isolated. The petroleum ether extraction step was repeated four times, and the supernatant was combined, evaporated and reconstituted in 16 mL toluene. A 1 mL aliquot of the toluene extract was transferred to conical reaction vial to which 2 mL 7% BF_3 in methanol was added.

Figure 2

GC Results of Cis/
Trans Fractionation of
Microwave Popcorn Oil

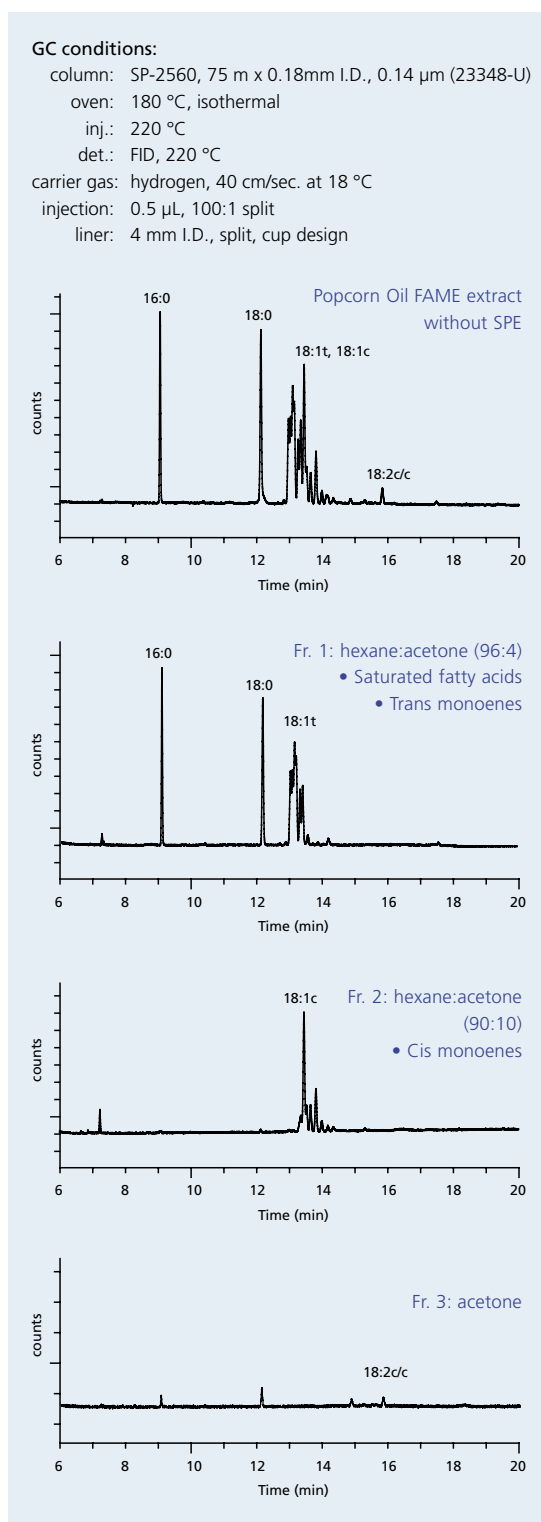


Table 1 Recovery Distribution of FAMES Fraction of Microwave Popcorn using Discovery Ag-Ion SPE

Fraction No.	Eluent (v/v)	Vol (mL)	18:0	Trans 18:1	Cis 18:1	Cis/Cis 18:2
1	Hexane:Acetone 96:4	6	100%	100%	2%	
2	Hexane:Acetone 90:10	4			98%	
3	Acetone	4				100%

The reaction vial was incubated at 80°C for 15 minutes using a heating block and subsequently cooled to room temperature. A 1 mL aliquot of DI H₂O was added and FAMES were extracted twice using 1 mL hexane each time. The upper hexane layers were combined into a fresh vial, evaporated and reconstituted with 5 mL hexane and 50 mg anhydrous Na₂SO₄.

A 1 mL aliquot of the hexane FAMES extract was loaded onto the Discovery Ag-Ion SPE tube, 750 mg bed weight, 6 mL tube volume (54225-U), which had been pre-conditioned with 4 mL acetone followed by 4 mL hexane. Classes of FAMES were fractionated using the elution scheme described in Table 1 and separated on a Supelco SP-2560, 75 m x 0.18mm I.D., 0.14 µm film thickness (23348-U) capillary GC column. Resulting GC chromatograms and recovery data are described in Figure 2 and Table 1, respectively.

Conclusion

When silver is immobilized as a counter-ion on an SCX SPE phase, FAMES can be resolved/fractionated on the basis of degree of unsaturation. Discovery Ag-Ion SPE also offers sufficient selectivity to resolve structural isomers such as cis/trans fatty acids. With the new trans fat labeling regulation mandated by the FDA, Discovery Ag-Ion SPE serves as a powerful tool for simplifying GC analysis and improving method accuracy. In this report, we demonstrated its use by fractionating cis/trans isomers in microwave popcorn oil prior to GC analysis.

Table 2 Ordering Information & Related Products

Cat. No	Brand	Description	Package Size
Discovery Ag-Ion SPE			
54225-U		750 mg / 6 mL SPE Tube	30
54226-U	Supelco	750 mg / 1 mL Rezorian Cartridge	10
SP-2560 Column			
23348-U	Supelco	75 m x 0.18 mm I.D., 0.14 µm	
24056	Supelco	75 m x 0.18 mm I.D., 0.14 µm	
SP-2380 Column			
24116-U	Supelco	30 m x 0.32 mm I.D., 0.20 µm	
24110-U	Supelco	30 m x 0.25 mm I.D., 0.20 µm	
Standards			
47885-U	Supelco	37 Component FAME Mix	

References

- [1] Ascherio, A.; Willett, W. C.; Health effects of *trans* fatty acids, *Am. J. Clin. Nutr.* **1997**,66(suppl), 1006S-1010S.
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- [3] Official Methods of Analysis of AOAC International, **2002**, 17th edition, Revision 1.
- [4] Christie, W. W.; *Silver ion chromatography*. Available at: <http://www.lipidlibrary.co.uk/topics/silver/index.htm>

HYDRANAL® Medium K Improved safety and accuracy for the volumetric Karl Fischer titration of aldehydes and ketones

By Helga Hoffmann, Technical Support HYDRANAL® Manager ... hhoffman@europe.sial.com
and Michael Jeitziner, Product Manager Analytical Reagents ... mjeitziner@sial.com



The Karl Fischer (KF) titration for determination of moisture in aldehydes and ketones can be challenging. Their reaction with conventional reagents produces water, which is also titrated and leads to erroneously high results and vanishing end points. Additionally, a second side reaction, the bisulfite addition, can occur during the titration of aldehydes, which consumes water and leads to erroneously low results.

Addressing this reactivity problem, researchers at Sigma-Aldrich developed 34698 HYDRANAL®-Medium K, which does not produce these detrimental side reactions, eliminating or significantly reducing them as sources of error. Its innovative formulation includes specific solvents not found in competitive KF reagents.

If you already use 34871 HYDRANAL®-Working Medium K, our original medium for aldehydes and ketones, new 34698 HYDRANAL®-Medium K gives comparable speed and accuracy, yet offers important application, safety and transportation benefits.

Reduced toxicity – In 34698 HYDRANAL®-Medium K, we have replaced the 2-chloroethanol found in 34817 HYDRANAL®-Working Medium K with considerably less hazardous alcohols, resulting in a downgrading from T+ (Very Toxic) to Xn (Harmful) hazardous classification. (The Xn classification is because of the amount of chloroform it contains.) A benefit of the reduced toxicity, the Xn category permits wider transportation options.

Application suitability – 34698 HYDRANAL®-Medium K is just as reactive as 34871 HYDRANAL®-Working Medium K, guaranteeing short titration times and accurate results. Using 34816 HYDRANAL®-Composite 5K as the reagent for titration, we compared results of titrations in both media on many aldehydes and ketones, including acetone, 4-methyl-2-pentanone, octanone, cyclohexanone, acetophenone, 2-acetylpyridine, 2-pyrrolidinone, diacetyl, cyclohexanone, pyruvic acid, levulinic acid, acetaldehyde, paraldehyde, 3-hydroxy-benzaldehyde, anisaldehyde among others and found:

- Comparable capacity for the substance to be analysed in 30 mL medium
- Comparable water content, mean and standard deviation
- Comparable titration speed
- Comparable accuracy of the recovery of added water after the titration of a sample

Table Comparability of 34698 HYDRANAL®-Medium K and 34817 HYDRANAL®-Working Medium K for aliphatic ketones

Sample (5 mL)	NEW 34698 HYDRANAL®-Medium K			34817 HYDRANAL®-Working Medium K		
	Titration time per sample (min.)	% H ₂ O (mean +/- std. dev.) n=5	Recovery of water after one sample	Titration time per sample (min.)	% H ₂ O (mean +/- std. dev.) n=5	Recovery of water after one sample
Acetone	2	0.11% +/- 0.24%	100.4%	2	0.12% +/- 1.00%	102.2%
4-Methyl-2-pentanone	0.8	0.009% +/- 9.6%	99.8%	1	0.007% +/- 3.8%	99.8%
3-Octanone	1.5	0.23% +/- 0.37%	100.7%	1.4	0.24% +/- 0.4%	101.0%
Cyclohexanone	1.5	0.021% +/- 3.2%	100.0%	1.5	0.025% +/- 4.1%	102.9%

Application advantages of new HYDRANAL®-Medium K Improved Karl Fischer titrations of reactive aldehydes

By Helga Hoffmann, Technical Support HYDRANAL® Manager ... hhoffman@europe.sial.com
and Michael Jeitziner, Product Manager Analytical Reagents ... mjeitziner@sial.com

New 34698 HYDRANAL®-Medium K offers performance advantages with very reactive aldehydes such as propionaldehyde, butyraldehyde and crotonaldehyde. Capacity and accuracy is also improved for other compounds, including salicylaldehyde, acetylacetone 2,4-dihydroxyacetophenone and 2-benzoylpyridine.

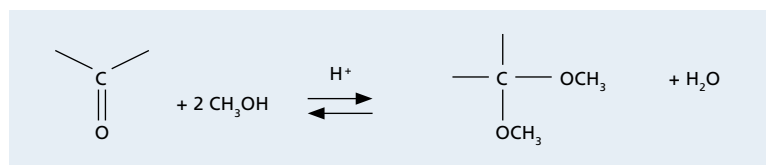
The elimination of methanol in 34698 HYDRANAL®-Medium K presents a two-fold advantage: it eliminates unwanted side reactions and improves the solubility of some samples. For example, we found that as much as 5 g of 3-benzoylpropionic acid can be titrated in this new medium without any problem.

Eliminate nucleophilic addition of methanol by using HYDRANAL®-Medium K

Standard KF titration uses methanol or methanol-containing solvents in the titration vessel. However, aldehydes and ketones undergo nucleophilic addition reaction in methanol, forming an acetal and water. This water causes vanishing end-points or titrations that never reach completion.

Figure 1

Aldehydes and ketones undergo nucleophilic addition of methanol resulting in acetal or ketal and water formation

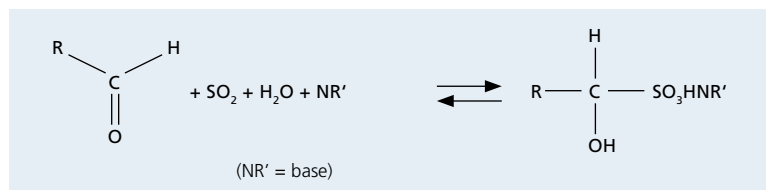


Reduce bisulfite addition by using HYDRANAL®-Composite 5K and HYDRANAL®-Medium K

The bisulfite addition is also an unwanted side reaction of aldehydes during KF titration. The reaction occurs between KF reagent ingredients, the aldehyde and water from the sample, leading to erroneously low moisture levels. To avoid the bisulfite addition, we recommend HYDRANAL®-Composite 5K, which is designed for this application.

Figure 2

Bisulfite addition reaction consumes water



HYDRANAL®-Medium K: Excellent performance without unwanted side reactions

For titrations of aldehydes and ketones, the methanol should be replaced with a solvent that does not produce these side reactions, yet maintains solubility of sample and reagents and fulfills other requirements of the titration parameters for end-point determination.

New 34698 HYDRANAL®-Medium K meets these requirements, and has reduced toxicity compared to our original 34817 HYDRANAL®-Working Medium K. Both products give comparable accuracy and short titration times. The efficacy and improved safety of the 34698 HYDRANAL®-Medium K make it the ideal solvent for titration of even highly reactive aldehydes and ketones.

Application: Water determination in acetone

30 mL HYDRANAL®-Medium K are added to the titration vessel and titrated to dryness with HYDRANAL®-Composite 5K. 5 mL of acetone are weighed by difference, added to the vessel and its water content titrated with HYDRANAL®-Composite 5K.

Application: Water determination in benzaldehyde

Benzaldehyde is very reactive. 30 mL HYDRANAL®-Medium K are added to the titration vessel and titrated to dryness with HYDRANAL®-Composite 5K. 2 mL of benzaldehyde are weighed by difference, added to the vessel and its water content is titrated with HYDRANAL®-Composite 5K.

Product Listing

Cat. No.	Brand	Product	Package size
34698	Riedel-de Haën	HYDRANAL®-Medium K	1 L, 6 x 1L
34816	Riedel-de Haën	HYDRANAL®-Composite 5K	500 mL, 6 x 500 mL, 1 L, 6 x 1 L, 2.5 L, 4 x 2.5 L

A sophisticated solution for water analysis: AQUANAL®-SPECTRO 3 Photometer

Operating in both UV and infrared spectral ranges, AQUANAL®-SPECTRO 3 Photometer has many features that make it flexible, accurate, reliable and easy to operate

Flexibility

- For both AQUANAL®-plus and AQUANAL®-professional reagents
- Operates in both UV and infrared spectral ranges
- Built-in user prompts in six languages
- Wide spectral range: 380 / 430 / 470 / 500 / 520 / 560 / 610 / 700 / 810 nm
- Wide absorbance range: -0.500 to 3.500 Abs
- Test result displayed in mg/L or in extinction mode
- Software operates with Windows 98 and higher versions

Accuracy & Reliability

- Electro-optical accuracy: $\pm 1.5\%$
- Combined LED and fiber-optic technology
- GLP compliant (results given with time / date / place)
- Comprehensive recognition of measurement faults
- Data memory for 1000 results, with data protection

Convenience

- Selection of the most frequently used curves
- Built-in calibration curves for 100 determinations
- Automatic blank setting
- History view lists the latest 50 results
- Power-saving function, automatic power off
- Automatic wavelength selection

AQUANAL®-SPECTRO 3 Photometer comes with all you need to get started



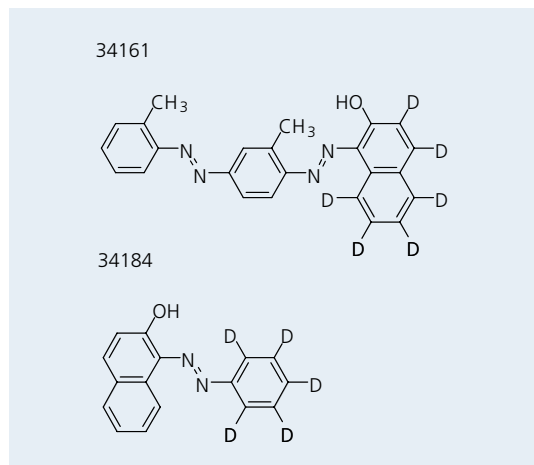
To get you up and running quickly, the SPECTRO 3 is supplied with

- Software (operates with Windows 98 and higher versions)
- External power pack
- Four rechargeable batteries (1.5 V)
- Infrared interface
- Funnel
- Six 16 mm ID tubes
- Four 20 mL sample tubes
- USB to IrDA adapter
- Carrying case
- Instruction manual

New Product Corner Deuterated Sudan Dyes

By Rainer Walz, Product Manager Analytical ... rwalz@europe.sial.com

The Sudan dyes are red dyes that are used for coloring solvents, oils, waxes, petrol, shoe and floor polishes and have been found in some chili powder imported from India. They have also been found in a number of relishes, chutneys and seasonings containing this chili powder. Sudan dyes are not allowed to be added to food in the EU. Sigma-Aldrich is pleased to offer new deuterated Sudan dye standards and solutions for LC-MS.



Product Listing New Deuterated Sudan Dyes by Sigma-Aldrich

Cat. No.	Brand	Description	Package Size
34161	OEKANAL (Riedel-de Haën)	Sudan-IV-d ₆	10 mg
34163	OEKANAL (Riedel-de Haën)	Sudan-IV-d ₆ Solution, 100 µg/mL in acetonitrile	2 mL
34184	OEKANAL (Riedel-de Haën)	Sudan-I-d ₅	10 mg
34181	OEKANAL (Riedel-de Haën)	Sudan-I-d ₅ Solution, 100 µg/mL in acetonitrile	2 mL

www.sigma-aldrich.com/standards

Upcoming Events HYDRANAL®-Seminars



HYDRANAL® is Sigma-Aldrich's premiere line of reagents for pyridine free moisture analysis by Karl Fischer titration. As a service to the scientific community, we routinely offer seminars to provide training on the chemistry behind the technique and information specific to the HYDRANAL® line. Following is a list of upcoming seminars:

June

- 07. Bologna, Italy
- 08. Madrid, Spain
- 13. Barcelona, Spain
- 14. Basel, Switzerland
- 21. Lausanne, Switzerland

September

- 26. Leverkusen, Germany

November

- 28.-29. 2 days KF-Seminar, Seelze, Germany

www.sigma-aldrich.com/events

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