

Analytix

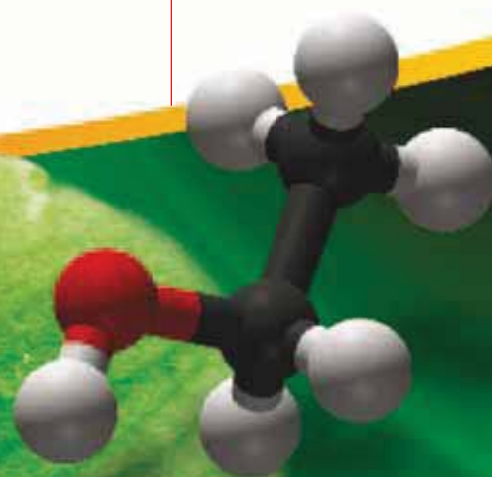
Issue 5 • 2010

 Fluka
Analytical

Riedel-deHaën®

HYDRANAL® going green!

Karl Fischer instrument adjustments for endpoint indication with ethanol-based reagents



- Ethanol-based Karl Fischer Reagents
- New Highly Efficient Silylating Reagents
- Microbiology Base Ingredients
- Certified Amino Acid Standards
- Lipoic Acid Standards
- High Purity Salts

HYDRANAL® Technical Helpline



Helga Hoffmann
Manager Technical Service
HYDRANAL

Dear Colleague,

What would our professional life be like without the challenges that we meet and the problems that we solve every day? Certainly not very exciting. But what would our professional life be like with never-ending challenges and ongoing problems that we have to face over and over again? Quite unproductive and exhausting. We need a healthy mix of challenges, routine work and problem solving to be optimally effective and productive at work.

Sometimes it is inevitable to work out a solution for a special problem on our own, even if it is time-consuming and extensive. Often, however, we can easily access the know-how of specialists that have already found an answer for the same problem.

This principle applies well to the Karl Fischer titration method. Though it is the established method for water content determination in many matrices, in some cases there may be complications that require special consideration in order to achieve an accurate or efficient titration. For example, there may be an issue of sample solubility, some materials may react with a component of the titration reagent, or the implementation of the method may require optimization.

For these types of challenging cases, we at Sigma-Aldrich have expert scientists that would be happy to serve you through our HYDRANAL Technical Helpline. Call us or email us with your comments, questions, or concerns. We look forward to sharing our expertise with you! Furthermore, we offer an extensive array of KF applications with protocols for over 600 applications. You can find a complete list on our website, sigma-aldrich.com/hydranal, where you can conveniently request products to meet your specific needs.

And don't forget our Karl Fischer seminars which we regularly organize in countries throughout the world! There you will have the opportunity to personally discuss special application topics with our experts. The seminar dates are regularly published on our website.

After 30 years of research and application work, Sigma-Aldrich has the most extensive knowledge base regarding Karl Fischer titration. We are happy to share this with you!

Best regards,

Helga Hoffmann
Manager Technical Service HYDRANAL
helga.hoffmann@sial.com



Analytix is published five times per year by Sigma-Aldrich Chemie GmbH,
MarCom Europe, Industriestrasse 25, CH-9471 Buchs SG, Switzerland
Publisher: Sigma-Aldrich Marketing Communications Europe
Publication director: Ingo Haag, PhD
Editor: Daniel Vogler

Feature Article

- 4 **HYDRANAL® going green!**
Karl Fischer instrument adjustments for endpoint indication with ethanol-based reagents

Microbiology

- 7 **Microbiology Base Ingredients**
Media consist of diverse interesting base ingredients, ranging from simple sugars to peptone, salts, antibiotics, and more complex indicators.

Standards

- 9 **Better Understanding the Processes of Life**
Solution with 17 Amino Acids as *TraceCERT*® Certified Reference Material
- 11 **Analytical Standards for Disinfection Byproducts (DPBs)**
- 13 **New Analytical Standards for Sunscreen Lotion Ingredients**
- 14 **New certified reference material from the IRMM**
- 15 **New Analytical Standards for R-(+)- α -Lipoic Acid**

Chromatography

- 15 **Metabolites – A Serious Challenge for LC/MS Separations and Usual Detection Techniques**
- 18 **Silyl methallylsulfinates – New Highly Efficient Silylating Reagent**
Chemoselective silylation of alcohols, polyols, phenols and carboxylic acids
- 21 **Headspace Grade Solvents**
for the Analysis of Organic Volatile Impurities

Spectroscopy

- 22 **New High Purity Salts**
for melting digestion in environmental, water, and food analysis

Titration

- 23 **High quality titration reagents for all your needs in volumetric titration**
Special offer for ready-to-use volumetric standard solutions

HYDRANAL® going green!

Karl Fischer instrument adjustments for endpoint indication with ethanol-based reagents

Helga Hoffmann, Technical Service HYDRANAL Manager helga.hoffmann@sial.com

Andrea Felgner, Product Manager Analytical Reagents andrea.felgner@sial.com



Features and Benefits of HYDRANAL E-type reagents:

- Reduced toxicity over methanol-containing reagents
- Available for both volumetric and coulometric titrations
- Increased reaction rate and conductivity over pure ethanol by additives (patent protected)
- Endpoint color appears visually more intense compared to methanol
- Compatible with all titration equipment (indication parameters may need adjusting)
- Improved solubility for long-chained hydrocarbons
- Enables titration of ketones like acetone

A central focus of the ongoing improvements to our HYDRANAL line of pyridine-free Karl Fischer (KF) reagents is the reduction or elimination of toxic components. One such component is methanol, which is widely used as a single solvent in the titration vessel and as a solvent for other KF reagents. Methanol is an excellent component for the KF reaction, however it is also noxious. Methanol is classified as toxic according to chemical regulations in the European Union. Such poisonous chemicals represent a danger to the environment as well as to the health of the analyst.

Eliminating Methanol: HYDRANAL E-type reagents

The patented HYDRANAL E-type reagents contain ethanol in place of toxic methanol and represent the non-toxic reagent line for volumetric and coulometric KF titration. Not only can ethanol-based KF reagents replace methanol-based reagents in nearly all applications, they also offer advantages for hydrophobic samples. The solubility for long-chained hydrocarbons in ethanol-based HYDRANAL-CompoSolver E is improved over methanol and methanol-containing reagents. Furthermore, alcoholic side-reactions with ketones are often less pronounced in ethanol than in methanol. Consequently, the water content of certain ketones, including acetone, can be determined using HYDRANAL-CompoSolver E in combination with HYDRANAL-Composite titrating agents. NOTE: This is valid only for ketones causing weak side-reactions. Sample sizes should be small. For other ketones, dedicated HYDRANAL media for ketones and aldehydes are recommended.

Endpoint indication techniques for KF titration

Karl Fischer himself had to use visual detection when he carried out his first titration, but today we can fortunately rely on more accurate and standardizable techniques. Double platinum wire electrodes are generally used for endpoint indication in KF titration. For direct KF titration, primarily two indication methods are used: biamperometric and bipotentiometric (or bivoltametric) indication. A constant voltage or current, respectively, is applied between the two electrodes, and the resulting effect upon the response parameter is detected. Before reaching the equivalence point of the reaction, the working medium does not contain free iodine, so the detected current to maintain the constant voltage is low (or the recorded voltage to maintain the constant current is high). As soon as there is a slight amount of excess iodine present, it depolarizes the electrodes and a drastic drop in voltage (or a jump of the current) occurs and indicates the endpoint of the titration.

Biamperometric indication

A constant voltage is applied to the double platinum indicator electrode and the resulting electrical current is recorded. As long as there is an excess of water in the working medium (beginning of the titration), the current necessary to maintain the set voltage is low. However, toward the end of the titration and at the equivalence point, the excess iodine present depolarizes the electrode, and the current rises significantly. The titration curve is usually shown as current [μA] vs. reagent volume [mL].

Bivoltametric or bipotentiometric indication

A constant current is applied to the indicator electrode, and the resulting voltage is recorded. At the beginning of the titration, the voltage is high; at the end of the titration, however, excess iodine molecules decrease the electrical resistance of the solution (cathodic reduction of I_2/I_3^- and anodic oxidation of I^-), making it possible to maintain the constant current at a much lower voltage. Hence, the voltage drops suddenly at the equivalent point of the titration. The titration curve is usually shown as voltage [mV] vs. reagent volume [mL].

Practical experiences with HYDRANAL® E-type reagents

Coulometric titration

HYDRANAL-Coulomat E can generally be used in all coulometric instruments without any complications. When changing reagents in a coulometer to Coulomat E, over-titration may occur at the beginning. However, this happens very often when changing to a new reagent and is quickly resolved by cleaning the platinum pins of the indicator electrode with a soft paper tissue. Caution needs to be taken so that the pins are not bent or twisted. Afterwards, the electrode is sensitized for accurate endpoint indication. Note: HYDRANAL-Coulomat E can be used as both anolyte and catholyte for coulometric KF titration.

Volumetric titration

When carrying out volumetric titrations with methanol or methanol-based KF reagents, a current of up to 50 μA is programmed as a parameter for end point indication (depending on the instrument). However, when using ethanol-based reagents, over-titration often occurs at this strength of current. Over-titration can be recognized by a color change to dark yellow or brown at the endpoint, instead of yellow. Depending on the instrument that is used, the strength of current must be reduced to 15 or 20 μA . More detailed information can be provided from the instrument manufacturer. Just as with the coulometric titration method, the platinum pins of the electrode might need cleaning when changing to a type of ethanol-based reagent such as HYDRANAL-CompoSolver E or HYDRANAL-Solvent E.

For accurate endpoint indication, special switch-off parameters must be defined in the method. For example, a switch-off voltage selected too low can lead to over-titration and erroneous results. More detailed information about the correct indication parameters, as well as the specially recommended parameter adjustments for ethanol-containing reagents, should be obtained from the instrument manufacturer.

Examples of application reports for samples where use of ethanolic reagents is preferred over methanol-containing reagents include:

- L539 Surface preservative, wood decking protector
- L540 5-Hydroxy-1-methylpyrazole
- L456 Peppermint oil and spearmint oil
- L452 Lacquer

Visit our website for an E-types application list and find out how you can eliminate toxic reagents from your KF application:

Take advantage of our expertise gained from thirty years experience and our extensive applications database on Karl Fischer titration. On our website sigma-aldrich.com/hydranal-e-types we provide a list of samples where application reports for ethanol-based reagents are available. Find out how your KF application can be transferred to non-toxic reagents. To obtain an application report, please contact our HYDRANAL specialists at hydranal@sial.com



(continued on page 6)

Non-toxic HYDRANAL® Karl Fischer reagents

Cat. No.	Brand	Description	Pack Size
Reagents for volumetric titration (one-component technique)			
34805	Fluka®	HYDRANAL-Composite 5 Water equivalent approx. 5 mg H ₂ O/mL	500 mL, 1 L, 2.5 L
34806	Fluka	HYDRANAL-Composite 2 Water equivalent approx. 2 mg H ₂ O/mL	500 mL, 1 L, 2.5 L
34827	Fluka	HYDRANAL-Composite 1 Water equivalent approx. 0.7–1 mg H ₂ O/mL	500 mL, 1 L
34734	Fluka	HYDRANAL-CompoSolver E To be used with HYDRANAL Composite.	1 L, 2.5 L
Reagents for volumetric titration (two-component technique)			
34732	Fluka	HYDRANAL-Titrant 5 E Water equivalent approx. 5.00 mg H ₂ O/mL	500 mL, 1 L, 2.5 L
34723	Fluka	HYDRANAL-Titrant 2 E Water equivalent approx. 2.00 mg H ₂ O/mL	1 L
34730	Fluka	HYDRANAL-Solvent E To be used with HYDRANAL Titrant.	500 mL, 1 L, 2.5 L
Reagents for coulometric titration			
34726	Fluka	HYDRANAL-Coulomat E Ethanol-based reagent, suitable as analyte and catholyte.	500 mL

If you need technical help with your Karl Fischer application, please don't hesitate to contact us at hydranal@sial.com or speak to our experts:

Europe and Global Market

Ms. Helga Hoffmann
 Technical Service HYDRANAL
 Wunstorfer Straße 40
 D-30926 Seelze, Germany
 Tel. +49 (0) 5137 8238-353
 Fax +49 (0) 5137 8238-698
 E-mail: helga.hoffmann@sial.com

USA and Canada

Mr. Doug Clark
 HYDRANAL Technical Center
 545 S. Ewing Ave
 St. Louis MO 63103, USA
 Toll free: +1 800 493-7262 (USA and Canada)
 Fax: +1 314 286-6699
 E-mail: doug.clark@sial.com



Celebrating 30 years of HYDRANAL reagents

Outstanding performance and excellent quality
for Karl Fischer titration

- Improved reagents' safety, simplified use and handling
- Highest quality: large water capacity, high reaction speed, stable end points, excellent reproducibility, and accurate results
- Extensive storage stability and extensive shelf life, no crystallization of reagents (patented formulation)

Please quote promotional code **965**. Offer valid until **December 31, 2010**.

SPECIAL OFFER:

Celebrate with HYDRANAL and receive a 25% discount on these non-toxic HYDRANAL-E-types!

34734	Fluka	HYDRANAL-CompoSolver E
34732	Fluka	HYDRANAL-Titrant 5 E
34723	Fluka	HYDRANAL-Titrant 2 E
34730	Fluka	HYDRANAL-Solvent E
34726	Fluka	HYDRANAL-Coulomat E

Microbiology Base Ingredients

Media consist of diverse interesting base ingredients, ranging from simple sugars to peptone, salts, antibiotics, and more complex indicators.



Figure 1
Fluka® Media Bottle
from Tryptic Soy Agar
and a typical vial of
supplement

A medium has one primary function – to promote the growth of organisms. The components of a medium are often based upon the organism's natural habitat. For example, an organism growing on meat may require meat peptone, and an organism growing on nutrients with a high carbohydrate content may thrive on malt extract. In addition to this growth purpose, media may serve in a number of other applications, including the differentiation and identification of organisms, the selective isolation or enrichment of organisms, and the study of a certain reaction of the organism. A vast array of peptones, extracts and other additives is available to promote and sustain the growth of most organisms.

Proteins (Protein Hydrolysate, Amino Acids, ...)

Although synthetic growth media are available, most media still use complex compounds, such as peptone or yeast extract, since synthetic media lack the complexity and richness of nutrients. Peptones and protein extracts are excellent natural sources of amino acids, peptides, proteins and many other growth factors. They are most often obtained by enzymatic digestion or acid hydrolysis of natural protein sources, such as animal tissues, milk, plants or microbial cultures. The range of available peptones is extensive and comprises a major role in the growth conditions of most organisms (see **Table 1**).

Carbohydrates (Extracts, Sugars, ...)

Carbohydrates are an important energy source. Mono-, di-, oligo- and polysaccharides, as well as natural extracts like rice or malt extracts, provide a versatile possibility of substrates for mold or bacteria cultures. They can also be used to make the media more selective or to identify fermentation profiles. Today, a broad range of media with chromogenic substrates is available (see **Table 2**).

Biological Acids

Pyruvate, one type of biological acid, is known to promote growth and to improve the recovery rate. Other acids such as orange extracts, citric or acetic acid are also used for selective growth.

Buffering Agents

Potassium phosphates are the primary agents used for the buffering system.

Salts

Sodium chloride is used primarily for osmotic balance, however it can also be used to make the medium more selective to halophilic and halotolerant bacteria. In addition,

Jvo Siegrist, Product Manager Microbiology [ivo.siegrist@sial.com](mailto:jvo.siegrist@sial.com)

other salts such as lithium chloride or ammonium bismuth citrate are also used to make the medium more species specific.

Fatty Acids and Lipids

Fatty acids and lipids, such as lecithin, are necessary nutrients and a valuable source of proteins. Fluka offers egg powder and liquid sterile egg supplements, as well as pure lipids and fatty acids.

Vitamins and Trace Elements

Yeast extract, present in numerous complex media, is the most common source for Vitamin B₁₂ (see **Table 3**). Yeast extract also contains a large number of amino acids, additional vitamins and trace elements. Some media also commonly incorporate the addition of pure vitamins and trace elements.

Selective Agents (Detergents, Bile Salts, Antibiotics, ...)

Bile is often used as an inhibitory agent against most gram-positive bacteria. Cholates (see **Table 4**), biological detergent-like compounds with anti-microbial activity, are major constituents of bile. Alternatively, SDS and other detergents are used for the same purpose. For the most part, however, selective agents are comprised of antibiotics that are often added as a mixture in supplemental vials (see **Figure 1**).

Indicators and dyes

These help to indicate biochemical properties or metabolic pathways and are vital for the identification and differentiation of organisms.

Agar

Agar is the solidifying agent in solid growth media, and its selection should be carefully considered based upon certain criteria and dependent upon the application. For example, when high transparency and brightness is needed, as in nutritional studies (Vitamin Assay Media) and sensitivity testing procedures, or when high purity and efficient diffusion of substances is essential, a highly purified agar (Fluka 05038) is recommended. For identification and differentiation, we recommend using a purified or even highly purified agar. However, when isolating a single colony, a standard quality will suffice in most cases. Typical solid media have an agar concentration of 1.0–1.5% to accommodate the requirements of different applications and the growth habits of target microorganisms (see **Table 5**).

(continued on page 8)

Cat. No.	Description
A2427	Amicase
B4888	Beef extract
B3551	Biopeptone
53283	Brain Heart Infusion
C7970	Casein acid hydrolysate vitamin free
22078	Casein from bovine milk
22090	Casein Hydrolysate
39396	Casein Yeast Peptone
C4773	Corn gluten meal
55871	Egg powder
49760	Gluten Hydrolysate from maize
C0501	Hy-Case® Amino
C9386	Hy-Case® SF
57462	Infusion powder from bovine heart
57466	Infusion powder from porcine heart
61300	Lactalbumin hydrolysate
03077	Liver Hydrolysate
70164	Meat extract
C0626	N-Z-Amine® A
C1026	N-Z-Case®
51841	Peptone (vegetable) acid hydrolysate
19942	Peptone (vegetable), No. 1
61854	Peptone (vegetable), No. 2
77180	Peptone from animal proteins
70173	Peptone from casein and other animal proteins
70171	Peptone from casein, acid digest
82303	Peptone from casein, enzymatic digest
70169	Peptone from casein, pancreatic digest
93490	Peptone from Fish
70951	Peptone from gelatin, enzymatic digest
70176	Peptone from gelatin, pancreatic digest
P0521	Peptone from Glycine max (soybean)
70177	Peptone from lactalbumin
93733	Peptone from meat and soybean meal
82962	Peptone from meat, enzymatic digest
70174	Peptone from meat, peptic digest
96174	Peptone from pea
70178	Peptone from soybean
87972	Peptone from soybean, enzymatic digest
90765	Peptone from soybean, enzymatic digest
83059	Peptone from potatoes
93491	Peptone from broadbean
18332	Peptone from vegetable
93492	Peptone from Wheat
P6463	Peptone Hy-Soy® T
P6713	Peptone N-Z-Soy® BL 7
P4838	Peptone Primatone® HS
P4963	Peptone Primatone® RL
P5088	Peptone Primatone® RLT
68971	Peptone special
92976	Peptone special (vegetable)
77199	Peptone, mycological
07915	Potato Extract
P8388	Primatone®
82514	Protein Hydrolysate Amicase®
82524	Protein Hydrolysate N-Z-Amine AS
29185	Proteose Peptone (vegetable)
P0431	Proteose Peptone Enzymatic hydrolysate
82450	Proteose-Peptone
70166	Skim Milk Powder
S1674	Soy protein acid hydrolysate

Cat. No.	Description
70172	Tryptone
95039	Tryptone
16922	Tryptone (vegetable)
61044	Tryptone Plus
70937	Tryptose
12331	Tryptose (vegetable)
05138	Vegetable Extract
04316	Vegetable Extract No. 1
49869	Vegetable Extract No. 2
07436	Vegetable Hydrolysate No. 2
67381	Vegetable Infusion
95757	Vegetable Special Infusion

Table 1 Common protein sources for media (a complete list and links to specification details can be found on sigma-aldrich.com/peptone)

Cat. No.	Description
10850	D(-)-Arabinose
22150	D-(+)-Cellobiose
22160	D-(+)-Cellobiose octaacetate
31405	Dextrin from potato starch for biotechnological purpose
31400	Dextrin from potato starch for microbiology
44590	Dulcitol
47740	D(-)-Fructose
48260	D-(+)-Galactose
49159	D-(+)-Glucose monohydrate
49200	α-D-(+)-Glucose pentaacetate
70167	Malt extract
63560	D-Mannitol
63580	D-(+)-Mannose
63620	D-(+)-Melezitose monohydrate
63630	D-(+)-Melibiose
66940	Methyl α-D-glucopyranoside
67770	Methyl α-D-mannopyranoside
57570	myo-Inositol
07915	Potato Extract
83400	D-(+)-Raffinose pentahydrate
83650	L-Rhamnose monohydrate
95261	Rice extract
84100	Sucrose
90210	D-(+)-Trehalose dihydrate
56217	Verbascose
95720	L(-)-Xylose

Table 2 Common carbohydrate sources for media

Cat. No.	Description
73145	Yeast Autolysate
70161	Yeast Extract (premium quality)
92144	Yeast Extract
09182	Yeast Extract for technical purposes

Table 3 Yeast extracts for media and fermentations

Cat. #	Description
48305	Bile salts
70168	Ox-bile, dehydrated, purified
B8381	Bile from bovine and ovine

Table 4 Bile salts

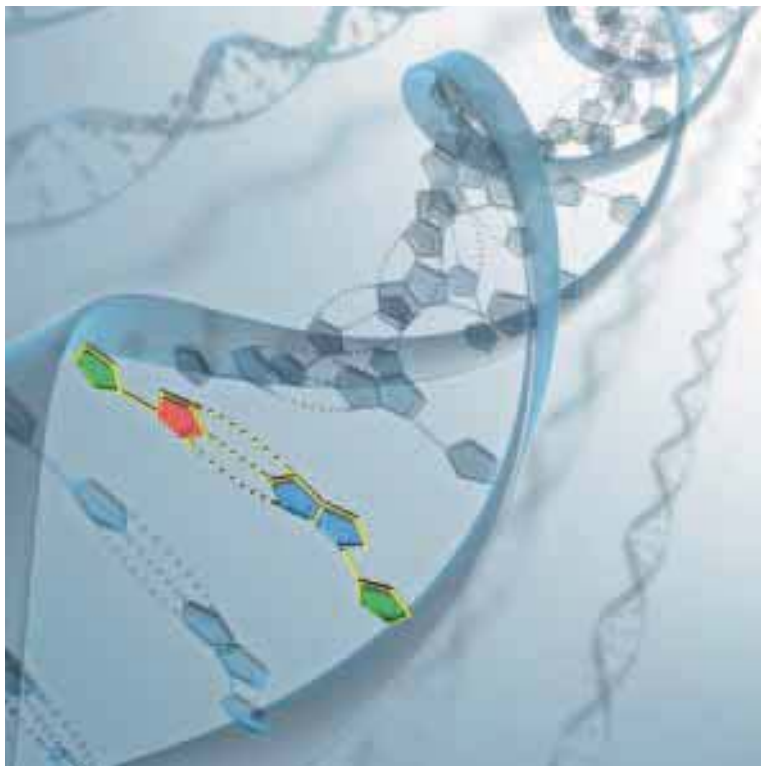
Cat. #	Description
05038	Agar highly purified
05039	Agar purified
05040	Agar standard

Table 5 Agars for microbiology

Better Understanding the Processes of Life

Solution with 17 Amino Acids as *TraceCERT*[®] Certified Reference Material

Jürg Wüthrich, Senior Scientist R&D Europe juerg.wuethrich@sial.com



Chromatographical purity not sufficient

Conventionally, the purity of an organic substance is determined by means of GC or HPLC. The reported chemical composition is determined by identifying the relative amounts of water, residue solvents and inorganic impurities based on information from the generated chromatographs. However, since every chemical substance has its own characteristic absorption behavior, direct traceability is only possible if an internationally accepted CRM is available for the compound in question.

Certification of amino acids by qNMR

Quantitative NMR (qNMR) offers many advantages over other analytical techniques with regard to quantification or purity determination of organic substances. The most outstanding attribute of qNMR is that it is a relative primary method: the signal intensity is in direct proportion to the number of protons contributing to the resonance. Thus, the structures of the chemical substances are completely irrelevant. In addition, no significant empirical factors or unknown biases contribute to the ratio of signals. In other words, the direct response of a qNMR experiment is of highest trueness, leading to certified values with low uncertainties (see also ANALYTIX 03-2010 "Launch of a new generation of organic CRMs").

Production of Amino Acid Solution

The 17 amino acids used as starting materials for the CRM solution have been certified using qNMR for content determination with direct traceability to NIST SRM. These amino acids are also available as neat CRMs as part of our *TraceCERT* product line (see Analytix 03/2010 page 4). The next steps include high-precision weighing of the amino acids and filling the gravimetrically produced bulk solution under argon atmosphere in 2 mL glass ampoules. The complete production process and certification are performed under double accreditation ISO/IEC 17025 and ISO Guide 34.

With this new Fluka[®] branded CRM, scientists working in the field of biochemistry now have a better tool in their hands to obtain a sharper view on the processes of life. For the complete portfolio of the organic CRMs of the *TraceCERT* product line, please check sigma-aldrich.com/organiccrm

Amino acids, especially the proteinogenic alpha-amino acids, are critical to nearly every living organism since they have many functions in metabolism. Of course, their most important function is to act as building blocks of proteins. However, amino acids are also important in many other biological molecules, such as forming parts of coenzymes or as precursors for biosynthesis. Due to this central role in biochemistry, amino acids are very important in nutrition and are commonly used in food technology. They are also used in industry where applications include production of biodegradable plastics, drugs and chiral catalysts.

The most common means of understanding chemical or biochemical processes occurs by measuring relevant analytes using an appropriate analytical method. The more accurate the analytical results, the more dependable are the conclusions that can be found.

The quality of the reference material used for calibration of the instruments is crucial for the reliability of analytical results. Most trustworthy are certified reference materials (CRMs), which must fulfill at least two important requirements: traceability to an internationally accepted reference or reference material, and the certified value must be stated with a properly calculated uncertainty.

(continued on page 10)

Figure 1 Chromatogram of the new certified reference material P/N 79248. The separation of the 17 amino acids is performed using pre-column derivatization (OPA/FMOC derivatives) and reversed-phase chromatography (Hypersil AA-ODS) according to Agilent Application Note 5968-5658E (Angelika Gratzfeld-Huesgen, Sensitive and Reliable Amino Acid Analysis in Protein Hydrolysates using the Agilent 1100 Series HPLC).



Cat. No.	Name/ Packaging	Composition
79248	Amino Acids Mix Solution 5 x 2 mL glass ampoules	2.50 mMol/L: alanine, arginine, aspartic acid, glutamic acid, glycine, histidine, isoleucine, leucine, lysine, methionine, phenylalanine, proline, serine, threonine, tyrosine, valine 1.25 mMol/L: cystine matrix: 0.1 mol/L HCl



TraceCERT[®] Organic and Inorganic CRMs

- CRMs for: **AAS, ICP, IC, qNMR** and **Chromatography**
- Production in accordance with **ISO/IEC 17025** and **ISO Guide 34**
- Superior level of accuracy, calculated uncertainties and lot-specific values
- Traceability to internationally accepted reference materials (i.e. NIST SRM)

For more information and product listings, please visit our website sigma-aldrich.com/inorganiccrm or sigma-aldrich.com/organiccrm. To obtain a copy of our TraceCERT Inorganic CRMs brochure, click the "Request Literature" link at sigma-aldrich.com/standards



Analytical Standards for Disinfection Byproducts (DBPs)

Matthias Nold, Product Manager Analytical Standards matthias.nold@sial.com



The disinfection of drinking water is without doubt one of the biggest achievements for public health within the past century. Many epidemic diseases that were still common in developed countries in the first part of the twentieth century (such as typhoid fever and cholera) have been subdued, largely due to advancements in public sanitation and the treatment of drinking water.

Disinfection is typically done by chemical treatment, most commonly with chlorine. However, chlorine reacts with organic water contaminants to form unintended disinfection byproducts (DBPs). Some of these DBPs have been shown to be carcinogenic or to cause adverse reproductive or developmental effects in laboratory animals. Health risks associated with exposure to DBPs may seem small compared to the benefits of disinfecting drinking water. However, these risks need to be regarded carefully since millions of people are exposed to chlorine disinfected water.

The formation of DBPs may be reduced by removing dissolved organic matter from the water prior to chlorination or by using alternative disinfection techniques, such as ozonation or treatment with UV irradiation. However, since chlorination continues to be the most commonly used disinfection technique, control of the most critical DBPs in drinking water remains very important. In the US, DBPs are regulated by the Disinfectants and Disinfection Byproducts

Rule as part of the Safe Drinking Water Act (SDWA); in Europe, regulations occur in Council Directive 98/83/EC or in the WHO guideline for drinking water quality.

Apart from the inorganic DBPs bromate and chlorite, two organic groups are of primary concern:

Trihalomethanes (THM): This group is comprised of chloroform, bromoform, bromodichloromethane and dibromochloromethane. In the disinfection byproducts rule, total trihalomethanes are regulated at a maximum allowable annual average level of 80 parts per billion.

Haloacetic acids (HAA5): This group includes chloroacetic acid, dichloroacetic acid, trichloroacetic acid, monobromoacetic acid and dibromoacetic acid. The annual average level has been defined at 60 parts per billion by the disinfection byproducts rule.

For the determination of disinfection byproducts, several analytical methods have been published (e.g. ISO 23631:2006, APHA 6232B or 6233B). Usually, the DBPs are analyzed by GC-ECD or GC-MS after liquid-liquid extraction. The HAA5 additionally require derivatization using diazomethane prior to GC injection.

Sigma-Aldrich offers analytical standards for the trihalomethanes as well as for the haloacetic acids. **Table 1** lists the analytical standards available for disinfection byproducts.

Literature

ISO 23631:2006: <http://webstore.ansi.org/RecordDetail.aspx?sku=ISO%2023631:2006>

WHO Guidelines for drinking water quality:

www.who.int/water_sanitation_health/dwq/gdwq3rev/en/

Brand	Cat. No.	Description	Pack Size
FLUKA®	36544	Chloroacetic acid	1 g
FLUKA	36545	Dichloroacetic acid	1 g
FLUKA	31267	Trichloroacetic acid	250 mg
FLUKA	06079	Bromoacetic acid	1 g
FLUKA	77838	Dibromoacetic acid	1 g
FLUKA	36970	Bromodichloromethane	1 g
FLUKA	36971	Dibromochloromethane	1 g
FLUKA	36972	Bromoform	1 g
FLUKA	02487	Chloroform	5 mL
FLUKA	02575	Dichloromethane	5 mL

Table 1 Analytical Standards for Disinfection Byproducts



Primary Standards for the Analysis of Herbal Medicinal Products

- For use in quality control, in-process control and stability testing of herbal medicinal products
- Content assignment by quantitative NMR

For more information and a list of products, please visit our website at sigma-aldrich.com/phytopharma



New Analytical Standards for Sunscreen Lotion Ingredients

Matthias Nold, Product Manager Analytical Standards matthias.nold@sial.com



Sunscreen lotions are used to protect the skin against sunburn and skin cancer caused by the UV-radiation of sunlight. The active ingredients of sunscreen are commonly aromatic compounds which absorb high energy UV-light and release the energy as lower energy rays.

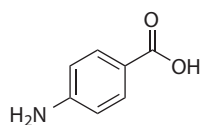
The compounds permitted to be used in sunscreens and the allowed maximum concentration differ between the US, Europe, Japan and other countries due to varying legislation.

The growing use of sunscreens has led to increased concern about environmental contamination and possible effects upon wildlife and human beings.

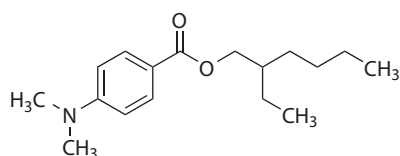
Sigma-Aldrich has now launched a new group of analytical standards for compounds employed as ingredients in sunscreens. **Table 1** provides a listing of our initial set of standards offered in this new product line.

Brand	Cat. No.	Description	US	EU	JP	Pack Size
FLUKA®	01973	4-Aminobenzoic acid	●	●		100 mg
FLUKA	74309	Padimate O	●	●	●	1 mL
FLUKA	92841	Dioxybenzone	●			100 mg
FLUKA	73675	Menthyl anthranilate	●			1 mL
FLUKA	02343	Octocrylene	●	●	●	1 mL
FLUKA	55529	2-Ethylhexyl 4-methoxycinnamate	●	●	●	100 mg
FLUKA	52184	2-Ethylhexyl salicylate	●	●	●	1 mL
FLUKA	50194	Sulisobenzone	●	●	●	100 mg
FLUKA	93632	Zinc oxide	●	●	●	100 mg
FLUKA	66158	3-(4-Methylbenzylidene)camphor		●		100 mg
FLUKA	30184	Bisotrizole		●	●	100 mg

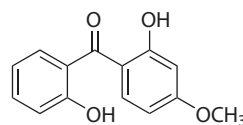
Table 1 First series of analytical Standards for Sunscreen Lotion ingredients



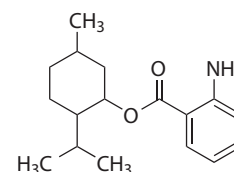
Aminobenzoic acid
01973



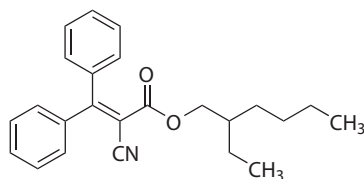
Padimate O
74309



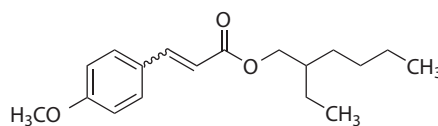
Dioxybenzone
92841



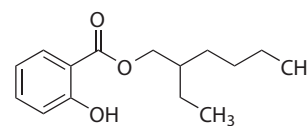
Menthyl anthranilate
73675



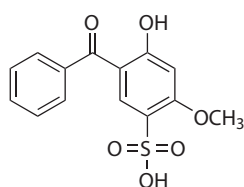
Octocrylene
74309



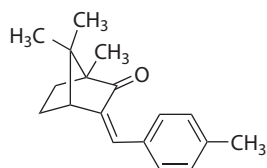
2-Ethylhexyl 4-methoxycinnamate
55529



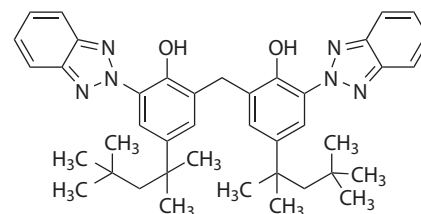
2-Ethylhexyl salicylate
52184



Sulisobenzone
50194



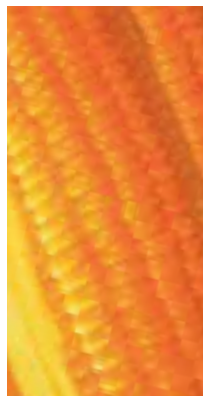
3-(4-Methylbenzylidene)camphor
66158



Bisotrizole
30184

New certified reference material from the IRMM

Matthias Nold, Product Manager Analytical Standards matthias.nold@sial.com



The IRMM (Institute of Reference Materials and Measurements) is one of the seven institutes of the Joint Research Center, a Directorate-General of the European Commission.

Within this structure, the IRMM supplies certified reference materials (pure and matrix materials) for various applications, including environmental analysis, food analysis, clinical chemistry, physical properties or industrial applications. Sigma-Aldrich is proud to be an authorized distributor of IRMM reference materials.

The CRMs for the content of genetically modified organisms (GMO) constitute an important group among the IRMM product range. These standards are available as sets of



products containing plant material (maize powder or cotton seed) with different mass fractions of the GMO material. These products are produced by mixing GMO-containing material with GMO-free material gravimetrically in different ratios.

Table 1 lists GMO standards recently added to the portfolio. **Table 2** lists recently added matrix standards for environmental, food or clinical analysis.

Brand	Description	Pack Size
Maize GMO Standard 98140		
ERMBF427A	0% 98140 maize	1 g
ERMBF427B	0.5% 98140 maize	1 g
ERMBF427C	2% 98140 maize	1 g
ERMBF427D	10% 98140 maize	1 g
Cotton seed GMO standard GHB119		
ERMBF428A	0% GHB119 Cotton seed	1 g
ERMBF428B	1% GHB119 Cotton seed	1 g
ERMBF428C	10% GHB119 Cotton seed	1 g
Cotton seed GMO standard T304-40		
ERMBF429A	0% T304-40 Cotton seed	1 g
ERMBF429B	1% T304-40 Cotton seed	1 g
ERMBF429C	10% T304-40 Cotton seed	1 g

Table 1 New GMO standards

Cat. No.	Material	Certified for	Application Area	Pack Size
ERMCD281	Rye Grass	Element Content	Environmental	10 g
ERMBB350	Fish oil	Organic pollutants	Environmental	2 g
ERMCA408	Simulated rain water	Conductivity; pH; elemental content	Environmental	95 mL
ERMCA616	Ground water	Conductivity; pH; elemental content	Environmental	95 mL
ERMBB130	Pork muscle	Chloramphenicol	Food	7.5 g
ERMBC381	Rye flour	Ash; Kjehldahl (N); Starch, Total fat; Ca, K, Mg, P	Food	37 g
ERMBC382	Wheat flour	Ash; Kjehldahl (N); Starch, Total fat; Ca, K, Mg, P	Food	37 g
ERMBB384	Pork Muscle	Ash; Kjehldahl (N); Starch, Total fat; Ca, Mg, Na, P	Food	2 x 18 g
ERMDA471/IFCC	Human serum	Cystatin C	Health	1 vial

Table 2 New matrix CRMs from the IRMM

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

R-(+)- α -Lipoic acid (RLA) is an important cofactor in aerobic metabolism. Attached to a lysine side chain group via an amide bond, it is part of several enzyme complexes, such as the pyruvate dehydrogenase complex (PDC). In the course of the enzymatic reaction, its reduced form, dihydrolipoic acid, is generated. Both compounds are very abundant in nature and are, therefore, present in many foods, especially kidney, heart, liver, spinach, broccoli and yeast extract.

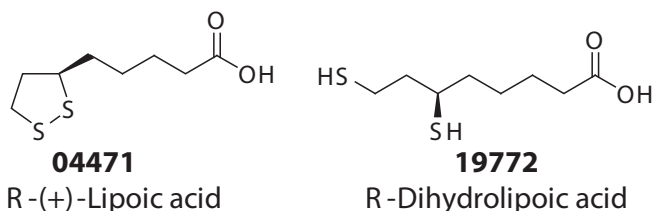
Since it was discovered that R-Lipoic acid acts not only as an antioxidant, but also demonstrates anti-toxin effects against a wide range of chemical toxins, it has been widely used as a food supplement as well as for clinical applications.

Brand	Cat. No.	Description	Pack Size
FLUKA*	04471	R-(+)-Lipoic acid	100 mg
FLUKA	19772	R- α -Dihydrolipoic acid	100 mg

Matthias Nold, Product Manager Analytical Standards matthias.nold@sial.com

Although only the R form is bioactive, the synthetically produced racemic form (R/S-LA) is commonly used because it is less expensive and more readily available.

Sigma-Aldrich has now launched new analytical standards for the naturally occurring R enantiomer of lipoic acid as well as for its reduced form, R-Dihydrolipoic acid.



Metabolites – A Serious Challenge for LC/MS Separations and Usual Detection Techniques

Although the genome of several organisms has been decoded, the knowledge about the expressed proteins and their complex interactions and catalyzed chemical reactions remains incomplete. Metabolomics encompasses the study of all compounds and reaction products taking part in these cellular processes and regulating them. Metabolomics may be applied to a single cell or an organism as a whole [1].

Biosynthesis of Cholesterol and Fatty-Acids (Biofuels)

Cholesterol is an important molecule in the human body. It is incorporated in cell membranes and stabilizes the lipid bi-layers. Modifications to this molecule by other enzymes yield another important group of compounds: steroid hormones. These hormones assume a role in signal transduction and control the protein biosynthesis; for example, estrogens such as estradiol are responsible for the growth of female genitals and regulate the menstruation. Surprisingly, the cholesterol building blocks also take part in the biosynthesis of fatty-acids, which can replace mineral oil-based fuels [2].

Rudi Köhling, Senior Scientist LC/MS Application rudolf.koehling@sial.com

Figure 1 displays some of the steps of the pathway leading to the formation of cholesterol. In a large number of these reactions, a phosphorylation of hydroxyl moieties with ATP takes place, which in turn means that very hydrophilic compounds coexist with hydrophobic compounds, and a separation by chromatography places varying demands on the different stationary phases.

The example above provides only a small insight into the innumerable reactions with thousands of reaction products and intermediates. Some of these metabolites have pathological relevance and refer to several different disease patterns, e. g. phenylketonuria (PKU).

Sigma-Aldrich offers the tools and the knowledge for the study of metabolic processes and the analysis of the metabolites. A large number of certified reference standards, enzymes, substrates, and chromatographic products can be found on the Sigma-Aldrich website.

(continued on page 16)

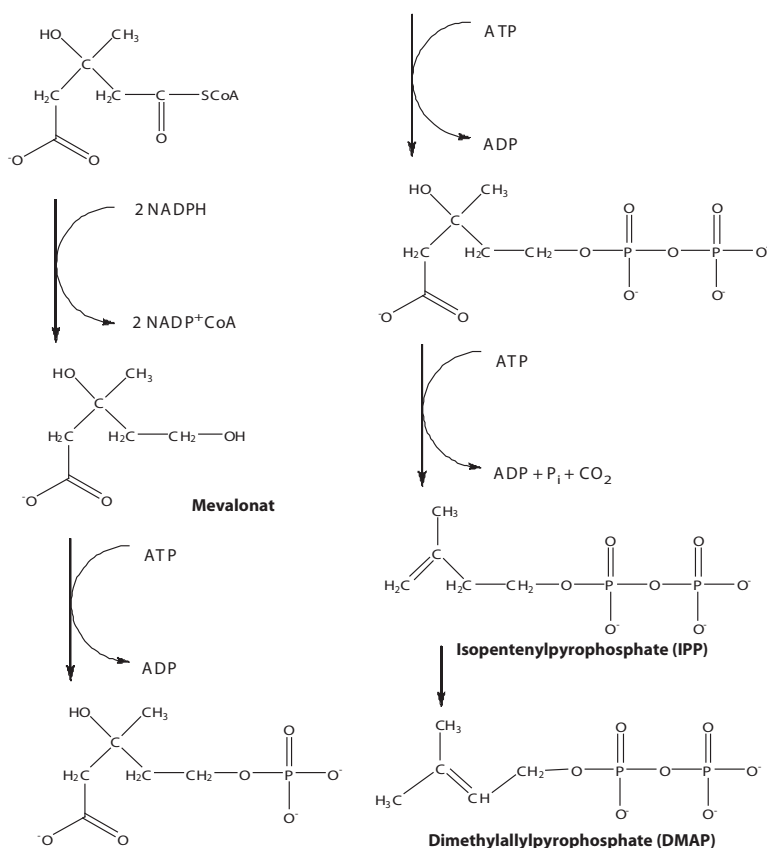


Figure 1 Biosynthesis of IPP and DMAP via mevalonate pathway. These isoprenoid phosphates are the smallest units involved in the production of a large number of vitamins, coenzymes, hormones, lipids.

Compound Group	Relevance	Link
Myo-inositol phosphates	Signal transduction	sigma-aldrich.com/metabolomics
Amino acids	Nutrition/proteins	sigma-aldrich.com/metabolomics
Isoprenoide phosphates	Fatty-acids (biofuels)/hormones	sigma-aldrich.com/metabolomics

Table 1 Links to compound groups used in this study for further information

Analysis of Metabolites

NMR, LC/MS, CE(/MS) and GC/MS are typically the methods of choice to separate, identify and quantify in the analysis of biological samples. However, most of the metabolites, especially those listed in **Table 1**, are very polar and do not show any UV absorption, which excludes the use of UV or fluorescence detectors for HPLC. Sample size: approx. 0.3 g. The sample can be easily handled using a syringe without a needle.

The Corona CAD represents a new detection technique which closes the gap between optical and mass detectors and offers both sensitivity and responses, depending on the concentration of the analyte. It utilizes ionization processes comparable to APCI sources and detects charged particles. The particles are formed by drying the mobile phase with nitrogen, which is also used to charge the

particles by passing a corona discharge needle. These detectors have gained increasing importance in analytical laboratories because of their ease of use, low price and high sensitivity compared to other detectors, e. g. refractive index. They can be used for controlling the quality, purity and content of pure metabolites (**Figures 2 and 3**).

Liquid Chromatography

The separation of such polar compounds by HPLC poses a challenge for the method development. Ion exchange, HILIC or some chiral stationary phases (e. g. Chirobiotic™/Cyclobond columns) may be a good starting point for LC method development using MS or CAD detection. High buffer concentrations and varying pH conditions influence retention and peak shape crucially, which is useful for the optimization. Myo-inositol tri- and -pentaphosphates can be separated by HILIC using a Supelco® Ascentis Express® HILIC column. **Figure 3** shows the extracted ion chromatograms of the [M+H]⁺ ions.

Another more difficult separation is related to the isoprenoid phosphate compounds in **Figure 1**. Isopentenyl phosphate (IP), dimethylallyl phosphate (DMAP) and their related pyrophosphates, IPP and DMAPP, differ only in the position of the double bond. However, the HPLC separation is possible with a Supelco Cyclobond I 2000 stationary phase (**Figure 4**).

Finally, the separation of OPA-derivatized amino acids is presented in **Figure 5**. There are additional methods for the analysis of amino acids by HPLC described in the literature [3-5]. Most of these techniques use pre-column derivatization to change the polarity of the analytes and to make them detectable for UV and fluorescence detectors. Unfortunately, additional peaks of the derivatization agent may overlay with analyte peaks; however, this problem can be avoided by using guard columns and switching between various wave lengths.

The OPA/FMOC reaction [4] can easily be transferred to Ascentis Express C8/C18 columns; in particular, standard HPLC systems profit from their very high resolution. Only a 5 cm column is necessary to separate all 16 amino acid derivatives within a run-time of 10 min (Agilent® 1200 system).

The various methods and techniques used for metabolomics all require the use of reference standards. Sigma-Aldrich has introduced a new quality of organic TraceCERT® standards, beginning with a series of amino acids, which are used as external standards for the determination of amino acid concentrations in various matrices. Amino acids are also important biomarkers for some hereditary metabolic diseases and can be detected within a prenatal/neo-

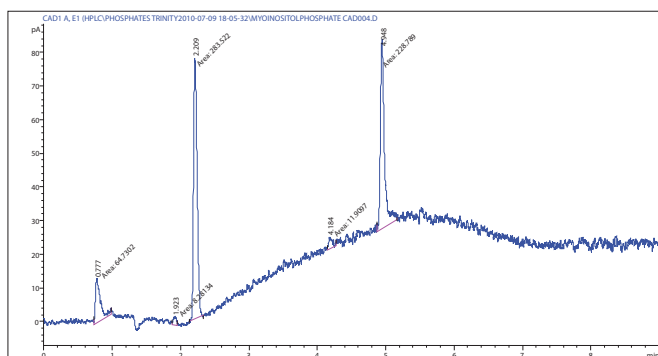


Figure 2 CAD chromatogram of myo-ionisitol triphosphate sodium salt. One special feature of this detector is the ability to detect alkali cations as well (2.2 min). Mobile phase compositions can be transferred from LC/MS methods as the Corona CAD also depends on volatile additives.

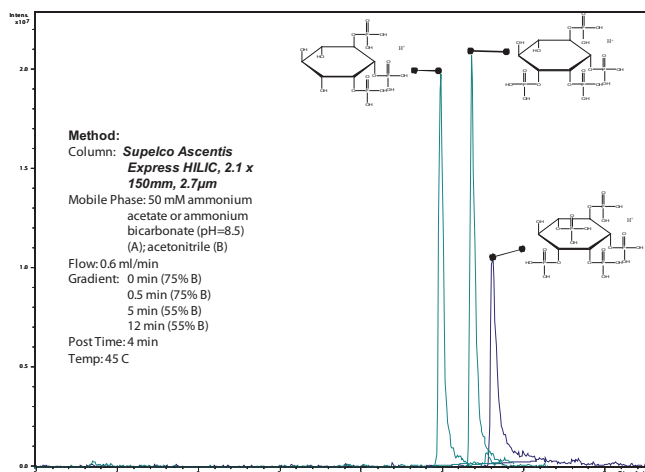


Figure 3 Separation of myo-inositol phosphates by HILIC. The method is suitable for MS and CAD detection. Best peak shapes can be obtained at a higher pH.

natal screening [1,5]. The precision of the analysis strongly depends upon the exact knowledge of the analyte content in the reference standards. This is guaranteed by using high-precision qNMR measurements and the production of such standards in an accredited laboratory (see previous article by J. Wüthrich).

Part. No.	Product
53946-U	Supelco® Ascentis Express® HILIC, 2.1 x 150 mm, 2.7 µm
53831-U	Supelco Ascentis Express C8, 2.1 x 50 mm, 2.7 µm
20124AST	Supelco Cyclobond I 2000, 4.6 x 250 mm, 5 µm
34967-1L	Acetonitrile for LC/MS
34966-1L	Methanol for LC/MS
39253-1L	Water for LC/MS
73594	Ammonium acetate for mass spectrometry
40867	Ammonium bicarbonate for LC/MS
79248-2ML	AA Mix Solution

Table 2 Related Products (additional LC/MS products are listed at www.sigma-aldrich.com/lc-ms)

References

- [1] Voet, Voet, Biochemistry, 2nd edition.
- [2] R.Köhling et al, Analytix 3/2010, pp. 18–19.
- [3] T. Takeuchi, Journal of Chromatography Library, 70, 2005, pp. 229–241.
- [4] C. Woodward et al, Agilent Application Note 5989–6297 (2007).
- [5] M. Zoppa, Journal of Chromatography B, 831 (2006) 267–273.

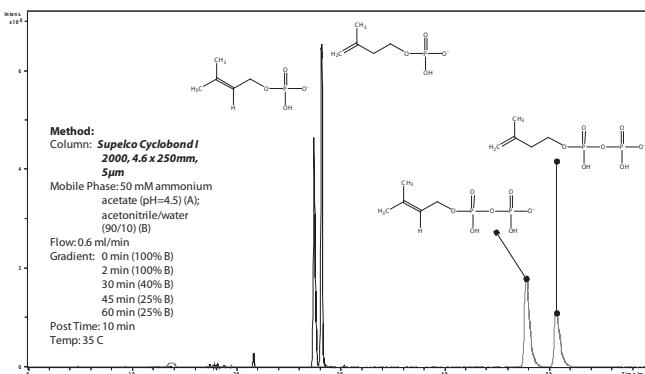


Figure 4 Separation of IP, DMAP, IPP and DMAPP by chiral chromatography with a cyclodextrine phase (Cyclobond I 2000).

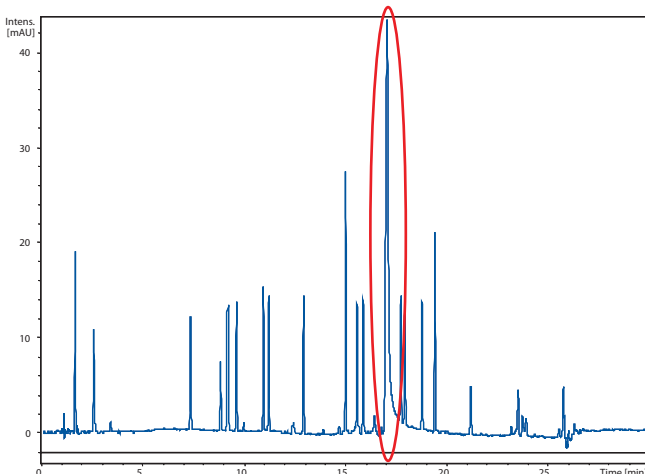


Figure 5 Separation of 15 amino acids (OPA derivatives) using pre-column derivatization and reversed-phase chromatography (Ascentis Express C8 column) according to [4]. All amino acid derivatives as well as the agents are separated very well. The peak of the OPA reagent can be suppressed by using another wave length [4].

Silyl methallylsulfonates – New Highly Efficient Silylating Reagent

Chemoselective silylation of alcohols, polyols, phenols and carboxylic acids

Xiaogen Huang, Ph.D.

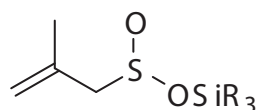
Contact: michael.kiselewsky@sial.com



Silylation is an important tool in both analytical and synthetic chemistry. Silyl derivatization of alcohols, carboxylic acids and phenols is widely applied in the field of modern organic synthesis. Classical methods involve trialkylsilyl halides or triflates combining with a stoichiometric amount of a tertiary amine. Other methods employ silazanes, hexa-methyldisilazane, hydrosilanes, disilanes, alkyl silanes, methallyl silanes, trimethylsilyl azide and silyl phosphines. Quite often, a catalytic amount of acid is introduced to enhance the transformations. We describe here a series of efficient silylating reagents (**Figure 1**) with which the silylation process does not require any acid or base to promote the conversion.

Reagents **1a-c** were combined with different types of alcohol as shown below (**Figure 2**). It was observed that **1a** and **1b** reacted with primary, secondary and tertiary alcohols with approximately the same reaction rate. The conversions were completed in 5 min. Only 1.0 equivalent of **1a-b** was required. Normally, 1.05-1.1 equivalents of reagents are employed to ensure the completion of the reaction.

For **1c**, when it was treated with primary alcohol, the reaction was also completed in 5 min. However, the reactions with secondary and tertiary alcohols were slower. The slow reaction rate with secondary alcohol was easily addressed by effective stirring and vacuum pumping techniques. For tertiary alcohols, 1.5 eq. of **1c** were introduced, and the reaction was held for a sufficiently long time. The excess of silylating reagents could be simply quenched with MeOH generating volatile by-products. Compounds with fragile structures, sensitive to acids or bases, survived under our silylation conditions.



1a, R = Me (Fluka® 79271)
1b, R = Et (Fluka 79264)
1c, R = TBDM (Fluka 79262)

Figure 1 Structure of Silyl methallylsulfonates

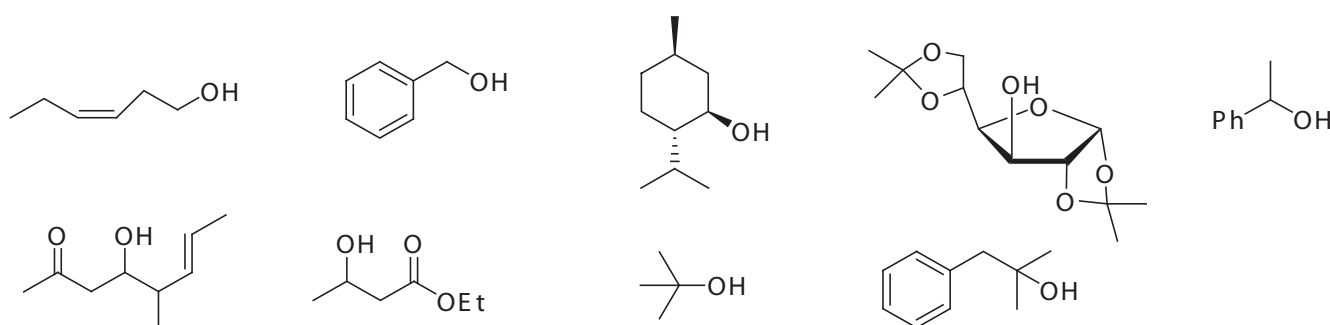


Figure 2 Variety of functional hydroxyl groups for silylation reaction. Even base-sensitive substrates, such as β -hydroxy ketones, can be smoothly silylated.

The effectiveness of our silylating reagents is well demonstrated by the following example **2** could not be silylated under nearly any of the different silylating conditions before the invention of our reagents. The difficulty in attaining silylation is due to steric hindrance. However, with **1b**, the transformation proceeds smoothly into a TES-derived product (**Figure 3**). This is a prime example illustrating the accessible sites in **1a-c** that facilitate direct interaction without dependence on additional reagents. Two is enough; when they impinge, then there is transfer.

We also explored the ability of our reagents to discriminate different types of hydroxy groups present in the same molecule. It was found that it followed the basic steric progres-

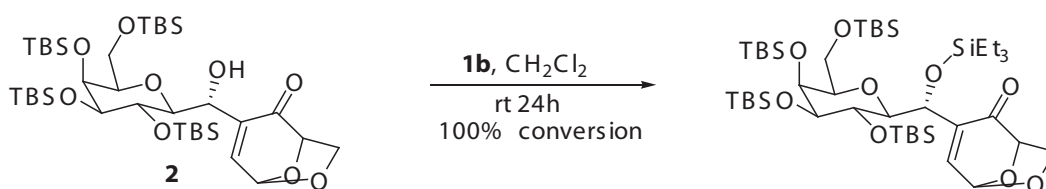


Figure 3 Smooth transformation of sterically hindered substrates by TES **1b**.

sion: primary > secondary > tertiary (**Figure 4**). Carboxylic acids can also be easily silylated. It is interesting to mention that the silylation of primary alcohol is dominant over that of phenol.

In conclusion, we describe here a new series of silylating reagents which demonstrate the following advantages: (1) the silylating process does not require any additive; (2) the

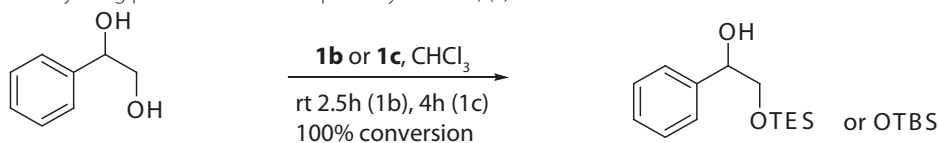


Figure 4 Selective silylation of diols with 1 equiv. of **1b** or **1c**.

Brand	Cat. No.	Description	Pack Size
FLUKA®	79271	Trimethylsilyl methallylsulfinate, for GC derivatization	5 mL
FLUKA	79264	Triethylsilyl methallylsulfinate, for GC derivatization	5 mL
FLUKA	79262	tert-Butyldimethylsilyl methallylsulfinate, for GC derivatization	5 mL

process is a kind of face-to-face mode in which one reagent and one substrate talk with each other and the silyl group is attached within a few minutes; (3) the solvent may not be necessary if the substrate is a liquid.

Further Reading

[1] Xiaogen Huang, Cotinica Craita, Loay Awad and Pierre Vogel, Silyl methallylsulfonates: efficient and powerful agents for the chemoselective silylation of alcohols, polyols, phenols and carboxylic acids, *Chem. Commun.*, 2005, 1297–1299.



Your Day-to-Day Demand for TLC Plates



Sigma-Aldrich offers you a new quality of TLC plates on aluminium with a standard silica gel matrix. Our TLC plates now provide a reliable and easy-to-cut sheet and an outstanding wettability. Discover the excellent separation efficiency for your daily thin layer chromatography workload.

Material	Brand	Name	Size	Fluorescence Indicator	Thickness of Layer
49859-50EA	Supelco®	Silica gel on TLC-Al foils	4 cm x 8 cm	254 nm	0.20 mm
52038-20EA	Supelco	Silica gel on TLC-Al foils	5 cm x 7.5 cm	254 nm	0.20 mm
23478-50EA	Supelco	Silica gel on TLC-Al foils	5 cm x 10 cm	254 nm	0.20 mm
12606-50EA	Supelco	Silica gel on TLC-Al foils	5 cm x 20 cm	254 nm	0.20 mm
56524-25EA	Supelco	Silica gel on TLC-Al foils	20 cm x 20 cm	254 nm	0.20 mm
55811-20EA	Supelco	Silica gel on TLC-Al foils	5 cm x 7.5 cm	without	0.20 mm
75196-50EA	Supelco	Silica gel on TLC-Al foils	5 cm x 10 cm	without	0.20 mm
92572-50EA	Supelco	Silica gel on TLC-Al foils	5 cm x 20 cm	without	0.20 mm
53356-25EA	Supelco	Silica gel on TLC-Al foils	20 cm x 20 cm	without	0.20 mm

Silica with an average pore size of 60 Å, specific pore volume 0.75 mL/g and a particle size of 5–17 µm, for reliable and reproducible results. A new binder system offers easy cutting.

Headspace Grade Solvents

for the Analysis of Organic Volatile Impurities

Michael Kiselewsky, Product Manager Chromatography Reagents & Michael Jeitziner, Market Segment Manager Analytical Reagents & Standards

michael.kiselewsky@sial.com



Static headspace GC (GC-HS) is a technique used to concentrate volatile analytes prior to analysis. It can improve detection of low levels of volatile analytes and minimizes matrix interference by eliminating the need to inject the sample directly. An important application of GC-HS is for the determination of residual volatile organic impurities in active drug substances or excipients in drug formulations. Other consumer-oriented applications include the detection of residual solvents in foods, dietary supplements and packaging materials.

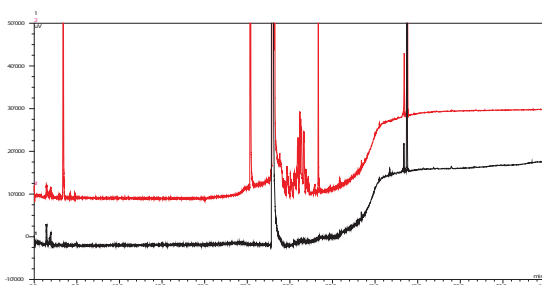


Figure 1 Headspace gas chromatogram of two DMSO grades: GC-HS grade (black trace) and conventional grade (red trace) [2]

GC-HS is a relatively straightforward technique and the methodology, as it applies to residual solvents in pharmaceuticals, is described and validated in specific monographs [1-3]. These guidelines recommend both the types of solvents and the acceptable levels of residual solvents in pharmaceuticals and formulations to help ensure consumer safety.

New Headspace grade solvents

When developing a GC-HS method, such parameters as sample solvent, extraction temperature, extraction time, sample volume and headspace volume are optimized [4, 5]. Because the composition and purity of the sample solvent have significant effects on the recovery and quality of the chromatogram (see **Figure 1**), we have developed solvents specifically for GC-HS applications. Their purity and handling specifications meet the requirements of European Pharmacopoeia (Ph.Eur.) and United States Pharmacopoeia (USP), as well as ICH guidelines. The new GC-HS line includes water and three of the most commonly used organic solvents: dimethyl sulfoxide (DMSO), N,N-dimethylformamide (DMF) and N,N-dimethylacetamide (DMA). DMF and DMSO are specified in Ph.Eur. and USP for water-insoluble substances. Water is the preferred solvent for water-soluble solutions, as described in Ph.Eur. and USP monographs. All solvents are microfiltered at 0.2 μm and packed under inert gas for longer shelf life.

References

- [1] United States Pharmacopeia, 31st Edition (2008), <467> Residual Solvents.
- [2] Ph.Eur. 6.0 (2008) Method 2.4.24, Identification and control of residual solvents.
- [3] ICH Guideline Q3C, Impurities: Guideline for Residual Solvents, The Fourth International Conference on Harmonization, July 17, 1997.

For more information, please visit our website:

sigma-aldrich.com/gc-hs

Brand	Cat. No.	Product Name	Abbreviation	Boiling Point	Pack size
FLUKA®	NEW 68809	Cyclohexanone, for GC-HS	-	155 °C	1 L
FLUKA	44901	N,N-Dimethylacetamide, for GC-HS	DMA	166 °C	1 L
FLUKA	51781	N,N-Dimethylformamide, for GC-HS	DMF	153 °C	1 L
FLUKA	67484	1,3-Dimethyl-2-imidazolidinone, for GC-HS	DMI	225 °C	100 mL, 1 L
FLUKA	51779	Dimethyl sulfoxide, for GC-HS	DMSO	189 °C	1 L
FLUKA	NEW 69337	1-Methyl-2-pyrrolidinone, for GC-HS	NMP	202 °C	1 L
FLUKA	53463	Water, for GC-HS	-	100 °C	1 L

Product Table Solvents for GC Headspace Analysis

New High Purity Salts

for melting digestion in environmental, water, and food analysis

Michael Jeitziner, Market Segment Manager Analytical Reagents & Standards michael.jeitziner@sial.com



Melting digestion is used for solid samples such as ores, rock, metals, alloys, ceramic, and cement, in order to obtain a homogenous residue which can be dissolved in diluted **TraceSELECT**® acids. Our **TraceSELECT** salts are very high purity, with metal traces typically below 10 µg/kg (10 ppb). Purity and composition are guaranteed with our careful preparation, testing, and verification of the final product for metal content and ionic trace impurities using ICP-OES, ICP-MS and ion chromatography. To further guarantee purity and stability, **TraceSELECT** products are packaged in high-quality containers appropriate for the particular product.

The Sigma-Aldrich Quality Management System guarantees consistent quality and safety for all **TraceSELECT**Ultra and **TraceSELECT** products. The reagents are produced and bottled under clean-room conditions and are delivered with a Certificate of Analysis.

For more information, please visit our website:
sigma-aldrich.com/traceselect

Cat. No.	Brand	Product Name	Specification	Pack Size
73432	FLUKA®	Ammonium acetate	≥ 99.9999%	100 g
09725	FLUKA	Ammonium chloride	≥ 99.9995%	25 g, 100 g
09726	FLUKA	Ammonium phosphate monobasic	≥ 99.9999%	100 g
09979	FLUKA	Ammonium sulfate	≥ 99.9999%	100 g
40581	FLUKA	Cesium bromide	≥ 99.999%	25 g
90033	FLUKA	Cesium chloride	≥ 99.9995%	25 g, 100 g
62462	FLUKA	Lithium carbonate	≥ 99.995%	25 g, 100 g
60348	FLUKA	Potassium bisulfate	≥ 99.999%	25 g, 100 g
60111	FLUKA	Potassium carbonate	≥ 99.995%	50 g
05257	FLUKA	Potassium chloride	≥ 99.9995%	25 g, 100 g
60371	FLUKA	Potassium hydroxide hydrate	≥ 99.995%	25 g
30533	FLUKA	Potassium iodide	≥ 99.999%	100 g
60429	FLUKA	Potassium nitrate	≥ 99.995%	25 g, 100 g
60216	FLUKA	Potassium phosphate monobasic	≥ 99.995%	25 g, 100 g
60347	FLUKA	Potassium phosphate dibasic	≥ 99.999%	100 g
59929	FLUKA	Sodium acetate	≥ 99.999%	25 g, 100 g
01963	FLUKA	Sodium bromide	≥ 99.995%	25 g
71347	FLUKA	Sodium carbonate	≥ 99.9999%	25 g, 100 g
38979	FLUKA	Sodium chloride	≥ 99.999%	25 g, 100 g
01968	FLUKA	Sodium hydroxide monohydrate	≥ 99.9995%	25 g, 100 g
71752	FLUKA	Sodium nitrate	≥ 99.999%	25 g, 100 g
71629	FLUKA	Sodium phosphate dibasic	≥ 99.999%	100 g
71492	FLUKA	Sodium phosphate monobasic	≥ 99.999%	25 g, 100 g
44355	FLUKA	Tin(II) chloride dihydrate, for AAS	≥ 99.999%	250 g, 2.5 kg

Product Table **TraceSELECT** salts

High quality titration reagents for all your needs in volumetric titration

Special offer for ready-to-use volumetric standard solutions

Andrea Felgner, Product Manager Analytical Reagents andrea.felgner@sial.com

Sigma-Aldrich offers a wide variety of volumetric acid, base and salt standard solutions in different concentrations that can be used for diverse volumetric titration applications, such as acidimetric, alkalimetric, redox and precipitation titration methods. More information is available on our website sigma-aldrich.com/titration

The following Volumetric Solutions are available with a **HUGE 45% OFF** savings:

Brand	Cat. No.	Description
FLUKA®	35245	Sodium thiosulfate solution, Reag. Ph.Eur., 0.1 mol/L
FLUKA	35418	Perchloric acid solution, 0.1 mol/L in acetic acid
FLUKA	35328	Hydrochloric acid solution, Reag. Ph.Eur., 1 mol/L
FLUKA	35329	Hydrochloric acid solution, Reag. Ph.Eur., 0.5 mol/L
FLUKA	35335	Hydrochloric acid solution, Reag. Ph.Eur., 0.1 mol/L
FLUKA	35354	Sulphuric acid solution, Reag. Ph.Eur., 0.5 mol/L
FLUKA	35256	Sodium hydroxide solution, Reag. Ph.Eur., 1 mol/L
FLUKA	35257	Sodium hydroxide solution, 0.5 mol/L
FLUKA	35263	Sodium hydroxide solution, Reag. Ph.Eur., 0.1 mol/L
FLUKA	35115	Potassium hydroxide solution, 0.5 mol/L denat. ethanol with Toluene
FLUKA	35127	Potassium hydroxide solution, 0.1 mol/L denat. ethanol with Toluene

To take advantage of this offer, please use promotion code 976. Offer is valid until December 31, 2010.







Volumetric Titration Brochure

All you need to know about reagents for volumetric titration from Sigma-Aldrich:

- Ready-to-use solutions
- Concentrates
- pH buffers
- Indicators

Get your free copy on our website sigma-aldrich.com/titration, or order it by ticking the box on the attached reply card, or contact your local sales representative. (Brochure Code LNH)

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 9650
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

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 5923

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



*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
Development/Custom Manufacturing Inquiries **SAFC® (800) 244-1173**
Safety-related Information sigma-aldrich.com/safetycenter

World Headquarters
3050 Spruce St.
St. Louis, MO 63103
(314) 771-5765
sigma-aldrich.com

©2010 Sigma-Aldrich Co. All rights reserved. SIGMA, SAFC, SIGMA-ALDRICH, ALDRICH, FLUKA, and SUPELCO are trademarks belonging to Sigma-Aldrich Co. and its affiliate Sigma-Aldrich Biotechnology, L.P. Sigma brand products are sold through Sigma-Aldrich, Inc. Sigma-Aldrich, Inc. warrants that its products conform to the information contained in this and other Sigma-Aldrich publications. Purchaser must determine the suitability of the product(s) for their particular use. Additional terms and conditions may apply. Please see reverse side of the invoice or packing slip.

Date: 11/2010;
SAMS Code: MSX