

Analytix

Advances in Analytical Chemistry

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 **Fluka**

Riedel-deHaën®

Celebrating 25 Years of HYDRANAL®

Titration

- VOLPAC® Package

Standards

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- GC-MS Tuning
- Stable Isotopes
- FAME Solutions

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- Ferrioxamine E
- Identification of *Salmonella*

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- EPA 324 Mercury Traps

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- MALDI-MS
- Trace Analysis

New Product Corner

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SIGMA-ALDRICH

Small Unit Packaging Service Put Supelco's experience with preparation of a wide variety of analytical standards, lot retention and shelf-life tracking to use in your lab



Picture Don Hobbs, Director of Marketing, Supelco

Dear Colleague,

Some of our most successful ideas for products and services have come from customers like you.

One example is Supelco's Small Unit Packaging Service.

Several years ago, a customer in the pharmaceutical industry asked us to consider a repackaging project. They required that a bulk reference material be repackaged into smaller units to protect its integrity. By having a ready source of ampulized product, two of their research chemists could get back to doing research, rather than constantly aliquoting reference material for other labs.

Repackaging requires careful attention to details, including labeling, documentation, cleaning and distribution. The benefits of using Supelco's Small Unit Packaging Service include our experience with preparing a wide variety of analytical standards and our ability to retain lots and track shelf-life.

Today, Supelco's Small Unit Packaging Service handles several thousand units on a weekly basis. Our customers include pharmaceutical, diagnostic, petrochemical, industrial companies and environmental testing labs. We have extensive experience repackaging solid active pharmaceutical ingredients, stains and dyes, biocides, proteins, light sensitive vitamins, solvents and calibration solutions. One customer's request has resulted in Sigma-Aldrich becoming a valued supply-chain partner.

Supelco's small unit repackaging service has grown to include:

- Customized and OEM labeling
- Custom documentation and certificates
- Qualitative and quantitative testing
- Lot control and inventory management
- Distribution services for product samples

We are able to focus on giving you comprehensive and easy-to-use solutions, so that you can focus on the other demands of your business.

What can we do for you?

If you would like to learn if we have the right packaging solution for your company, please contact our Technical Service Team. Their information appears on the back page of this Analytix issue. Our chemists will enjoy answering your questions and finding the right solution for your business challenge.

We hope you can put our Small Unit Package Service and the other fine Sigma-Aldrich products and services described in this issue of the Analytix newsletter to work for you today.

Regards,

Don Hobbs
Director of Marketing
Supelco/Division of Sigma-Aldrich
dhobbs@sial.com



Picture

Supelco facilities in
Bellefonte, PA (USA)

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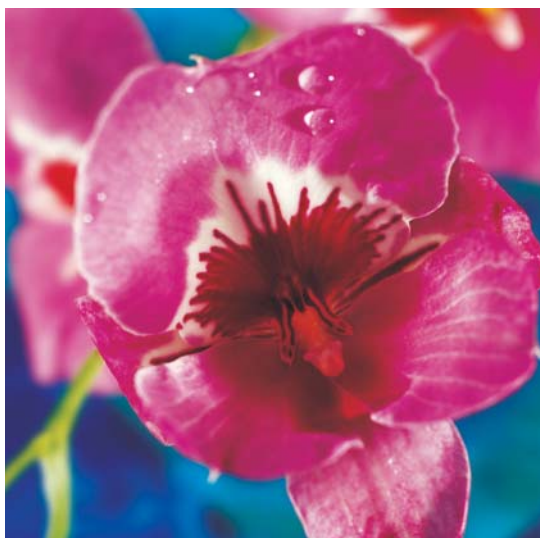
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Celebrating 25 Years of HYDRANAL® The history of HYDRANAL® demonstrates continuous innovations in reagents and techniques for moisture determination by Karl Fischer titration

By Michael Jeitziner, Product Manager, Fluka / Riedel-de Haën...mjeitziner@sial.com



This year marks the 25th anniversary of HYDRANAL®, Sigma-Aldrich's high quality products for reliable moisture determination by Karl Fischer titration. Whether or not Riedel-de Haën chemist Eugen Scholz who founded the HYDRANAL® line foresaw its impact we'll never be sure, but today HYDRANAL® users are found in every industry sector, from foods to semi-conductors, from pharmaceuticals to petrochemicals around the globe. This article highlights the key developments in the HYDRANAL® story.

Karl Fischer titration

Scientists have long realized the influence moisture content has on chemical processes, from promoting or inhibiting chemical reactions to altering product stability and shelf life and promoting microbial growth. However, a defined, reliable and truly practical method for moisture measurement was beyond the reach of analysts until 1935 when chemist Karl Fischer published his manuscript "New procedure for the determination of the water content in liquids and solids." (1)

Dr. Fischer outlined a procedure, called Karl Fischer titration, which nearly any laboratory interested in moisture determination could follow. Since then, the Karl Fischer method has become one of the most frequently employed methods in analytical chemistry. But the technique was open to improvement; some of the reagents were toxic or harmful to the environment, including sulfur dioxide, iodine, pyridine, benzene and methanol,

reactions could be slow, endpoints were not always clear and some substrates were difficult to titrate accurately. Beginning in 1979, Riedel-de Haën chemists Eugen Scholz and Helga Hoffmann looked at ways to improve the Karl Fischer titration, making it safer, more accurate, easier to use and applicable to a wider range of substrates. These improvements became the foundation of the HYDRANAL® product line.

Innovation No. 1: Replacing the pyridine

One of the first areas of improvement was the replacement of the noxious base, pyridine. Dr. Scholz's research showed that pyridine could be replaced by bases that were both safer and more effective. The most significant results were published in the Fresenius journal "Zeitschrift für Analytische Chemie" in the series entitled "Karl Fischer Reagents without Pyridine." (2-10) The initial publications reported the use of diethanolamine as a base, while later research concluded that imidazole has advantages in some titrations. The HYDRANAL® range of reagents includes both imidazole and diethanolamine, which are relatively safe, have excellent buffering capacity and produce rapid and accurate titrations. Which base you choose depends upon your sample matrix. Our expert HYDRANAL® technical service will help you select the right one for your application.

Innovation No. 2: New reagents

Dr. Scholz's investigations soon turned toward the development of reagents and techniques to improve upon the classic Karl Fischer titrations, reducing the number of components required, making endpoints clearer and more stable, reactions faster and more sensitive. The one-component reagent, HYDRANAL®-Composite, which contains imidazole as the base, was introduced in 1980. The first two-component reagent containing diethanolamine as the base, HYDRANAL®-Solvent and HYDRANAL®-Titrant, was introduced in 1980. This two-component reagent allowed a rapid titration with a stable end point. In 1986, the diethanolamine was changed to imidazole to improve the buffering capacity of the system and increase the water capacity of the solvent to 7 mg/mL. In later years HYDRANAL®-Coulomat A and HYDRANAL®-Coulomat C enabled coulometric detection to be applied to moisture analysis. The range of titration reagents was extended with the addition of HYDRANAL®-Standards, titration reagents for back titrations and buffering solutions.

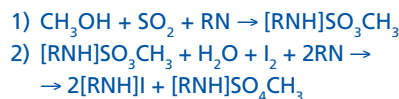
Innovation No. 3: Improving reagent safety

The foundation of the HYDRANAL® line and the first safety innovation was replacing toxic pyridine. Safety improvements continued in 1991 when a new set of coulometric reagents free of halogenated hydrocarbons was introduced into the HYDRANAL® line. Toxic methanol was addressed by the methanol-free reagents used for the determination of water in aldehydes and ketones: HYDRANAL®-K reagents (HYDRANAL®-Composite 5K and HYDRANAL®-Medium K for volumetric determination, HYDRANAL®-Coulomat AK and HYDRANAL®-Coulomat CG-K for coulometric determination). Our most recent introductions are the non-toxic HYDRANAL®-E Type reagents. We are committed to further research to improve the safety of HYDRANAL® reagents while enhancing their efficacy.

Innovation No. 4: Defining the science

While the reagent safety and efficacy studies were underway in Dr. Scholz's lab, fundamental questions about the chemistry behind the Karl Fischer reaction were being raised, including the verification of the stoichiometry of the reaction. It was concluded that the well known equations for the Karl Fischer reaction did not fully describe its observed reaction course and required further investigation. The results of these investigations undertaken by Dr. Scholz and his team have since been published in both English and German (11).

The chemical principle behind the Karl Fischer titration is the following set of equations:



(RN = Base)

The sulfur dioxide reacts with the alcohol (methanol or ethanol) to form an ester which is neutralized by the base. The anion of the alkylsulfurous acid is the reactive component and is already present in the Karl Fischer reagent. The titration of water constitutes the oxidation of the alkylsulfite anion to alkylsulfate by the iodine. This reaction consumes water that is derived from the sample. Since water and iodine (I₂) are consumed in a 1:1 stoichiometric ratio, the amount of water in the original sample is calculated by measuring the concentration of I₂ remaining after the reaction is complete.

Two significant prerequisites are necessary to assure a stoichiometric course of the Karl Fischer reaction. First, a suitable alcohol must be present to esterify the sulfur dioxide completely (reaction 1). Second, a suitable base is necessary for the complete neutralization of the acids produced during the reaction. The basicity of pyridine is too low to completely neutralize the acid and is the cause of the sluggish titration observed when using the classic pyridine-containing Karl Fischer reagents. However, if the base is too strong the solution becomes too alkaline and an end point will not be reached. A titration in the pH range of 5–7 is preferred. Maintaining the proper pH throughout the titration is of prime importance.



Picture HYDRANAL®-Literature overview

Patents

The results of Dr. Scholz and his team's investigations were of such fundamental importance that patents were received for many of the new reagents and their use. So far, more than fifty patents have been issued or are pending in Europe, the US, Japan and other countries. A new US patent was accepted in September 2005 for product improvements to the HYDRANAL®-Composites.

HYDRANAL®-Reagents

The HYDRANAL® product range consists of both one-component and two-component reagents for volumetric and coulometric determinations and special reagents for the determination of water in ketones and other problematic substrates. The HYDRANAL® product range is complemented by buffer solutions and calibration standards for titre determination.

The instrumentation used in Karl Fischer titrations is neither complex nor expensive, but none-the-less it is specially designed for the application. HYDRANAL®

reagents are compatible with all brands of titrators. In our laboratories, we develop applications on a variety of manufacturers' equipment to ensure HYDRANAL® reagents will work for you in your laboratory.

Technical support

As one of the key scientists in the HYDRANAL® story, Helga Hoffmann still heads up the HYDRANAL®-Team. As a result of our quarter century of research into Karl Fischer titrations, we have built up quite a large knowledge base that is at your disposal to help you choose and properly use the right HYDRANAL® reagent and technique for your sample and analytical requirements. Please contact our HYDRANAL®-Laboratories for help with both the principles behind Karl Fischer titration and how to choose and use the proper reagents and methods for your samples. We can also offer specific assistance in the form of the actual analysis of difficult samples (free of charge) and send you complete protocols for your samples.

For more details about HYDRANAL®-Reagents, please visit our website
www.sigma-aldrich.com/hydranal.

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References:

- [1] K. Fischer, "Neues Verfahren zur Bestimmung des Wassergehaltes in Flüssigkeiten und Feststoffen," *Angew. Chemie* **1935**, 48, 394.
- [2] E. Scholz, "Karl-Fischer-Reagenzien ohne Pyridin," *Fresenius Z. Anal. Chem.* **1980**, 303, 203.
- [3] E. Scholz, "Karl-Fischer-Reagenzien ohne Pyridin (2). "Wasserbestimmung in Cysteinhydrochlorid-Monohydrat.," *Fresenius Z. Anal. Chem.* **1981**, 305, 416.
- [4] E. Scholz, "Karl-Fischer-Reagenzien ohne Pyridin (3). "Die Genauigkeit der Wasserbestimmung," *Fresenius Z. Anal. Chem.* **1981**, 306, 394.
- [5] E. Scholz, "Karl-Fischer-Reagenzien ohne Pyridin (4). "Einkomponenten-Reagenzien," *Fresenius Z. Anal. Chem.* **1981**, 309, 30.
- [6] E. Scholz, "Karl-Fischer-Reagenzien ohne Pyridin (5). "Neue Eichsubstanzen," *Fresenius Z. Anal. Chem.* **1981**, 309, 123.
- [7] E. Scholz, "Karl-Fischer-Reagenzien ohne Pyridin (6). "Wasserbestimmung von Carbonsäuren.," *Fresenius Z. Anal. Chem.* **1982**, 310, 423.
- [8] E. Scholz, "Karl-Fischer-Reagenzien ohne Pyridin (7). "Zweikomponenten-Reagenzien mit Imidazol.," *Fresenius Z. Anal. Chem.* **1982**, 312, 460.
- [9] E. Scholz, "Karl-Fischer-Reagenzien without Pyridine (8). "Coulometric Determination of Water," *Fresenius Z. Anal. Chem.* **1983**, 314, 567.
- [10] E. Scholz, "Karl-Fischer-Reagenzien ohne Pyridin (9). "Die Wasserbestimmung in Lebensmitteln," *Dt. Lebensm. Rundsch.*, **1983**, 79, 302.
- [11] E. Scholz, "Karl-Fischer Titration," Springer Verlag, Berlin, Heidelberg, New York, Tokyo, 1984 (ISBN 3-540-12846-8 German Edition, ISBN 3-540-13734-3 English Edition).

For a comprehensive listing of HYDRANAL® products, request a copy of Analyx Notes HYDRANAL® Product Overview (HRM) using the enclosed reply card, or visit us on-line at www.sigma-aldrich.com/hydranal

New VOLPAC® Package Sizes 5 liter VOLPAC® bags complement the popular 10 liter size

By Michael Jeitziner, Product Manager, Fluka / Riedel-de Haën...mjeitziner@sial.com

A common task facing nearly every scientist is the preparation of buffer, acid, base and reagent solutions. If this sounds like you, then consider switching to convenient pre-prepared solutions from Sigma-Aldrich in VOLPAC®-containers. VOLPAC®-containers will save preparation time and guarantee consistency, purity and accuracy.

The 10 L and new 5 L VOLPAC®-containers consist of a flexible polyethylene bag with an outlet tap and a cubic cardboard supporting frame. As the volume is depleted, the design of the bag prevents air entering the bag, eliminating the possibility of contamination – even to the very last drop!

Advantages of VOLPAC®-containers include:

- High quality of contents and packaging
- Easy handling
- No contamination during use
- Small space requirement
- Reduced packing material
- Easy disposal of empty containers



Picture 5 liter VOLPAC®-containers

Table 1 VOLPAC® Volumetric Solutions

Cat. No.	Brand	Product	Package Size*
33643	Riedel-de Haën	Buffer solution pH 4.0 (20 °C); (citric acid / sodium hydroxide solution / sodium chloride) solution ready for use, with fungicide	5 L, 10 L
33646	Riedel-de Haën	Buffer solution pH 7.0 (20 °C); (potassium dihydrogen phosphate / di-sodium hydrogen phosphate) solution ready for use, with fungicide	5 L, 10 L
33648	Riedel-de Haën	Buffer solution pH 9.0 (20 °C); (borax / hydrochloric acid) solution ready for use	5 L, 10 L
35375	Riedel-de Haën	Silver nitrate solution; Ph Eur 0.1 M	5 L, 10 L
34544	Riedel-de Haën	IDRANAL® B; IDRANAL® III solution with zinc complex added, for water hardness determination (1 mL = 1 German degree of hardness in 100 mL of water)	5 L, 10 L
34550	Riedel-de Haën	IDRANAL® III standard solution; Ph Eur 0.1 M	5 L, 10 L
35102	Riedel-de Haën	IDRANAL® III standard solution; 0.2 M	5 L, 10 L
35103	Riedel-de Haën	IDRANAL® IV standard solution; Ph Eur 0.1 M	5 L, 10 L
34631	Riedel-de Haën	Potassium dichromate standard solution; for COD determination according to DIN 38409, part 41; 0.02 M	5 L, 10 L
35245	Riedel-de Haën	Sodium thiosulfate standard solution; Ph Eur 0.1 M	5 L, 10 L
35256	Riedel-de Haën	Sodium hydroxide standard solution; Ph Eur 1.0 M	5 L, 10 L
35257	Riedel-de Haën	Sodium hydroxide standard solution; 0.5 M	5 L, 10 L
35263	Riedel-de Haën	Sodium hydroxide standard solution; Ph Eur 0.1 M	5 L, 10 L
35328	Riedel-de Haën	Hydrochloric acid standard solution; Ph Eur 1.0 M	5 L, 10 L
35329	Riedel-de Haën	Hydrochloric acid standard solution; Ph Eur 0.5 M	5 L, 10 L
35335	Riedel-de Haën	Hydrochloric acid standard solution; Ph Eur 0.1 M	5 L, 10 L
35357	Riedel-de Haën	Sulfuric acid standard solution; Ph Eur 0.1 M	5 L, 10 L
35358	Riedel-de Haën	Sulfuric acid standard solution; Ph Eur 0.05 M	5 L, 10 L

* All of the 10 L sizes are available in Europe only

Custom Standard Solutions for All of Your Analytical Applications The Sigma-Aldrich family's extensive product offering allows you to access thousands of chemicals that can be customized and engineered to fit your standard or reference solution needs.



Picture Custom Standard solutions

The extensive product offering of the Sigma-Aldrich family allows you to access thousands of chemicals that can be customized and engineered to fit your standard or reference solution needs. We can formulate, test and package custom standard solutions for chromatographic, spectroscopic and titrimetric applications. Our custom standard chemists will gladly discuss stability and solubility concerns with you, and make suggestions where needed to maximize the quality of your standard. You can rely on Sigma-Aldrich custom standard solutions to include:

- Raw materials and solvents screened for identity and purity

- Your choice of gravimetric, qualitative or quantitative testing
- Packaging choices from ampoules to bottles
- Manufacturing processes that follow ISO 9001/2000 guidelines
- Documentation and Material Safety Data Sheets
- Strict adherence to all shipping regulations
- Free and capable technical support before and after your purchase

If you are interested in a customized standard, please contact us at customstandards@sial.com

GC-MS Tuning Solutions, Internal and Surrogate Standards Optimize the reliability of your GC-MS results using these high quality, convenient products from Supelco

By Kathy Kiefer, R&D Chemical Standards Chemist, [Supelco...kkiefer@sial.com](mailto:kkiefer@sial.com)
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Because of its sensitivity and specificity, GC-MS is a widely used technique in environmental and forensic chemistry. Some important applications within these markets include many EPA methods, drug abuse monitoring and fire and explosives residue investigations. To get the most out of your GC-MS analysis, it is important to choose the proper internal standards, surrogates and tuning solutions, like those offered by Sigma-Aldrich under its Supelco brand.

Tuning solutions

Tuning solutions help ensure the accuracy of the mass and ion abundance measurements by auto-tuning the GC-MS instrument. Tuning solutions contain compounds that are specified in the particular EPA method and are important tests for the qualification of the GC-MS system.

Internal standards

GC-MS protocols typically utilize the internal standard method of quantification. Internal standards gauge the

method's ability to extract target analytes from the sample matrix, and are used to monitor and adjust instrument fluctuations. Pentafluorobenzene, 1,4-difluorobenzene, 1,4-dichlorobenzene- d_4 and chlorobenzene- d_5 are examples of internal standards.

Surrogate standards

Surrogate standards are used to monitor the entire analytical process from sample preparation to instrument performance. Toluene- d_8 , 4-bromofluorobenzene and dibromofluorobenzene are examples of surrogate standards.

The table below lists the GC-MS standards and tuning solutions available from Sigma-Aldrich. A Certificate of Analysis is supplied with each product. The single and multi-component solutions are certified for purity, identity and concentration. Each component is guaranteed to be $\pm 0.5\%$ of the stated concentration.

Table 1 Surrogate standard solutions

Cat. No.	Brand	Description	Package Size
48466	Supelco	EPA 524 Surrogate Standard Mix (1,2-Dichlorobenzene- d_4 , Fluorobenzene)	1 mL
47960-U	Supelco	8270 Surrogate Standard (4000 $\mu\text{g/mL}$ in methylene chloride: 2,4,6-Tribromophenol, 2-Fluorobiphenyl, 2-Fluorophenol, Nitrobenzene- d_5 , Phenol- d_6 , p-Terphenyl- d_{14})	1 mL
48593	Supelco	Toluene- d_8 Solution (2000 $\mu\text{g/mL}$ in methanol)	1 mL
48083	Supelco	1-Bromo-4-fluorobenzene (2000 $\mu\text{g/mL}$ in methanol)	1 mL
48941	Supelco	1,2-Dichloroethane- d_4 Solution (2000 $\mu\text{g/mL}$ in methanol)	1 mL
48940-U	Supelco	Benzene- d_6 Solution (2000 $\mu\text{g/mL}$ in methanol)	1 mL

Table 2 Tuning solutions

Cat. No.	Brand	Description	Package Size
47077	Supelco	EPA 8240B/8260A GC-MS Tuning Mix (25 µg/mL methanol: 4-Bromofluorobenzene)	1 mL
47415	Supelco	EPA 8270 GC-MS Tuning Solution (50 µg/mL of each in methylene chloride: Benzidine, 4,4'-DDT, Pentachlorophenol)	4 x 1 mL
47387	Supelco	EPA 8270 GC-MS Tuning Solution (50 µg/mL of each in methylene chloride: Benzidine, 4,4'-DDT, Pentachlorophenol)	1 mL
47548-U	Supelco	EPA 8270 GC-MS Tuning Solution II (1000 µg/mL of following in methylene chloride: Benzidine, 4,4'-DDT, DFTPP, Pentachlorophenol)	1 mL
442543	Supelco	Decafluorotriphenylphosphine (DFTPP)	100 mg NEAT
47941	Supelco	Decafluorotriphenylphosphine (DFTPP) (25 000 µg/mL in acetone)	1 mL
48724-U	Supelco	Decafluorotriphenylphosphine (DFTPP) (25 000 µg/mL in methylene chloride)	1 mL
48082	Supelco	Decafluorotriphenylphosphine (DFTPP) (2000 µg/mL in methylene chloride)	1 mL
48800	Supelco	1-Bromo-4-fluorobenzene (25 000 µg/mL in methanol)	1 mL
48945	Supelco	Pentafluorobenzene Solution (2000 µg/mL in methanol)	1 mL
48727	Supelco	Benzidine-DFTPP (250 µg/mL each in methanol)	1 mL
48728	Supelco	Pentachlorophenol-DFTPP (250 µg/mL each in methanol)	1 mL

Table 3 Internal standard solutions

Cat. No.	Brand	Description	Package Size
48948	Supelco	EPA 524.2 Internal Standard Mix (2000 µg/mL each in methanol: 1,2-Dichlorobenzene-d ₄ , Fluorobenzene)	1 mL
47358-U	Supelco	EPA 524.2 Internal Standard Mix (2000 µg/mL each in methanol: 1,2-Dichlorobenzene-d ₄ , 4-Bromofluorobenzene, Fluorobenzene)	1 mL
48864	Supelco	EPA Purgeable Internal Standard Mix (20 000 µg/mL each in methanol: 1,4-Dichlorobutane, 1-Chloro-2-bromopropane, Bromochloromethane)	1 mL
47081-U	Supelco	EPA 8620 Internal Standards Mix (25 000 µg/mL in methanol: 1,4-Dichlorobenzene-d ₄ , 1,4-Difluorobenzene, Chlorobenzene-d ₅ , Pentafluorobenzene)	1 mL
48958	Supelco	EPA 8620 Internal Standards Mix (1000 µg/mL in methanol: 1,4-Dichlorobenzene-d ₄ , 1,4-Difluorobenzene, Chlorobenzene-d ₅ , Pentafluorobenzene)	1 mL
47082-U	Supelco	EPA 8620 Internal Standards Mix (10 000 µg/mL in methanol: 1,4-Dichlorobenzene-d ₄ , 1,4-Difluorobenzene, Chlorobenzene-d ₅ , Pentafluorobenzene)	1 mL
47776	Supelco	EPA 8620 Internal Standards Mix (250 µg/mL in methanol: 1,4-Dichlorobenzene-d ₄ , Chlorobenzene-d ₅ , Fluorobenzene)	1 mL
47777	Supelco	EPA 8620 Internal Standards Mix (25 µg/mL in methanol: 1,4-Dichlorobenzene-d ₄ , Chlorobenzene-d ₅ , Fluorobenzene)	1 mL
46955-U	Supelco	Semivolatile Internal Standards Mix (2000 µg/mL in methylene chloride: 1,4-Dichlorobenzene-d ₄ , Acenaphthene-d ₁₀ , Chrysene-d ₁₂ , Naphthalene-d ₈ , Perylene-d ₁₂ , Phenanthrene-d ₁₀)	1 ml
48086	Supelco	Chlorobenzene-d ₅ Solution (2000 µg/mL in methanol)	1 mL
48943	Supelco	Fluorobenzene Solution (2000 µg/mL in methanol)	1 mL
48086	Supelco	1,4-Chlorobenzene-d ₄ Solution (2000 µg/mL in methanol)	1 mL
48792	Supelco	Decafluorobiphenyl (2000 µg/mL in methylene chloride)	1 mL
48790-U	Supelco	4,4'-Dibromobiphenyl (2000 µg/mL in methylene chloride)	1 mL
48791	Supelco	4,4'-Dibromoctafluorobiphenyl (2000 µg/mL in methylene chloride)	1 mL
48787	Supelco	2,2'-Difluorobiphenyl (2000 µg/mL in methylene chloride)	1 mL
48720-U	Supelco	1-Fluoronaphthalene (2000 µg/mL in methylene chloride)	1 mL
48721-U	Supelco	2-Fluoronaphthalene (2000 µg/mL in methylene chloride)	1 mL
48715-U	Supelco	Naphthalene-d ₈ (2000 µg/mL in methylene chloride)	1 mL
48717-U	Supelco	Naphthalene-d ₅ (2000 µg/mL in methylene chloride)	1 mL
48710-U	Supelco	Phenanthrene-d ₁₀ (2000 µg/mL in methylene chloride)	1 mL
48714-U	Supelco	Pyridine-d ₅ (2000 µg/mL in methylene chloride)	1 mL
48788	Supelco	Aniline-d ₅ (2000 µg/mL in methylene chloride)	1 mL
48718	Supelco	Pentafluorophenol (2000 µg/mL in methylene chloride)	1 mL

New Deuterated Standards Stable isotopes for environmental and other analytical applications

By Rainer Walz, Product Manager Fluka / Riedel-de Haën...rwalz@sial.com

At the request of our customers and regulatory agencies, we have expanded our line of mass-labelled analytical standards to include standards that address and comply with new regulations in Europe, US, Japan and elsewhere. To ensure isotopic purity, the synthesis of these isotopes is carried out in dedicated laboratories, separate from other manufacturing operations and rigorously tested.

Table 1 Nitrofurans

Cat. No.	Brand	Description	Package Size
33880	Riedel-de Haën	AOZ-d ₄ (3-Aminooxazolidin-2-one-d ₄)	10 mg
33881	Riedel-de Haën	AMOZ-d ₅ (3-Amino-5-morpholinomethyl-2-oxazolidinone-d ₅)	10 mg
33882	Riedel-de Haën	SCA- ¹³ C- ¹⁵ N ₂ HCl (Semicarbazide- ¹³ C- ¹⁵ N ₂ hydrochloride)	10 mg
34006	Riedel-de Haën	AHD- ¹³ C ₃ (1-Amino-2,4-imidazolidinedione- ¹³ C ₃)	10 mg
34008	Riedel-de Haën	2-NP-AOZ-d ₄ (3-(2-Nitrobenzylidenamino)-2-oxazolidinone-d ₄)	10 mg
34009	Riedel-de Haën	2-NP-AMOZ-d ₅ (5-(Morpholinomethyl)-3-(2-nitrobenzylidenamino)-2-oxazolidinone-d ₅)	10 mg
34010	Riedel-de Haën	2-NP-AHD- ¹³ C ₃ (1-(2-Nitrobenzylidenamino)-2,4-imidazolidinedione-[^{2,4,5-¹³C}])	10 mg
34011	Riedel-de Haën	2-NP-SCA- ¹³ C, ¹⁵ N ₂ (2-Nitrobenzaldehyde semicarbazone- ¹³ C, ¹⁵ N ₂)	10 mg
34061	Riedel-de Haën	Furaltaldon-d ₅ 5-[Morpholino(methyl-d ₂)]-3-(5-nitrofurfurylidenamino)-2-oxazolidinone-d ₃	10 mg

Table 2 Pesticides

Cat. No.	Brand	Description	Package Size
34017	Riedel-de Haën	Isoproturon-d ₆ (3-(4-Isopropylphenyl)-1,1-dimethylurea-d ₆)	10 mg
34018	Riedel-de Haën	Diuron-d ₆ (3-(3,4-Dichlorophenyl)-1,1-dimethylurea-d ₆)	10 mg
34019	Riedel-de Haën	Carbofuran-d ₃ (2,3-Dihydro-2,2-dimethyl-7-benzofuranol N-methylcarbamate-d ₃)	10 mg
34021	Riedel-de Haën	4,4'-DDT-d ₈ (1,1,1-Trichloro-2,2-bis(4-chlorophenyl)ethane-d ₈)	10 mg
34023	Riedel-de Haën	2,4,6-Trichloranisol-d ₅	50 mg
34053	Riedel-de Haën	Atrazin-d ₃ 2-Chloro-4-pentadeuteroethylamino-6-isopropylamino-1,3,5-triazine	10 mg
34054	Riedel-de Haën	Simazine-d ₁₀ 2,4-Bis(pentadeuteroethylamino)-6-chloro-1,3,5-triazine	10 mg

Table 3 Antibiotics

Cat. No.	Brand	Description	Package Size
34058	Riedel-de Haën	Norfloxacin-d ₃ 6-Fluoro-1,4-dihydro-4-oxo-1-pentadeuteroethyl-7-piperazino-3-quinolinecarboxylic acid	10 mg
34083	Riedel-de Haën	Flunixin-d ₃ 2-[2-Methyl-d ₃ -3-(trifluoromethyl)phenylamino]nicotinic acid	10 mg
34109	Riedel-de Haën	Meloxicam-d ₃ (4-Hydroxy-2-(methyl-d ₃)-1,1-dioxo-benzo[e]-1,2-thiazine-3-carboxylic acid (5-methyl-2-thiazolyl)amide)	50 mg

Table 4 Coming soon: More deuterated standards

Compound	Class	Compound	Class
Bentazon-d ₇	Herbicide	Sudan-IV-d ₆	Dye
Dicamba-d ₃	Herbicide	Sudan-I-d ₅	Dye
Imidacloprid-d ₄	Insecticide	Leucomalachite green-d ₅	Dye
Linuron-d ₆	Herbicide	DNC-d ₈ (4,4-Dinitrocarbanilide-d ₈)	Antiprotozoal
Pirimicarb-d ₆	Insecticide	Dimetridazol-d ₃	Antibiotic
Prometryn-d ₇	Herbicide	HMMNI-d ₃	Antibiotic
Terbutryn-d ₅	Herbicide	Ipronidazol-d ₃	Antibiotic
Triforine-d ₈	Fungicide, Insecticide	Ronidazol-d ₃	Antibiotic

Take advantage of our special introductory offer of 20% off the listed mass-labelled analytical standards. Please quote code U52 when placing your order. Offer valid until 31 January 2006.

Solutions for Fatty Acid Analysis Supelco brand FAME standards and capillary GC columns from Sigma-Aldrich

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

For the food analyst, determining the fatty acid composition of a product is difficult because foods usually comprise a complex mixture of saturated, monounsaturated and polyunsaturated fatty acids with a variety of carbon chain lengths. For example:

- Milk and butter contain saturated C4 to C20, monounsaturated C16 and C18 and polyunsaturated C18 fatty acids
- Vegetable oils contain saturated C6 to C24, monounsaturated C16 and monounsaturated cis C18, C20 and C22 fatty acids
- Margarines contain the same fatty acids as vegetable oils plus monounsaturated trans C18, C20, and C22 and polyunsaturated C18 fatty acids
- Fish and meat contain saturated and monounsaturated fatty acids, plus polyunsaturated omega-3 C18, C20, and C22 and polyunsaturated omega-6 C18 and C20 fatty acids
- Fish tends to be richer in the polyunsaturated omega-3 fatty acids, whereas meats are richer in the polyunsaturated omega-6 fatty acids

To confirm the identification of key fatty acids, several different combinations of standards and capillary GC columns may be required. One such combination is the

Supelco 37 Component FAME Mix and the 75 m SP-2560 column shown in Figure 1. This standard contains methyl esters of fatty acids ranging from C4 to C24, including key monounsaturated and polyunsaturated fatty acids. It is very useful to food analysts because it can be used to identify fatty acids in many different types of foods.

Supelco's complete line of standards for food analysis includes:

- Underivatized fatty acids
- Fatty acid methyl ester (FAME) derivatives
- Triglycerides
- Highly characterized reference oils

You can find these standards conveniently packaged as:

- Multi-component mixes
- Single-component solutions
- Neat compounds
- Kits of either single-component solutions or of neat compounds

Whatever your food analysis application, Supelco likely has the standard to meet your requirements.

Table 1 Product listing

Part No.	Brand	Description	Package size
47885-U	Supelco	37 component FAME mix (See Figure 1 legend for ingredients, in CH ₂ Cl ₂)	1 mL
23348-U	Supelco	SP-2560 capillary GC column 75 m x 0.18 mm I.D., 0.14 μm d _f	1 unit

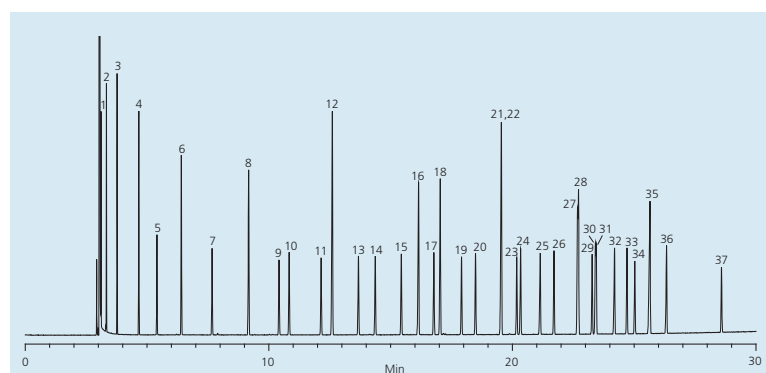


Figure 1
37 Component FAME
Mix resolved

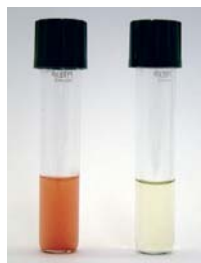
column: SP-2560, 75 m x 0.18 mm I.D., 0.14 μm (23348-U)
oven: 140 °C (5 min.), 4 °C/min. to 240 °C (2 min.)
inj.: 250 °C
det.: FID, 250 °C
carrier gas: hydrogen, 40 cm/sec @ 175 °C
injection: 1 μL, split 100:1
liner: 4 mm I.D split, cup design
sample: 37 component FAME mix at concentrations listed in methylene chloride (47885-U)

Peak IDs

1. Butyric Acid Methyl Ester (C4:0) at 4 wt %
2. Caproic Acid Methyl Ester (C6:0) at 4 wt %
3. Caprylic Acid Methyl Ester (C8:0) at 4 wt %
4. Capric Acid Methyl Ester (C10:0) at 4 wt %
5. Undecanoic Acid Methyl Ester (C11:0) at 2 wt %
6. Lauric Acid Methyl Ester (C12:0) at 4 wt %
7. Tridecanoic Acid Methyl Ester (C13:0) at 2 wt %
8. Myristic Acid Methyl Ester (C14:0) at 4 wt %
9. Myristoleic Acid Methyl Ester (C14:1) at 2 wt %
10. Pentadecanoic Acid Methyl Ester (C15:0) at 2 wt %
11. cis-10-Pentadecenoic Acid Methyl Ester (C15:1) at 2 wt %
12. Palmitic Acid Methyl Ester (C16:0) at 6 wt %
13. Palmitoleic Acid Methyl Ester (C16:1) at 2 wt %
14. Heptadecanoic Acid Methyl Ester (C17:0) at 2 wt %
15. cis-10-Heptadecenoic Acid Methyl Ester (C17:1) at 2 wt %
16. Stearic Acid Methyl Ester (C18:0) at 4 wt %
17. Elaidic Acid Methyl Ester (C18:1n9t) at 2 wt %
18. Oleic Acid Methyl Ester (C18:1n9c) at 4 wt %
19. Linolelaic Acid Methyl Ester (C18:2n6t) at 2 wt %
20. Linoleic Acid Methyl Ester (C18:2n6c) at 2 wt %
21. Arachidic Acid Methyl Ester (C20:0) at 4 wt %
22. γ-Linolenic Acid Methyl Ester (C18:3n6) at 2 wt %
23. Linolenic Acid Methyl Ester (C18:3n3) at 2 wt %
24. cis-11-Eicosenoic Acid Methyl Ester (C20:1) at 2 wt %
25. Heneicosanoic Acid Methyl Ester (C21:0) at 2 wt %
26. cis-11,14-Eicosadienoic Acid Methyl Ester (C20:2) at 2 wt %
27. cis-8,11,14-Eicosatrienoic Acid Methyl Ester (C20:3n6) at 2 wt %
28. Behenic Acid Methyl Ester (C22:0) at 4 wt %
29. cis-11,14,17-Eicosatrienoic Acid Methyl Ester (C20:3n3) at 2 wt %
30. Erucic Acid Methyl Ester (C22:1n9) at 2 wt %
31. Arachidonic Acid Methyl Ester (C20:4n6) at 2 wt %
32. Tricosanoic Acid Methyl Ester (C23:0) at 2 wt %
33. cis-13,16-Docosadienoic Acid Methyl Ester (C22:2) at 2 wt %
34. cis-5,8,11,14,17-Eicosapentaenoic Acid Methyl Ester (C20:5n3) at 2 wt %
35. Lignoceric Acid Methyl Ester (C24:0) at 4 wt %
36. Nervonic Acid Methyl Ester (C24:1) at 2 wt %
37. cis-4,7,10,13,16,19-Docosaheptaenoic Acid Methyl Ester (C22:6n3) at 2 wt %

Ferrioxamine E Scientifically proven supplement for culture media improves detection of *Salmonella*, *Enterobacter sakazakii* and *Yersinia enterocolitica*

By Jvo Siegrist, Product Manager, Fluka / Sigma...isiegris@sial.com



Picture 1
Salmonella detected in a selective enrichment broth (Selenite Broth); left: *Salmonella*, right: control

Ferrioxamine E from *Streptomyces antibioticum* is a siderophore which facilitates the supply of iron (III), an essential trace element, to bacteria involved in food poisoning, including *Salmonella*, *Enterobacter sakazakii* and *Yersinia enterocolitica*. It promotes rapid growth by reducing the lag phase in culture media and reactivates dormant bacteria. Use of ferrioxamine E can permit rapid detection of low bacterial cell counts, even from dry products like spices and tea after long storage periods. It is an essential component of quality control efforts in the food industry.

Ferrioxamine E is often used in Buffered Peptone Water, the medium recommended by the ISO-Norms for *Enterobacteriaceae*. Ferrioxamine E also improves the motility of *Salmonella*, which helps to improve the identification by semisolid selective motility media like SIM, MRSV, DIASSALM or SMS. Ferrioxamine E does

not improve growth of *Escherichia coli*, *Shigella*, *Proteus*, *Providencia* and *Morganella* species; a feature that makes it a semi-selective compound. The recommended concentration of Ferrioxamine E to promote bacterial growth is between 5 and 200 ng/mL.

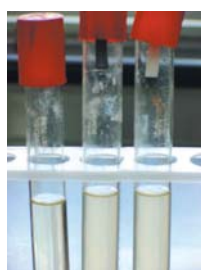
References:

- [1] R. Reissbrodt, H. Heier, H. Tschape, R.A. Kingsley, P.H. Williams, Resuscitation by ferrioxamine E of stressed *Salmonella enterica* serovar typhimurium from soil and water microcosms., *Appl. Environ. Microbiol.*, 66(9), 4128-30 (2000)
- [2] R. Reissbrodt, E. Vielitz, E. Kormann, W. Rabsch, H. Kuhn, Ferrioxamine E-supplemented pre-enrichment and enrichment media improve various isolation methods for *Salmonella*., *Int. J. Food Microbiol.*, 29(1), 81-91(1996)
- [3] P. Pless, R. Reissbrodt, Improvement of *Salmonella* detection on motility enrichment media by ferrioxamine E-supplementation of pre-enrichment culture., *Int. J. Food Microbiol.*, 27, 147-159 (1995)

Part No.	Brand	Description	Package Size
38266	Fluka	Ferrioxamine E (1,12,23-Trihydroxy-1,6,12,17,23,28-hexaazacyclotriactantane-2,5,13,16,24,27-hexone Iron(III) complex) Usage: Supplementation of pre-enrichment broth and enrichment broth media for improved recovery of <i>Salmonella</i> from artificially or naturally contaminated foods	3 mg, 9 mg

Differentiation and Identification of *Salmonella* Tests, agars, broth and supplements from Fluka to identify this potent food-borne pathogen

By Jvo Siegrist, Product Manager, Fluka / Sigma...isiegris@sial.com



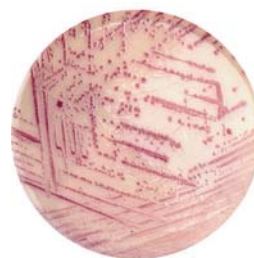
Picture 1
 Hydrogen Sulfide Test Strips (1. Control, 2. *Salmonella* serotype Typhimurim, 3. *Escherichia coli*)

Salmonella contamination is the second leading cause of food-borne illness worldwide. Controlling outbreaks of *Salmonella* is an important task for food regulators, restaurants and the food industry in general. Although *Salmonella* has been known to cause illness for more than 100 years, only recently have definitive tests been available to rapidly identify the culprit bacillus.

The *Salmonella* family includes over 2,300 serotypes of bacteria, but two types, *Salmonella enteritidis* and *Salmonella typhimurium*, are responsible for about half of all human infections. Most outbreaks of *Salmonella* are traced back to dairy, poultry and meat products, but *Salmonella* can grow on nearly any food. Chicken, eggs and their derivative products are particularly high risk.

Microbiologists in the food industry play a critical role in controlling *Salmonella* outbreaks. To allow them to rapidly identify *Salmonella* during an outbreak or to prevent one, Sigma-Aldrich developed growth media and

tests specific to or selective for *Salmonella*. These tests and media take advantage of unique aspects of *Salmonella* physiology or biochemistry relative to other genera within the family *Enterobacteriaceae*. For example, bacteria from the genus *Salmonella* are mostly facultative anaerobes, oxidase-negative, catalase-positive and gram-negative rods. Most strains are motile and ferment glucose with production of both acid and gas.



Picture 2
 HiCrome™ Fluka Agar



Picture 3
Salmonella on SS Agar

Table 1: Test for Detection and Identification of *Salmonella*

Test	Cat. No.	Brand	Testing Features
Aminopeptidase Test	75554	Fluka	detection of L-alanine amino peptidase found almost only in Gram-negative microorganisms.
Catalase Test (Hydrogen peroxide 3%)	88597	Fluka	testing of catalase production
Hydrogen Sulfide Test Strips	6728	Fluka	hydrogen sulfide production
Nitrate Reagent Disks	08086	Fluka	detection of the enzyme nitrate reductase
ONPG Disks	49940	Fluka	β -galactosidase detection
Oxidase Reagent acc. Gaby-Hadley A + Oxidase Reagent acc. Gaby-Hadley B	07345 + 07817	Fluka	oxidase presence
Oxidase Reagent acc. Gordon-McLeod	18502	Fluka	"
Oxidase Strips	40560	Fluka	"
Oxidase Test	70439	Fluka	"

Table 2: Media for Detection and Identification of *Salmonella*

Cat. No.	Brand	Agars for Identification and Differentiation	Cat. No.	Brand	Motility Medium
95388	Fluka	Bismuth sulfite Agar	85438	Fluka	SIM Medium
70133	Fluka	Blood Agar (Base) Supplement: 60 mL/L sterile defibrinated blood			
15835	Fluka	BPL Agar			
70134	Fluka	Brilliant Green Agar, modified			
16026	Fluka	Brilliant Green Phenol Red Lactose Sucrose Agar			
22520	Fluka	China Blue Lactose Agar			
70135	Fluka	DCLS Agar			
51490	Fluka	Hektoen Enteric Agar			
00563	Fluka	HiCrome™ MM Agar			
78419, 73318	Fluka	HiCrome™ <i>Salmonella</i> Agar			
60787	Fluka	Kligler Agar			
61738	Fluka	LDS Agar			
61792	Fluka	Leifson Agar			
62915	Fluka	Lysine Iron Agar			
70143	Fluka	Mac Conkey Agar No 1			
88644	Fluka	Mac Conkey WHO-Agar			
63014	Fluka	MacConkey MUG Agar			
51405	Fluka	MacConkey-Agar (without salt)			
75315	Fluka	OF Test Nutrient Agar			
81648	Fluka	Pril® Mannitol Agar			
84368	Fluka	<i>Salmonella</i> Agar according to Önöz			
84369	Fluka	<i>Salmonella</i> Chromogen Agar Supplement: 1 vial/l <i>Salmonella</i> Chromogen Agar Supplement (Fluka Cat. No. 38589)			
85463	Fluka	Simmons Citrate Agar			
85640	Fluka	SS-Agar			
44940	Fluka	Triple Sugar Iron Agar			
70189	Fluka	Violet Red Bile Glucose Agar			
41270	Fluka	Violet Red Bile Lactose Dextrose Agar			
95273	Fluka	VRB MUG Agar			
95586	Fluka	XLD Agar			
76721	Fluka	XLT4 Agar (Base) Supplement: 4.6 mL/L XLT4 Agar Supplement solution (Fluka Cat. No. 83714)			
			Cat. No.	Brand	Non-selective Enrichment Broths
			08105	Fluka	Buffered Peptone Water (ISO)
			77185	Fluka	Peptone Water
			77187	Fluka	Peptone Water, phosphate-buffered
			Cat. No.	Brand	Selective Enrichment Broths
			50738	Fluka	GN Enrichment Broth
			70144	Fluka	MacConkey Broth
			69965	Fluka	Mossel Broth
			17173	Fluka	Rappaport Vassiliadis Broth, modified
			84370	Fluka	<i>Salmonella</i> Enrichment Broth
			70153	Fluka	Selenite Broth (Base) Supplement: 4 g/L of sodium hydrogen selenite (Fluka Cat. No. 71658)
			84922	Fluka	Selenite Cystine Broth
			86352	Fluka	TBG Broth
			88151	Fluka	Tetrathionate Broth Supplements: 5 g/L iodine (Fluka Cat. No. 57650) 6 g/L potassium iodide (Fluka Cat. No. 60400) 10 mg/L brilliant green (Fluka Cat. No.16020)
			88148	Fluka	Tetrathionate Enrichment Broth acc. to Muller-Kauffmann Supplements: 4 g/L iodine (Fluka Cat. No. 57650) 5 g/L potassium iodide (Fluka Cat. No. 60400) 10 mg/L brilliant green (Fluka Cat. No. 16020)
			Cat. No.	Brand	Broths for Biochemical Confirmation
			69150	Fluka	Methyl Red Voges Proskauer Saline Broth
			39484	Fluka	MR VP Broth
			72548	Fluka	Nitrate Broth

US EPA 324 Vapor Phase Mercury Traps Supelco is the exclusive supplier of innovative and economical FSTM™ trap technology from Frontier GeoSciences

By Mark Robillard, Vice President, Research & Development, Supelco...mrobillard@sial.com
 Bob Cole, Strategic Project Chemist, Supelco...bcole@sial.com
 Bob Brunette, Frontier GeoSciences...bobb@frontiergeosciences.com



Picture 1
 FSTM™ traps

Introduction

US EPA Method 324 (40 CFR Part 75 Appendix K) is designed for short-term and continuous sampling of total mercury emissions in combustion flue gas streams. The method uses a specialized dry sorbent FSTM™ trap developed by Frontier GeoSciences, Inc. with cold vapor atomic fluorescence spectrometry (CVAFS) detection. Through a unique partnership, the combined expertise of Supelco and Frontier GeoSciences has been focused to offer rapid, sensitive, reliable and economical solutions for short-term sampling and continuous monitoring of mercury in combustion flue gas.

Mercury in the environment

The presence of mercury in the biosphere is an area of environmental concern. Although it occurs naturally, anthropogenic sources of mercury vapor in the atmosphere include coal-, oil- and natural gas-fired electric power plants, municipal solid waste incinerators, Hg retort facilities, Zn and Au smelters, chloro-alkali production and industrial boilers. According to the U.S. Environmental Protection Agency (EPA), coal-fired electric power plants are the largest source of human-caused mercury air emissions in the U.S., pumping an estimated 50 – 70 tons of mercury per year into the atmosphere. The U.S. regulations governing mercury emissions, including the Clean Air Mercury Rule (CAMR, 2005) and the Clean Air Interstate Rule (CAIR) issued by the EPA are to reduce mercury emissions from coal-fired power plants. The CAMR reinforces the goal of the EPA's Clean Air Interstate Rule (CAIR) which is to reduce mercury emissions in the US by nearly 70 percent at full implementation.

Frontier GeoSciences and US EPA Method 324 (40 CFR Appendix K) for continuous Hg emissions monitoring

With the promulgation of the CAMR and the drawbacks inherent in existing Hg monitoring approaches, the EPA saw the need for a continuous monitoring method that was reliable, cost effective, accurate and provided more data points. Frontier GeoSciences developed and validated a novel mercury detection system based on a proprietary, chemically impregnated carbon sorbent and with key industry partners, developed a new field sampling method for continuous monitoring of mercury vapor. Because of Supelco's expertise in manufacturing products for air monitoring applications, Frontier GeoSciences collaborated with Supelco to pack their proprietary sorbent in specially designed glass tubes. The

resulting mercury trap was given the trademark acronym FSTM for Flue Gas Sorbent Total Mercury. Besides the innovative sorbent, Frontier GeoSciences leveraged the sensitivity advantages of cold vapor atomic fluorescence spectroscopy (CVAFS) in analyzing the traps post-sampling. The newly legislated CAMR regulations that require continuous emissions monitoring popularized the FSTM technique and it became the underpinnings of the new EPA Method 324 (40 CFR Part 75 Appendix K).

Principles of the FSTM™ method

Known volumes of flue gas are extracted from a duct through the FSTM trap containing the chemically impregnated carbon. The FSTM trap is then acid digested and the resulting digest is analyzed by CVAFS detection as described in EPA Method 1631, Revision E. Mercury concentrations are determined on a mass basis and then combined with flue gas flow data (m³/min) to calculate the continuous mass emission rate of total vapor phase mercury. The analyte measured by this method is total vapor-phase mercury, which represents the sum of elemental and oxidized forms of mercury:

$$\text{Hg (total)} = \text{PHg (particulate)} + \text{Hg(II)} + \text{Hg(0)}$$

The innovations of FSTM technology have simplified two important application areas of mercury monitoring, making them much more practical and economically feasible:

- The initial assessment in order to understand the baseline of mercury behavior and emissions** in a source is critical in order to enable cogent decision making for potential emission control measures. The FSTM traps permit rapid, sensitive and reliable multi-point measurement of total mercury in order to assess the behavior of mercury in existing air pollution control devices and develop the right mercury control strategy.
- The on-going, continuous emission monitoring** role of the FSTM trap in EPA 324 is designed to measure weekly integrated continuous emissions from Hg sources. The FSTM traps permit sampling for as little as 15 minutes and up to 10 days per solid sorbent trap.

FSTM trap features and benefits

The features of the FSTM traps include:

- Each trap uniquely numbered for traceability and chain-of-custody purposes

- High purity, chemically impregnated carbon adsorbent
- Captures all species of gas phase mercury
- Variable flow rate from 0.1 to 4.0 standard liters/min (SLPM)
- Fast total mercury determination (5 to 15 L in 15 to 30 minutes)
- No temperature constraints (ambient to 1000°F).
- Highly standardized Hg blank on trap
- Successfully intercompared to total mercury (THg) in coal-fired flue gas with Ontario Hydro, US EPA M29 and US EPA 101A

In addition, FSTM technology has the following benefits over Ontario Hydro and EPA 101A techniques:

- No hazardous chemicals, no HAZMAT shipping
- World-wide tube availability through Sigma-Aldrich
- Modern, highly-sensitive and fully validated analytical method (EPA 1631 employing CVAFS) gives 50- to 200-fold lower MDL than CVAAS method
- Low Hg background (<1 ng/trap) permits small sample volumes
- Short sample times allows more data points per unit time
- Minimal tube sample train surface area means no Hg wall loss
- No interference from SO₂/NO_x/ash
- Very low overall cost, including materials, labor and analysis
- Excellent field QA capability allows simultaneous field duplicates
- Small equipment package

Trap formats

FSTM traps are available in 2- and 3-bed formats designed for different environments, sampling times, volumes and flow rates. All tube formats can adsorb 5% or greater of their mass in mercury and have a detection limit of 0.005 µg/m³.

Short-term sampling traps allow for the collection of short-term samples with a dynamic range from 15 minutes up to 24 hours.

Continuous emission monitoring traps allows for a continuous emission monitoring approach with a dynamic range from 24 hours up to 10 day integrated samples.

Indoor / ambient air monitoring traps have been approved by the National Environmental Laboratory Accreditation program (NELAP) and the US Department of Health. They are ideal for monitoring personnel exposure to indoor and outdoor mercury vapor using standard active air sampling devices.

Conclusions

FSTM traps developed by Frontier GeoSciences in collaboration with Supelco/Sigma-Aldrich have been validated for use in EPA Method 324 (40 CFR Appendix K) for both short-term sampling and continuous monitoring of total mercury emissions from combustion sources. Using FSTM traps with CVAFS analysis greatly improves the ease-of-use and economy of routine mercury emissions testing. Measurements are accurate and precise, sampling techniques are simple, portable and easily adaptable to the conditions at each source. Compared to competing technologies, the FSTM traps have revolutionized mercury vapor monitoring.

Table 1 FSTM™ traps (Note: Spiked traps will be available in the near future, please inquire)

Short-term sampling traps (15 minutes to 24 hour sampling time)

Cat. No.	Brand	Description	Dimensions	Package Size
2270-U	Supelco	US EPA 324: Short-term Total Hg (2-section trap)	10.5 cm L x 6 mm OD	Box of 25 traps
2274-U	Supelco	US EPA 324: Short-term Total Hg 40 CFR Part 75 Appendix K* (3-Section trap)	13.5 cm L x 6 mm OD	Box of 25 traps

Continuous emission sampling traps (24 hour to 10 day sampling time)

Cat. No.	Brand	Description	Dimensions	Package Size
2272-U	Supelco	US EPA 324: Continuous Emission Monitoring - Total Hg (2-Section Trap)	19.0 cm L x 10 mm OD	Box of 25 traps
2273-U	Supelco	US EPA 324: Continuous Emission Monitoring - Total Hg 40 CFR Part 75 Appendix K* (3-Section trap)	19.0 cm L x 10 mm OD	Box of 25 traps

Indoor and ambient air sampling traps

Cat. No.	Brand	Description	Dimensions	Package Size
2271-U	Supelco	Indoor/Ambient Air Monitoring - Total Hg (2-section trap)	9.0 cm L x 10 mm OD	Box of 25 traps

*Appendix K of 40 CFR Part 75 establishes the procedures for operating a sorbent trap monitoring system to determine mercury mass emissions



Frontier GeoSciences Testing and Consulting services:

Frontier GeoSciences, Inc. is a state of the art analytical laboratory and research facility specializing in ultra low detection of trace metals in multiple matrices. For consultation, sample analysis, field set-up and remediation solutions please contact Bob Brunette at Frontier GeoSciences, 414 Pontius Ave. N., Seattle, WA 98109 U.S. Phone: (206) 957 1461, FAX: (206) 622-6870. www.frontiergeosciences.com

IP-HPLC of Risedronate Enhanced retention of polar pharmaceutical compounds using Fluka high-purity ion pair reagents

By Jacinth A. M. McKenzie, Senior R&D Scientist, Supelco...jmckenzie@sial.com

Risedronate, the active pharmaceutical ingredient in Actonel®, is a bisphosphonate drug used for the treatment and prevention of osteoporosis.* In this report, analysis of risedronate is performed on an Ascentis™ C18 column using a Fluka brand ion pair reagent. When ion pair chromatography is required, it is imperative to use the highest purity reagents available that produce clean baselines and reproducible separations.

Analytical challenge

Supelco's technical service was contacted by a pharmaceutical customer who required a method to analyze risedronate. The requirements were basic: keep the injection to injection time between 5 and 6 minutes, use a standard 15 cm length HPLC column, isocratic flow conditions and UV detection. Upon inspection, it was apparent that the HPLC analysis of risedronate (Figure 1) using silica-based particles under reversed phase conditions would be challenging. First, the polar phosphate and hydroxyl groups in the molecule are not offset by significant hydrophobic regions, conditions not amenable to reversed phase retention. Second, the phosphate and hydroxyl groups are likely chelation sites with metal ions that may be present in the system. Third, the basic pyridine group can interact with silanols on the silica surface. Both silanol interactions and chelation can lead to poor peak shape and low analyte recoveries.

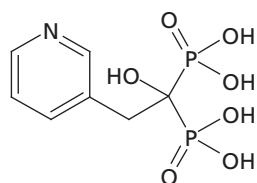


Figure 1 Risedronate structure

RP-HPLC method development strategy

1. Choosing initial mobile phase pH

In order to maximize the retention, the mobile phases for reversed phase HPLC of polar or ionizable compounds should be chosen to minimize the charge on the molecule. Charged molecules are highly solvated and have a greater affinity for the mobile phase over the stationary phase. Acidic compounds are less solvated below their pK_a , while basic compounds are less solvated above their pK_a . Estimations of pK_a for risedronate were performed using ACD Labs software (Toronto, Canada). The results of calculations gave three pK_a values, 0.32

and 0.94 for the acidic phosphate groups, and 5.07 for the nitrogen in the pyridine ring. The overall acidic character led us to start with acidic mobile phases.

2. Scouting pH conditions

At low pH, a gradient from 5 to 75% methanol over 20 minutes, 25 mM potassium phosphate (pH 2.5) at 1 mL/min on a 15 cm x 4.6 mm I.D., 5 μ m Ascentis C18 column did not give adequate retention ($k = 0.30$, Figure 2). In the event that the ionization of the pyridine nitrogen plays a significant role in the solvation character of the molecule, we increased the mobile phase pH to 7 and ran a fast gradient from 5 to 19% methanol over 4 minutes, 25 mM potassium phosphate (pH 7) at 1 mL/min on a 15 cm x 4.6 mm I.D., 5 μ m Ascentis C18 column. Retention was still not adequate ($k = 0.21$, Figure 3).

3. Changing stationary phase selectivity

Within the population of reversed phase columns, C18 is by far the most commonly employed. However, other stationary phases exist that still operate via a reversed phase mechanism, but give different selectivity or enhanced retention compared to a C18. Two such phases are Discovery HS F5 (pentafluorophenyl phase) and Ascentis RP-Amide (embedded polar group phase). The above steps were repeated on 15 cm x 4.6 mm columns of these two phases. Neither column provided significant retention enhancement of risedronate under these conditions.

4. Investigating ion pair chromatography

With silica-based HPLC phases, sometimes it is impossible to operate at a pH where the analyte is adequately retained. For risedronate with the two highly acidic phosphate groups, this condition would require pH values significantly below pH 1 where the stationary phase is readily hydrolyzed, destroying the HPLC column. One solution is to add an ion pair (IP) reagent to the mobile phase. IP reagents have polar groups that interact with functional groups of opposite charge on the analyte, and hydrophobic groups that interact with the reversed phase stationary phase. In this method Fluka brand tetrabutyl ammonium hydroxide (TBAH) was chosen as the IP reagent. TBAH enables the reversed phase retention of risedronate in two ways; the NH_4^+ groups of TBAH interact with the PO_4^- groups of risedronate negating their charge and the butyl groups can interact with the stationary phase.

* Actonel® is marketed by The Alliance for Better Bone Health, an alliance between Procter & Gamble Pharmaceuticals, Inc. and Aventis Pharmaceuticals, Inc., a member of the sanofi-aventis Group.

Although gradient elution is not used with ion pair HPLC, it is useful when scouting for optimum elution conditions. A fast gradient from 5 to 20% methanol over 5 minutes, held at 20% methanol for 10 minutes in 25 mM potassium phosphate containing 5 mM tetrabutyl ammonium hydroxide (pH 7 with phosphoric acid) at 1 mL/min was run on a 15 cm x 4.6 mm I.D., 5 µm Ascentis C18 column. Under these conditions, risedronate retention increased to 11 minutes ($k = 6.02$, **Figure 4**). Isocratic conditions of 25% methanol satisfied the customer's requirement of 5 to 6 minutes per injection (**Figure 5**).

Ion pair chromatography using Fluka brand TBAH and an Ascentis C18 HPLC column provided the customer with the desired results. Analysis was complete within 6 minutes and peak shape for this difficult compound was excellent.

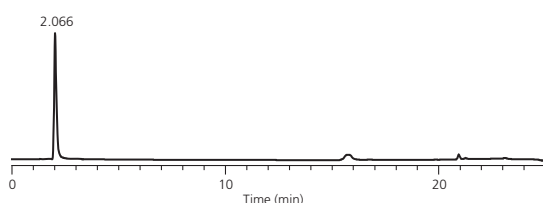


Figure 2 HPLC of risedronate at low pH, no ion pair, C18 column, gradient

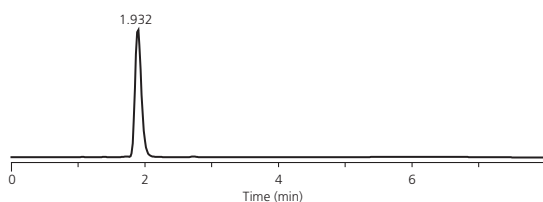


Figure 3 HPLC of risedronate at pH 7, no ion pair, C18 column, gradient

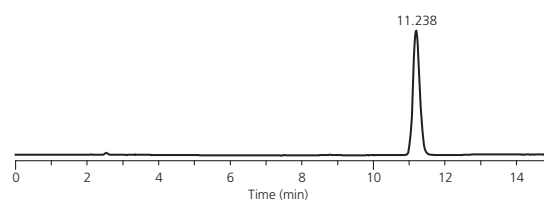


Figure 4 IP-HPLC of risedronate at pH 7, 5 mM TBAH, C18 column, gradient

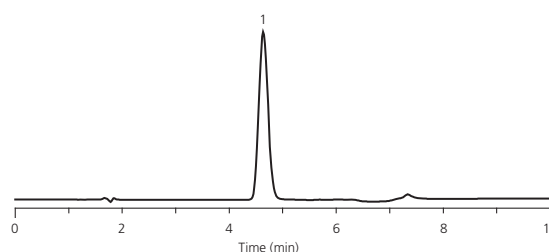


Figure 5 Desired isocratic analysis: IP-HPLC of risedronate at pH 7, 5 mM TBAH, C18 column

Column: Ascentis C18, 15 cm x 4.6 mm I.D., 5 µm particles

Mobile phase: 75:25, 25 mM potassium phosphate dibasic containing 5 mM tetrabutyl ammonium hydroxide (TBAH), pH 7 with phosphoric acid : methanol

Flow rate: 1.0 mL/min

Temp.: 35 °C

Detection: UV at 262 nm

Sample: 20 µL, risedronate (100 µg/mL) in aqueous mobile phase component

Table 1 Products Used for This Application

Cat. No.	Brand	Product Listing	Package Size
581324-U	Supelco	Ascentis™ C18 HPLC column, particle size 5 µm, 15 cm x 4.6 mm I.D.	1 Unit
86854	Fluka	Tetrabutylammonium hydroxide solution, 40% in water (CAS 2052-49-5)	100 mL, 500 mL, 2.5 L
34885	Riedel-de Haën	Methanol G CHROMASOLV® (designed for gradient and sensitive UV applications)	1, 2.5 L
34877	Riedel-de Haën	Water G CHROMASOLV® (designed for gradient and sensitive UV applications)	1 L

Table 2 Selection of High Purity IPC Reagents

Cat. No.	Brand	IPC Reagents (puriss., p.a. grade)	Package Size
52862	Fluka	1-Hexanesulfonic acid sodium salt monohydrate	2.5 g, 10 g, 50 g
51832	Fluka	1-Heptanesulfonic acid sodium salt monohydrate	2.5 g, 10 g, 50 g
74882	Fluka	1-Octanesulfonic acid sodium salt monohydrate	2.5 g, 10 g, 50 g
52864	Fluka	1-Hexanesulfonic acid sodium salt concentrate (~0.33 M)	1 mL Ampoule
51834	Fluka	1-Heptanesulfonic acid sodium salt concentrate (~0.33 M)	1 mL Ampoule
88106	Fluka	Tetrapropylammonium hydrogen sulfate	5 g, 10 g
86847	Fluka	Tetrabutylammonium hydrogen sulfate concentrate (~0.33 M)	1 mL Ampoule
86853	Fluka	Tetrabutylammonium hydrogen sulfate	2.5 g, 10 g, 50 g
87299	Fluka	Tetrahexylammonium hydrogen sulfate	2.5 g, 5 g, 25 g

Importance of Solvent Purity in LC-MS Eliminate noisy baselines and instrument downtime caused by solvent impurities by using LC-MS CHROMASOLV®

By Dave Bell, Applications and Technical Service Manager, Supelco...dbell@sial.com

LC-MS is usually chosen for the sensitivity and specificity it provides. However, this very sensitivity means that care must be taken to avoid contamination from outside sources, like the HPLC mobile phases. Sigma-Aldrich has developed LC-MS CHROMASOLV® solvents and additives to give analysts clean, reliable and reproducible LC-MS separations.

Blends containing the most commonly used additives, like TFA, formic and acetic acid, add convenience to the benefits of the CHROMASOLV® line.

The purity of LC-MS CHROMASOLV® is demonstrated in the accompanying figures. Figures 1a and 1b show the gradient baseline using standard grade HPLC acetonitrile in a clean system. The HPLC column was Ascentis™ C8. Note the presence of polyethylene glycol (m+44) from the solvent in the mass spectrum taken from the total ion chromatogram from 9-11 min.

Figures 2a and 2b show a bleed analysis on the same HPLC column and LC-MS instrument, but with LC-MS CHROMASOLV® acetonitrile. Note the absence of polyethylene glycol and very little baseline rise.

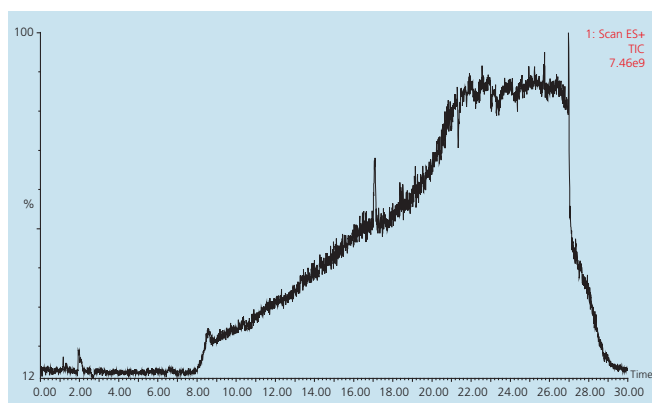


Figure 1a Total ion chromatogram using standard grade acetonitrile in the mobile phase.

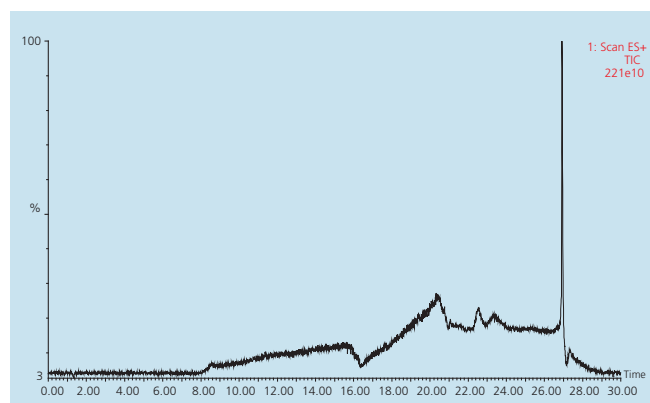


Figure 2a Total ion chromatogram using LC-MS CHROMASOLV® acetonitrile

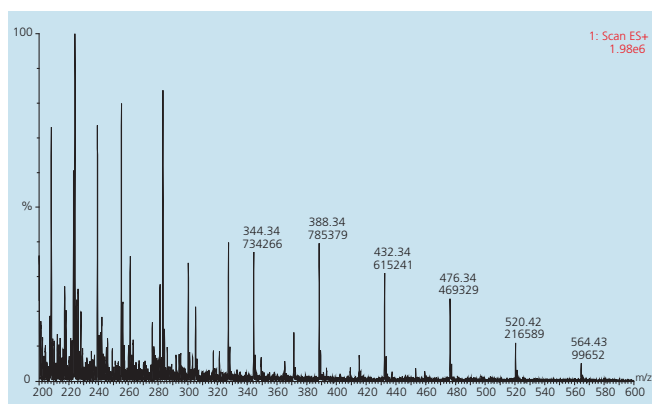


Figure 1b Mass spectrum of time 9-11 min of total ion chromatogram in Figure 1a. Note the presence of polyethylene glycol (m+44).

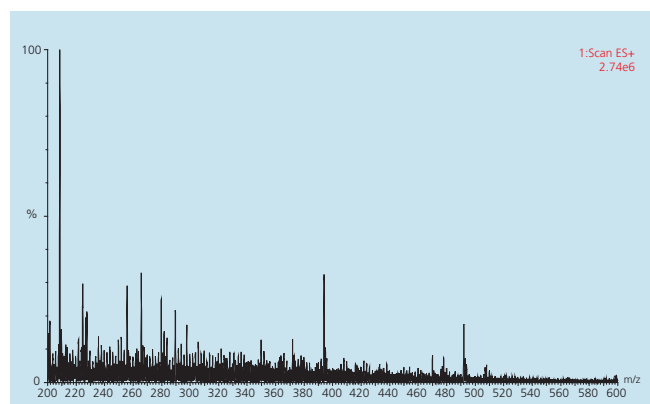


Figure 2b Mass spectrum of time 9-11 min of total ion chromatogram in Figure 2a. Note the absence of PEGs. m/z 224, 255, 371, 469 are bleed peaks, all others found in the control.

Table 1 LC-MS CHROMASOLV® Solvents

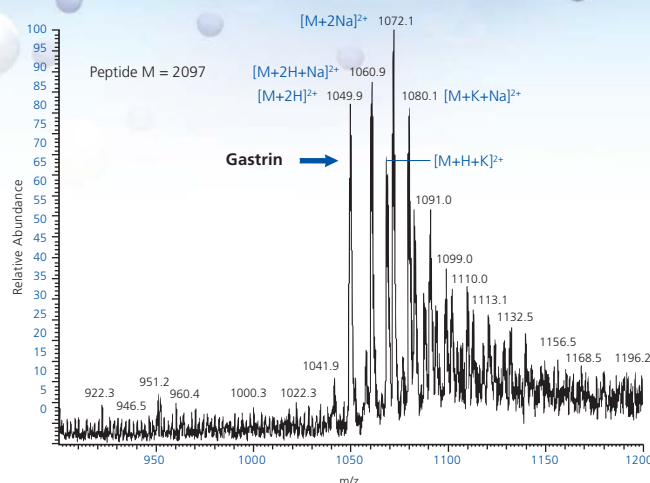
Cat. No.	Brand	Solvent	Package Size	Packaging
39253	Riedel-de Haën	Water LC-MS CHROMASOLV®	1 L	Clear glass bottle
34967	Riedel-de Haën	Acetonitrile LC-MS CHROMASOLV®	1 L, 6 x 1 L, 2.5 L, 4 x 2.5 L	Amber bottle
34966	Riedel-de Haën	Methanol LC-MS CHROMASOLV®	1 L, 6 x 1 L, 2.5 L, 4 x 2.5 L	Amber bottle
34965	Riedel-de Haën	2-Propanol LC-MS CHROMASOLV®	1 L, 6 x 1 L, 2.5 L, 4 x 2.5 L	Amber bottle
34972	Riedel-de Haën	Ethyl acetate LC-MS CHROMASOLV®	1 L, 6 x 1 L, 2.5 L, 4 x 2.5 L	Amber bottle

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.



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

Can you afford any of these problems?

Of course not.

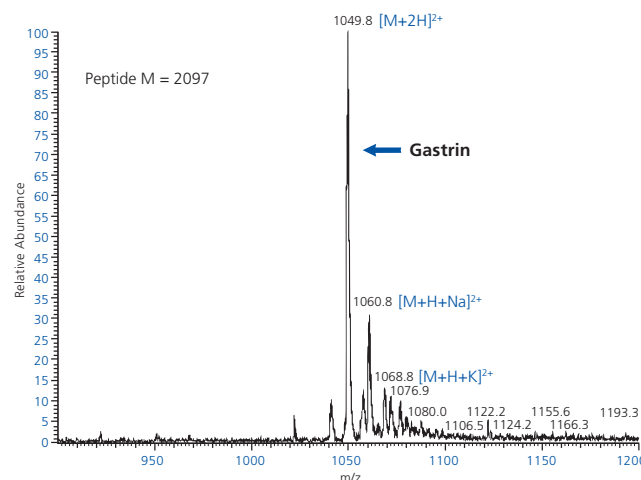
So switch to LC-MS CHROMASOLV® solvents, additives and blends that provide:

- 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.



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

Your LC-MS solvents: CHROMASOLV®

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

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



MALDI Mass Spectrometry Unique, high purity ioniq MALDI matrices from Fluka

By Pierre Nording, Product Manager Fluka / Riedel-de Häen...pnording@europe.sial.com

The MALDI technique involves mixing the sample with a matrix substance and crystallizing the mixture out of a small drop. The crystallized sample/matrix mixture is irradiated by laser light, usually UV. As the matrix absorbs the light energy, it evaporates carrying portions of the sample into the vapor phase, resulting in an indirect ionization of the sample molecules. MALDI is especially useful for protein and peptide identification using masses alone since the masses of ions can be determined with high accuracy.

Although there have been numerous attempts to replace solid chemical matrices by direct MS from solid supports like silicon surfaces or directly from 2-D-gels, most MALDI users still mix the samples within a chemical matrix.

MALDI matrices: Properties and requirements

A typical matrix substance is an aromatic acid with a chromophore that strongly absorbs the laser wavelength. Due to the nature of MALDI, its success mainly depends on the right choice and quality of matrix used. To be useful as a MALDI matrix, the compound must:

- be able to embed and isolate analytes (e.g. by co-crystallization)
- be soluble in solvents compatible with analyte
- be vacuum stable
- absorb the laser wavelength
- cause co-desorption of the analyte upon laser irradiation
- promote analyte ionization

Quality of MALDI matrices

Because MALDI is often used for extremely low-level detection, matrices must first and foremost be free of organic impurities and ions. Organic impurities lead to extraneous peaks, especially in the low mass range. Traces of ions, especially Na⁺ and K⁺, cause adducts of the sample molecules which complicate the MS spectrum, giving it a fence-like appearance; each "post" in the fence is differing in mass according to the mass differences between these various positive ion adducts.

Due to purity requirements, many MALDI users are forced to purify common laboratory grade chemicals. At Sigma-Aldrich's Fluka facility, we realized the advantages of offering highly purified MALDI matrix compounds, tailored to the specific type of sample and analyte. Through the years we have built up a broad range of matrices, which are extensively purified to meet stringent MALDI specifications, and provide sufficient quality even for highest sensitivity requirements.

One problem associated with commonly used matrix substances is that analytes are not fully and uniformly dispersed throughout a solid matrix. The analytes and other components of sample, including impurities, typically segregate and result in inhomogeneous preparations that suffer from poor reproducibility. Inhomogeneity of analyte distribution within the sample spot is a serious problem, especially when MALDI-MS is used for quantitative determinations (1).

Alternatively, liquid matrices can provide homogeneous preparations. However, common liquids suffer from high volatility which results in a variable and uncontrolled matrix. Low mass resolution, poor ionization efficiency and high chemical background have restricted their use in MALDI-MS with UV laser irradiation.

Ioniq matrices reduce hot spots, improve reproducibility and quantification

Ioniq matrix substances are a new class of MALDI matrices that comprise salts of organic acids and organic bases (ionic liquids). Ioniq matrices, especially those with low melting points, provide several distinct advantages. First, they have excellent solubilizing properties compared to other matrices. Second, they allow a homogeneous sample preparation with a thin ionic liquid layer having negligible vapor pressure (2). This leads to facilitated qualitative and quantitative measurements compared to classical matrices. Third, in MALDI, the drop size, stability (surface tension) and uniformity of concentration within the drop are very important. Ioniq matrices yield a very homogeneous drop, so that performance of a single laser shot is much more reproducible and searching for a "hot spot" is less relevant (4).

New ioniq matrices from Sigma-Aldrich

We are pleased to introduce the first products within our ioniq matrix line: α -cyano-4-hydroxycinnamic acid diethyl amine (Figure 1a) and α -cyano-4-hydroxycinnamic acid butyl amine (Figure 1b). In addition to being of very high purity, these compounds crystallize in a more homogeneous manner than the standard HCCA matrix.

Ioniq matrices: Features and Benefits

Compared to other matrices, ioniq matrices provide:

- Improved resolving power
- Reproducibility
- More homogeneous crystallization
- High signal-to-noise ratio
- Improved suitability for quantification

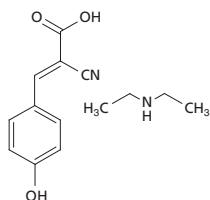


Figure 1a
Ioniq α -cyano-4-hydroxycinnamic acid diethylamine salt (HCCA-diethylamine)

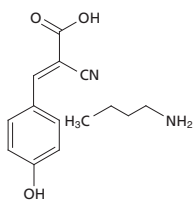


Figure 1b
Ioniq α -cyano-4-hydroxycinnamic acid butylamine salt (HCCA-butylamine)

Ioniq matrices are suitable for qualitative and quantitative analysis of small molecules (3, 4) as well as peptides and proteins, as demonstrated in **Figure 2**. Here, a comparison of MALDI mass spectra using HCCA (**Figure 2a**) and Ioniq HCCA-diethylamine matrix (**Figure 2b**) is shown. The Ioniq matrices show improved resolution (note the $m/\Delta m = 1218$ vs. 314 for Angiotensin II) and higher signal-to-noise ratio ($S/N = 104$ vs. 74 for Angiotensin II). The Ioniq matrix also gave a different peak pattern compared to the standard matrix. This different selectivity may be a benefit when analyzing complex mixtures of peptides.

MALDI-TOF-MS following 2-D gel electrophoresis

We used the Ioniq HCCA-diethylamine matrix to investigate the suitability of new fluorescent protein stains for staining gels prior to MALDI-MS analysis. The sample was 100 μg E. coli extract, separated by 2-D gel electrophoresis (IPG-strip I-2531 pH 3-10, 2-dimension: 4-20% gel) and stained with the new fluorescent Lucy-stains (Lucy 506, Lucy 565, Lucy 569). These stains provide a very fast, robust and economic staining method for SDS- and 2D-gels. Twenty spots were excised and digested with trypsin within the gel slice (PP0100-Kit). Peptides were extracted by standard procedure, placed onto a MALDI target with Ioniq HCCA-diethylamine (dried-droplet) and measured with Bruker OmniFlex instrument. The resulting peaks were used for protein identification by database analysis.

Conclusion

MALDI-MS is an important tool to study proteins and other biomolecules that are not amenable to other types of MS detection. Problems associated with MALDI are often derived from the matrix and include hot spots, irreproducibility, noisy baselines and impurity-derived peaks. By using the new Ioniq MALDI matrix compounds from Sigma-Aldrich, these drawbacks can be avoided.

Table 1 Ioniq matrices from Fluka

Cat. No.	Brand	Name	Package Size
55341	Fluka	α -Cyano-4-hydroxycinnamic acid diethyl amine	100 mg, 1 g
67336	Fluka	α -Cyano-4-hydroxycinnamic acid butyl amine	100 mg, 1 g

Table 2 Fluorescent Stains for Protein Electrophoresis

Cat. No.	Brand	Stain	Package Size
14149	Fluka	Lucy 506	500 μL
41629	Fluka	Lucy 565	500 μL
43772	Fluka	Lucy 569	500 μL

References

- [1] Garden, R.W. and Swedler, J.V., *Anal. Chem.*, 72, 30 (2000).
- [2] Armstrong, D.W., Zhang, L.-K., He, L. and Gross, M. L., *Anal. Chem.*, 73, 3679-3686 (2001).
- [3] Zabet-Moghaddam, M., Heinzle, E., and Tholey, A., *Rapid Communications in Mass Spectrometry*, 18, 141-148 (2004).
- [4] Manko M., Stahl B. and Boehm G., *Anal. Chem.*, 76, 2938-2950 (2004).

Figure 2 MALDI-MS of peptide mix using
a) HCCA, b) Ioniq α -cyano-4-hydroxycinnamic acid diethylamine matrix, measured with Bruker OmniFlex instrument (linear mode). Note the improved resolution and higher signal-to-noise ratio provided by the Ioniq matrix. The analyzed peptide mix contains: bradykinin (1-7), $[M+H]^+ = 757.411$; angiotensin II, $[M+H]^+ = 1046.504$; angiotensin I, $[M+H]^+ = 1296.623$; substance P, $[M+H]^+ = 1347.673$; bombesin, $[M+H]^+ = 1619.741$; renin-substrate, $[M+H]^+ = 1758.832$

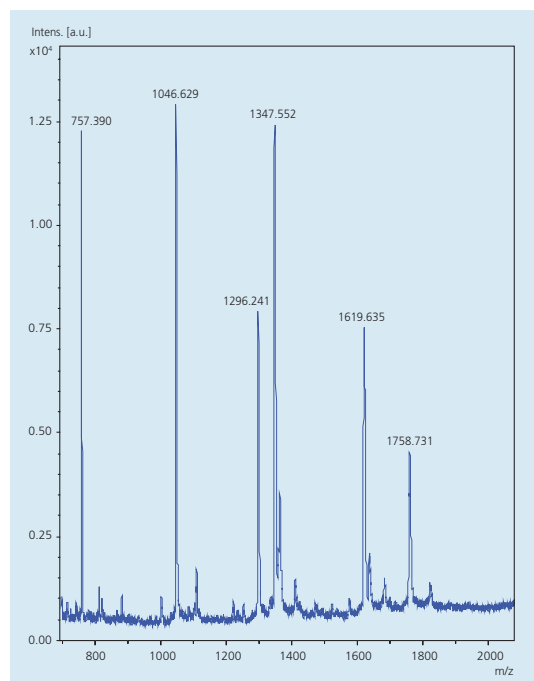


Figure 2a HCCA matrix, Angiotensin II ($m = 1046.5$), $S/N = 74$, Res. ($m / \Delta m$) = 341

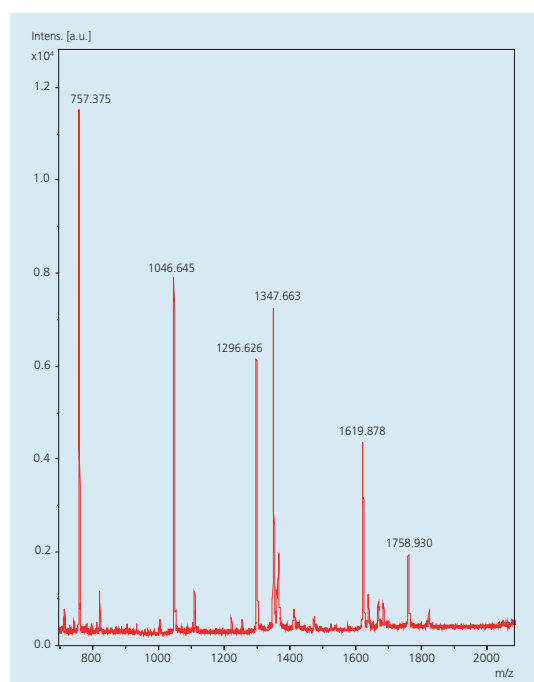


Figure 2b Ioniq HCCA-diethylamine matrix, Angiotensin II ($m = 1046.5$), $S/N = 104$, Res. ($m / \Delta m$) = 1218

New TraceSelect® Products High purity salts with increased purity levels for trace analysis applications

By Frederik Pillong, Product Manager, Fluka / Riedel-de Haën...fpillong@sial.com



... Nine new products
... Higher purity levels

Trace analysis applications require the highest purity sample preparation reagents that contain vanishingly low levels of metal ions and other impurities. Sigma-Aldrich offers Fluka brand TraceSelect® and TraceSelect®Ultra high purity acids, bases and salts designed specifically for trace analysis.

- TraceSelect® acids, bases and salts are intended for sample preparation and analysis in the ppm and ppb level. The blank values for trace metal impurities are typically below 0.01 mg/kg (<0.01 ppm).
- TraceSelect®Ultra acids and bases (salts are currently not yet part of this line) are ideal for ultra trace analysis down to ppb and ppt levels. Typical trace metal impurities are less than 0.1 µg/kg (<0.1 ppb) and in some cases as low as ppq.

Spectroscopy is an important analytical application for TraceSelect® and TraceSelect®Ultra products; as is any research area where high purity is required or is beneficial. When the complexity, solubility or inhomogeneity of the sample prevents total dissolution, as with soil and ore samples, the entire sample can be modified using

high purity salts as the digestion compound. Depending on the digestion compound, the resulting prepared samples have glass-like transparency and homogeneity that allows reliable analysis of metal content by techniques such as Röntgen-Fluorescence analysis (RFA). Or, they can be readily dissolved in acidified water for analysis by AAS or ICP methods. Typical TraceSelect® salts for these applications include lithium carbonate, lithium metaborate, lithium tetraborate, lithium fluoride potassium bisulfate, potassium carbonate and sodium carbonate.

TraceSelect® products and TraceSelect®Ultra water, acids and bases are tailor-made for high sensitivity spectroscopic applications. In this issue of Analytix we are pleased to announce the expansion of our TraceSelect® product line by introducing nine new products (see Table below). Also, we increased the purity of our salts. **All TraceSelect® products are guaranteed 99.99% to 99.9999% pure!**

Please see www.sigma-aldrich.com/traceselect for the complete list of TraceSelect® and TraceSelect®Ultra products.

Table 1 TraceSelect® salts for trace analysis from Fluka

Cat. No.	Brand	Description*	• Improved Purity Level •	Package Size
40581 NEW	Fluka	Cesium bromide	TraceSelect®, ≥ 99.995 % AT	25 g, 100 g
43503 NEW	Fluka	Potassium bromide	TraceSelect®, ≥ 99.999 % AT	25 g, 100 g
60429	Fluka	Potassium nitrate	TraceSelect®, ≥ 99.995 % T	25 g, 100 g
71347	Fluka	Sodium carbonate	TraceSelect®, anhydrous, ≥ 99.999 % T	25 g, 100 g
60347	Fluka	Potassium phosphate dibasic	TraceSelect®, anhydrous, ≥ 99.99 % T	25 g, 100 g
60348	Fluka	Potassium bisulfate	TraceSelect®, ≥ 99.999 % T	25 g, 100 g
71752	Fluka	Sodium nitrate	TraceSelect®, ≥ 99.99 % T	25 g, 100 g
71629	Fluka	Sodium phosphate dibasic	TraceSelect®, anhydrous, ≥ 99.99 % T	25 g, 100 g
09725	Fluka	Ammonium chloride	TraceSelect®, ≥ 99.995 % AT	25 g, 100 g
60371	Fluka	Potassium hydroxide hydrate	TraceSelect®, ≥ 99.995 % T	25 g, 100 g
71492	Fluka	Sodium phosphate monobasic	TraceSelect®, anhydrous, ≥ 99.99 % T	25 g, 100 g
09979	Fluka	Ammonium sulfate	TraceSelect®, ≥ 99.9999 % T	25 g, 100 g
73432 NEW	Fluka	Ammonium acetate	TraceSelect®, ≥ 99.995 % T	100 g
90033 NEW	Fluka	Cesium chloride	TraceSelect®, ≥ 99.999 % AT	25 g, 100 g
01963 NEW	Fluka	Sodium bromide	TraceSelect®, ≥ 99.999 % AT	25 g, 100 g
05257 NEW	Fluka	Potassium chloride	TraceSelect®, ≥ 99.999 % AT	25 g, 100 g
38979 NEW	Fluka	Sodium chloride	TraceSelect®, ≥ 99.99 % AT	25 g, 100 g
59929 NEW	Fluka	Sodium acetate	TraceSelect®, ≥ 99.99 % NT	25 g, 100 g
01968 NEW	Fluka	Sodium hydroxide monohydrate	TraceSelect®, ≥ 99.995 % T	25 g, 100 g

* Analysis by argentometric titration (AT), acidimetric titration (T), non-aqueous titration (NT)

www.sigma-aldrich.com/traceselect

Table 2 TraceSelect® Acids and Bases

Cat. No.	Brand	Description*	Package Size
06454	Fluka	Formic acid, TraceSelect®, ≥ 88.0%	250 mL
08256	Fluka	Hydrochloric acid, TraceSelect®, ≥ 30%	100 mL, 500 mL
09857	Fluka	Ammonium hydroxide solution, TraceSelect®, NH ₃ ≥ 25% water T	100 mL, 500 mL
13171	Fluka	Sodium hydroxide solution, TraceSelect®, ≥ 30% water T	250 mL
45727	Fluka	Acetic acid, TraceSelect®, ≥ 99.0% T	100 mL, 500 mL
47559	Fluka	Hydrofluoric acid, TraceSelect®, 47-51% AT	100 mL, 500 mL
77227	Fluka	Perchloric acid, TraceSelect®, 67-71% T	100 mL, 500 mL
79614	Fluka	Phosphoric acid, TraceSelect®, ≥ 85% T	100 mL, 500 mL
84385	Fluka	Nitric acid, TraceSelect®, ≥ 69.5% T	250 mL, 500 mL, 6 x 500 mL
84415	Fluka	Hydrochloric acid, TraceSelect®, fuming, ≥ 37% T	100 mL, 500 mL
84716	Fluka	Sulfuric acid, TraceSelect®, ≥ 95% T	100 mL, 500 mL
95321	Fluka	Hydrogen peroxide solution, TraceSelect®, ≥ 30% RT	100 mL, 500 mL

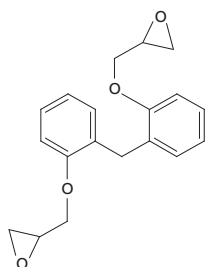
Table 3 TraceSelect®Ultra Acids and Bases

Cat. No.	Brand	Description*	Package Size
02650	Fluka	Nitric acid, TraceSelect®Ultra, ~ 65% T	250 mL
02658	Fluka	Hydrofluoric acid, TraceSelect®Ultra, ≥ 49% acidimetric	250 mL
07692	Fluka	Acetic acid, TraceSelect®Ultra, ≥ 99.0% T	250 mL
14211	Fluka	Water, TraceSelect®Ultra	1 L
16748	Fluka	Ammonium hydroxide solution, TraceSelect®Ultra, ≥ 25% T	250 mL
16911	Fluka	Hydrogen peroxide solution, TraceSelect®Ultra, ≥ 30% RT	250 mL
23828	Fluka	Hydrobromic acid, TraceSelect®Ultra, ≥ 44% T	250 mL
64957	Fluka	Phosphoric acid, TraceSelect®Ultra, ≥ 85% T	250 mL
77239	Fluka	Sulfuric acid, TraceSelect®Ultra, ≥ 95% T	250 mL
96208	Fluka	Hydrochloric acid, TraceSelect®Ultra, ≥ 30% T	250 mL

*Analysis by argentometric titration (AT), acidimetric titration (T), redox titration (RT)

New NOGE (Novolac Glycidyl Ether) Compounds from Fluka Continuing our tradition of offering analytical standards for contemporary food analysis applications

By Rainer Walz...rwalz@europe.sial.com



Epoxy derivatives, including NOGE (novolac glycidyl ether) and its by-products, are current areas of concern in the food packaging and regulatory industry. The main application of NOGE is in the epoxy coatings for cans. Because these compounds come into contact with food products, there is concern that they can migrate out of the packaging and into the food itself, posing a potential human health hazard.

In order to accurately measure residues in food, reliable analytical standards must be available. Sigma-Aldrich's Fluka brand has been a long-time provider of innovative, high quality standards for contemporary analytical applications in the food industry. We continue our tradition by offering three-, four-, five- and six-ring NOGE analytical standards.

For all NOGE standards, ¹H-NMR, ¹³C-NMR and MS-Spectra are available on request.

Table 1 NOGE (Novolac glycidyl ether) analytical standards

Cat. No.	Brand	Description	Package Size
68931	Fluka	3-Ring NOGE (Mixed isomers) Purity: >90%	50 mg
04976	Fluka	4-Ring NOGE (Mixed isomers, aliphatic or branched chains) Purity: >90%	50 mg
12109	Fluka	5-Ring NOGE (Mixed isomers, aliphatic or branched chains) Purity: >90%	50 mg
30977	Fluka	6-Ring NOGE (Mixed isomers, aliphatic or branched chains) Purity: >80%	50 mg

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