

Analytix

Advances in Analytical Chemistry

Issue 3 • 2004

 **Fluka**
Riedel-deHaën®

Introducing

Premixed Solvent Blends for LC-MS

HPLC Derivatization
Reagents

Dabsyl Chloride

Standards

Nitrofurans

Certified Dioxins

Petroleum Hydrocarbon

Certified Polymers

AAS-ICP

Hydrocarbon Index

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New Products Corner



SIGMA-ALDRICH

Introducing New Products and Applications At Sigma-Aldrich, we strive continually to add new and relevant products to help the challenging tasks facing analytical chemists.



Picture Kurt Vorburger, PhD
Manager for Innovation and Product Management

Dear Colleague,

This issue of The Analytix Newsletter presents numerous products and applications for many different fields of analytical chemistry. Today's analyst often faces the dual need to maximize both throughput and sensitivity; one cannot be sacrificed at the expense of the other. At Sigma-Aldrich, we strive continually to add new and relevant products to help the often times challenging tasks facing analytical chemists.

The TraceSelectUltra line of products is a new quality grade with trace impurities specified in the ppt (part per trillion) range. TraceSelectUltra meets the quality requirements of most sophisticated inorganic trace analysis in water, food and environmental samples. The decomposition reagents listed in this Analytix cover a broad range of applications. Look in future issues to see new additions to this product line which we plan to introduce to keep pace with changing market requirements.

Applications are the best way to demonstrate how a product may work in real analytical situations. Besides offering high quality products, our aim has also been to provide useful application information. Such applications on our **HYDRANAL**[®] line of Karl Fisher titration reagents for moisture determination are presented in this issue of The Analytix. They demonstrate the use of **HYDRANAL**[®] reagents and kits for cosmetic and food and beverage industry. Another highlight is the new kit for the determination of Hydrocarbon Index according to DIN EN ISO 9377-1-2-3/2000 (H53).

Ask Sigma-Aldrich for Custom Standards

In addition to our extensive line of chemical standards and reference materials, we also offer our proven capabilities to develop and produce standards according to your specific requirements. This service includes, but is not limited to, standards for chromatographic, spectroscopic and titrimetric applications. Every custom standard and reference material is delivered with a certificate of composition.

What can we do for you?

If you would like to find out whether the combined analytical expertise of scientists in our Fluka, Riedel-de Haën and Supelco brands can help solve your most challenging analytical problem, please contact our Technical Service Team using the contact data on the back page. Our chemists enjoy being challenged by your questions!

The Analytix: always a great source for analytical products and applications. I hope you enjoy reading this issue!

Kurt Vorburger, PhD
Manager for Innovation and
Product Management
Fluka and Riedel-de Haën
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LC-MS solvents and blends Recognizing the growing importance of LC-MS as an analytical tool, Riedel-de Haën is delighted to offer solvents and blends designed to meet the stringent purity standards of LC-MS.

By **Frederik Pillong**
fpillong@sial.com

The power of LC-MS is its ability to provide structural information and detect compounds at extremely low levels. To achieve this, the background noise arising from the mobile phase must be negligible. Minimizing the background and artifacts in LC-MS requires very well specified solvents spiked with ultra pure salts and acids. These additives are used to improve the chromatographic peak shape and to optimize ionization in the MS interface. The most commonly used LC-MS solvents are acetonitrile, methanol and water. Additives can include trifluoroacetic acid (TFA), formic acid, acetic acid and ammonium acetate. Riedel-de Haën now offers ready to use pre-blended solvents specified for LC-MS requirements. Using these precisely blended solvents eliminates time-consuming mobile phase preparation, and can eliminate lost sample information and instrument down-time caused by impure mobile phase.

Specifications:

Acetonitrile and Methanol Blends

LC gradient testing in UV and MS, metal impurities (Na < 2 ppm, K, Mg, Ca < 0.5 ppm), UV-transmittance, additive content: 0.093-0.107 TFA, FA, AA (v/v), ammonium acetate (w/v). Solvent content: (GC): > 99.0 % (Cat. No. 34669 – acetonitrile with 0.1% ammonium acetate; solvent content (GC)>98%)

Specifications: Water blends

LC gradient testing in UV and MS, metal impurities (Na < 2ppm, K, Mg, Ca < 0.5 ppm), UV-transmittance, additive content: 0.093-0.107 TFA, FA, AA (v/v), ammonium acetate (w/v), pH: effective +/- 0.1

Table 1 List of most popular solvent blends

Cat. No.	Brand	Solvent Blend	Pack Size	Packaging
34978	Riedel-de Haën	Water with 0.1% TFA LC-MS CHROMASOLV®	2.5 L	Amber bottle
34976	Riedel-de Haën	Acetonitrile with 0.1% TFA LC-MS CHROMASOLV®	2.5 L	Amber bottle
34974	Riedel-de Haën	Methanol with 0.1% TFA LC-MS CHROMASOLV®	2.5 L	Amber bottle
34673	Riedel-de Haën	Water with 0.1% formic acid LC-MS CHROMASOLV®	2.5 L	Amber bottle
34671	Riedel-de Haën	Water with 0.1% formic acid/0.01% TFA LC-MS CHROMASOLV®	2.5 L	Amber bottle
34668	Riedel-de Haën	Acetonitrile with 0.1% formic acid LC-MS CHROMASOLV®	2.5 L	Amber bottle
34676	Riedel-de Haën	Acetonitrile with 0.1% formic acid/0.01% TFA LC-MS CHROMASOLV®	2.5 L	Amber bottle
34675	Riedel-de Haën	Water with 0.1% acetic acid LC-MS CHROMASOLV®	2.5 L	Amber bottle
34678	Riedel-de Haën	Acetonitrile with 0.1% acetic acid LC-MS CHROMASOLV®	2.5 L	Amber bottle
34672	Riedel-de Haën	Methanol with 0.1% acetic acid LC-MS CHROMASOLV®	2.5 L	Amber bottle
34674	Riedel-de Haën	Water with 0.1% ammonium acetate LC-MS CHROMASOLV®	2.5 L	Amber bottle
34669	Riedel-de Haën	Acetonitrile with 0.1% ammonium acetate LC-MS CHROMASOLV®	2.5 L	Amber bottle
34670	Riedel-de Haën	Methanol with 0.1% ammonium acetate LC-MS CHROMASOLV®	2.5 L	Amber bottle

Note: Other blends available. Please inquire.

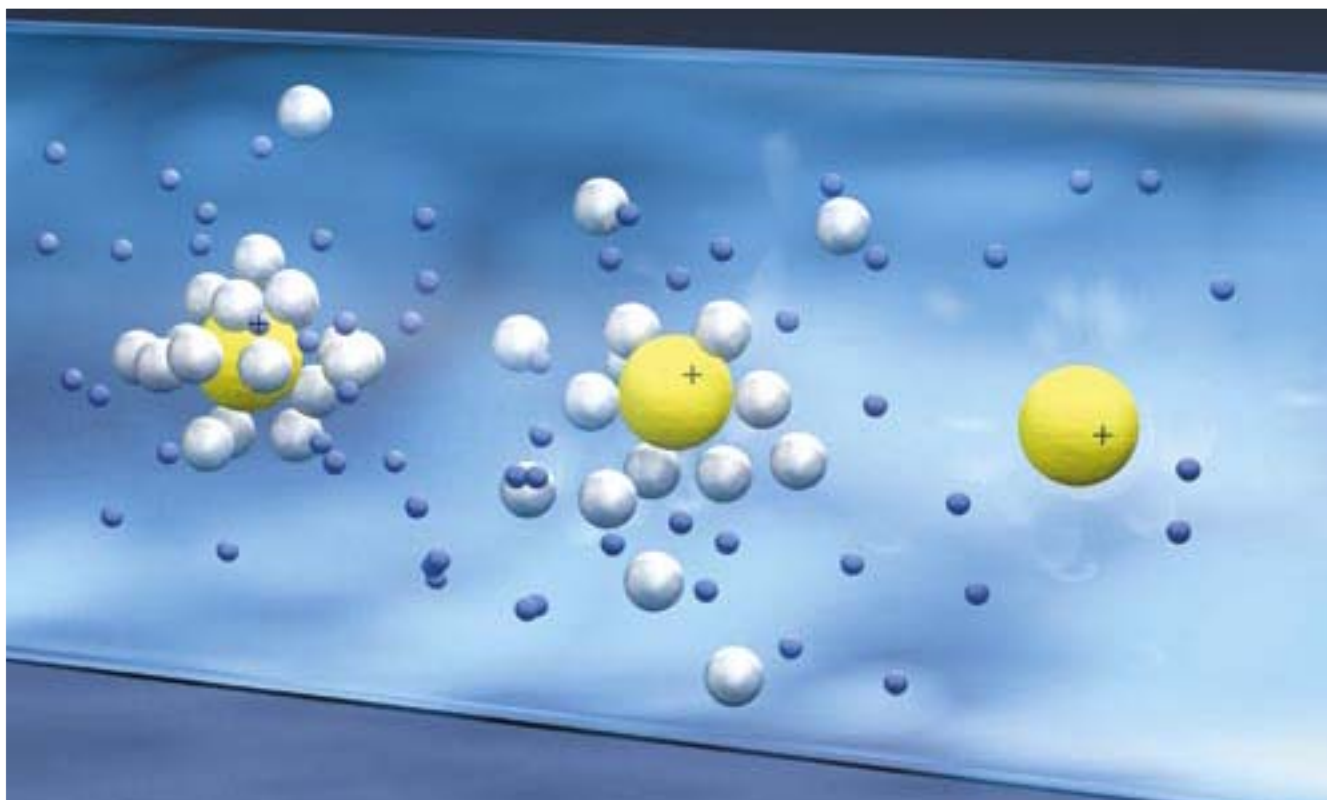
Top tip

After the last run of the day flush the entire LC-MS system, the solvent lines and the column with pure solvents free of salts and acid. This will clean your system and MS interface and protect it from corrosion and blockages. Flushing with pure water and then with a water/2-propanol mix will also stop bacterial growth, keeping your system clean and within specifications.

Looking for reliable, high efficiency, fast LC-MS columns? Please visit www.sigma-aldrich.com/lc-ms-columns to see the new Supelco Discovery rapid LC-MS columns.

Table 2 Ordering Information

Cat. No.	Brand	Rinsing Agent	Pack Size	Packaging
34689	Riedel-de Haën	Water/2-Propanol	1 L	White glass bottle



Picture 1 Charge transfer and ionization process in an electrospray-MS interface



Picture 2 LC-MS bottles PTFE adaptor (Cat. No 79212)

Introductory offer

LC-MS CHROMASOLV[®] solvents are supplied free of dissolved gasses and impurities, both of which can cause erroneous, unreliable LC-MS results and instrument downtime. To avoid the risk of contamination during transfer of the CHROMASOLV[®] solvent, use our new practical dispensing aid (Cat. No. 79212). The dispensing aid comprises a PTFE adaptor screwed directly to the CHROMASOLV[®] bottle and four pins that securely attach the connecting tubing thus eliminating the possibility of impurities entering the lines during dispensing operation.

For a limited time*, get a free dispensing aid with your first order of LC-MS CHROMASOLV[®]-solvents and blends. Reference dispensing Promotion Code 982 when placing your order.

*Offer valid until 31st December; offer limited to only 1 unit per customer.

HPLC Derivatization Reagents: Dabsyl Chloride Dabsyl chloride is a common amine-derivatization reagent for detecting proteins and other biomolecules by HPLC. Dabsyl-derivatives can be detected at visible wavelengths and at picomole concentrations.

By Vicki Yearick.....
vyearick@sial.com

Dabsyl chloride (4-N,N-dimethylaminoazobenzene-4'-sulfonyl chloride) is the reagent of choice for preparing primary and secondary amines, amino acids, thiols, imidazoles, phenols and aliphatic hydroxyl groups for analysis by HPLC. The reagent forms colored derivatives at room temperature that are easily detectable between 420-460 nm. This higher UV range eliminates interferences from most other biological compounds. The intense color permits detection of amines at nanomole levels.

Sigma-Aldrich's dabsyl chloride reagent, produced by our Supelco brand, is tested for both purity and reactivity. This ensures high yield of derivative, and prevents loss of sample due to unwanted side reactions. Complete instructions and a Certificate of Analysis are supplied with each purchase. If you need assistance our knowledgeable staff of Technical Service chemists can help you with reagent selection, derivatization procedures and troubleshooting. Please e-mail us at techservice@sial.com

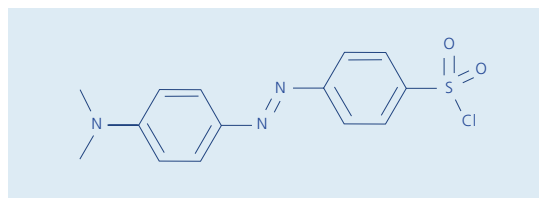


Figure 1 Dabsyl Chloride

Table..... Ordering information

Cat. No.	Brand	Product	Pack Size
502219	Supelco	Dabsyl chloride	500 mg

Special Offer to Analytix Readers!

50% off Dabsyl chloride (500mg) Cat. No. 502219

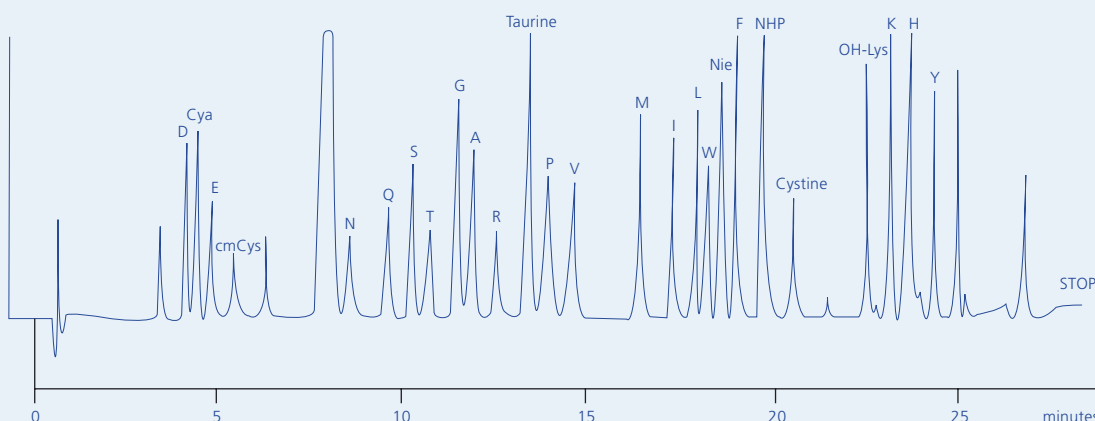
Specify Promotion Code 964 when placing your order to receive your discount. Offer good through December 31, 2004.

Application:

Fast, Simple Separation of Dabsylated Amino Acids

Precolumn derivatization of amino acids with DABS-Cl takes only 10 minutes at 70°C and permits complete reaction of primary and secondary amino acids. Dimethylaminoazobenzene sulfonyl amino acids (DABS-AA) can be detected at visible light wavelengths. This permits HPLC analysis of amino acids at picomole concentrations and eliminates baseline noise that can occur when using UV wavelengths. The HPLC method discussed here takes only 25 minutes for a room temperature separation of approximately 35 DABS-AAs. It was suggested that the best analysis is delivered using a

15 cm x 4.6 mm ID, 3 µm particles SUPELCOSIL LC-DABS column, a 2cm x 4.6 mm ID, 5µm particles Supelguard™ LC-18-T guard column, and a two-eluent mobile phase – consisting of 25 mM potassium dihydrogen phosphate (pH 6.8) as solvent A, and acetonitrile:2-propanol (75:25) as solvent B (Figure 2). This method also resolves DABS derivatives of taurine, D-hydroxylysine, norleucine, cysteine acid, cystine, S-carboxymethylcysteine, and S-sulfocysteine. The separation of DABS-norleucine adds dimension to the method because this derivative can serve as an internal standard. (See Supelco Application Note 124 for more detailed information)



DABS-Cl derivatives are prepared by

1. Add 50 μL of 1.5 M NaHCO_3 (pH 9.0), followed by 100 μL of 2 mg/mL dabsyl chloride in acetone, to each 2. 110 μL aliquot of amino acid standard
3. Vortex the mix
4. Heat to 70°C for 10 minutes
(or hold at room temperature for 30 minutes)
5. Dry under vacuum
6. Resuspend the mix on 200 μL of 70% ethanol and centrifuge for 2 minutes @ 14,000 x g, and transfer to a vial
7. Derivatives are very stable between temperatures of -20°C to room temperature

NOTE: the pH of NaHCO_3 is very important

Figure 2: DABS Amino Acids by HPLC

Column: SUPELCOSIL LC-DABS, 15 cm x 4.6 mm ID, 3 μm particles (Cat. No. 59137)

Sample: 5 μL DABS-derivatized amino acids

Mobile Phase: A = 25 mM potassium dihydrogen phosphate (pH 6.8), B = acetonitrile: 2-propanol, 75:25

Gradient Program (Time: % B): 0-1 min: 20 %; 1-4 min: 20-23 %; 4-9 min: 23 %; 9-10 min: 23-27 %; 10-14 min: 27 %;

14-19 min: 27-35 %; 19-25 min: 35-60 %; 25-26 min: 60-70 %; 26-29 min: 70 %; 29-29.1 min: 70-20 %; 29.1-35.1 min: 20

Flow Rate: 2 mL/min

Det.: 436 nm UV



References

- (1) Stocchi, et al, Journal of Chromatography 349:77-82, 1985.
- (2) Shang, et al, Biochemistry Journal 199:547-555, 1981.
- (3) Supelco Application Note 124

Picture Dabsyl chloride ampule
(Cat. No 502219)

Are you interested in learning about the latest developments in the analytical sciences?

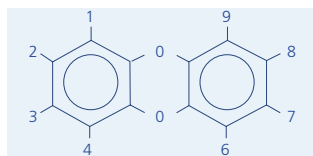
Sigma-Aldrich's SciBookSelect is a selected library of more than 2 000 titles designed to help you keep pace with new technologies and developments in analytical science. Keeping up with the literature is difficult and time-consuming, but very important. Our chemists continually search the scientific literature related to analytical

science and add the most relevant titles to our SciBook-Select offering. Whether your area of interest includes Drug Discovery, Proteomics, Analytical Chemistry, Spectroscopy, Materials Science, Medicinal Chemistry, Chromatography or Spectral Libraries, you can save time searching for the right book or CD in SciBook-Select. View the current listing by visiting our website: www.sigma-aldrich.com/books

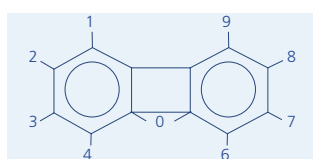
IRMM Certified Dioxin Standards Using Certified Reference Materials is one way for environmental laboratories to ensure they produce high quality and reliable measurements of organic pollutants.

By Rainer Walz.....

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Picture Dibenzo-para-dioxins (PCDDs)



Picture Dibenzofurans (PCDF's)

Polychlorodibenzo-p-dioxins (PCDDs) and polychlorodibenzofurans (PCDFs), formed by the combustion of organic compounds, present grave environmental danger, and are listed as priority pollutants by most international environmental agencies. They also are undesirable by-products from the manufacture or further processing of chlorinated organic compounds. The 2,3,7,8-substituted chlorinated congeners are especially toxic.

Most countries or regional authorities have strict laws controlling the environmental release of these compounds by industry and utilities and have established subsequent public exposure limits. In February 2004, the European Community established guidelines for the determination of PCDDs, PCDFs, and PCBs. Known as draft EN 1948-1-2-3, it presents guidelines for the determination of the mass concentration of PCDDs,

PCDFs, and dioxin-like PCBs. The IRMM (the European Commission's Institute for Reference Materials and Measurements) set up several matrix reference materials to determine PCDDs and PCDFs. Sigma-Aldrich offers high purity, high quality reference standards of these and other environmentally-significant polychlorinated compounds spiked into different representative soil samples. Product names were created to match the corresponding IRMM document.

Benefit from our fast and reliable delivery service and order the IRMM Standard of your choice from Sigma-Aldrich, official distributor of IRMM standards.

CRMs	Matrix	Certified Congeners	Pack Size (approximate quantity)	Packaging
BCR-490	Fly ash	D48, D54, D66, D67, D70, F83, F94, F114, F118, F121, F124, F130	30 g	Amber glass bottles
BCR-615	Fly ash	D48, D54, D66, D67, D70, D73, D75, F83, F94, F114, F118, F121, F124, F130, F131, F134, F135	50 g	Amber glass bottles
BCR-677	Sewage sludge	D48, D54, D67, D70, D73, D75, F83, F94, F114, F118, F121, F124, F130, F131, F134, F135	40 g	Amber glass bottles
BCR-529	Industrial sandy soil	D48, D54, D66, D67, D70, F83, F94, F114, F118, F121, F124, F130	50 g	Amber glass bottles
BCR-530	Industrial clay soil	D67, D70, F94, F114, F118, F121, F130	50 g	Amber glass bottles
BCR-607	Natural spray dried	D48, D54, D66, D67, D70, F83, F94, F114, F118, F121, F124, F130	100 g	Amber glass bottles

Table 1 Certified Reference Materials certified for their content of individual PCDD/F congeners

CRMs	Matrix	Description	Pack Size	Packaging
BCR-614	n-nonane	PCDDs, PCDFs and their ¹³ C-labelled analogues (calibration and spiking solutions)	1 set of 11 ampules	Brown glass ampules sealed under helium gas

Note: The calibration standard set BCR-614 is completed by Matrix-Certified Reference Materials.

Table 2 Certified Reference Materials certified for their content of individual PCDD/F congeners and ¹³C-labelled analogues

CRMs	Matrix	Certified Congeners	Pack Size (approximate quantity)	Packaging
BCR-532	Unspiked milk powder	D48, D54, D66, D67, D70, F83, F94, F114, F118, F121, F124, F130	60 g	Glass bottles
BCR-533	Spiked milk powder	D48, D54, D66, D67, D70, F83, F94, F114, F118, F121, F124, F130 (lower level)	60 g	Glass bottles
BCR-534	Spiked milk powder	D48, D54, D66, D67, D70, F83, F94, F114, F118, F121, F124, F130 (higher level)	60 g	Glass bottles

Table 3 Reference Materials certified for their content of individual PCDD/F congeners

New Products for Nitrofurans Analysis Sigma-Aldrich introduces seven new, derivatized and isotope-labelled standards for nitrofurans analysis.

By Rainer Walz.....

rwalz@sial.com

In 2002 nitrofurans antibiotic residues were found in fish products, including shrimp and catfish, originating in South East Asian countries. Later investigation revealed residues of the same compounds in poultry, duck and rabbit imported from this region. The analysis of nitrofurans drug residue is based on the detection of the tissue-bound metabolites of the nitrofurans parent compound. There are no immunochemical or microbiological screening methods presently available. Most

often the analysis is carried out by LC-MS or LC-MS-MS techniques. Since the discovery of the occurrence of residues of nitrofurans and their metabolites (AOZ (3-amino-2-oxazolidinone), AMOZ (5-methylmorpholino-3-amino-2-oxazolidinone), AHD (1-Aminohydantoin hydrochloride) and SEM (semicarbazide hydrochloride), numerous scientists contacted Sigma-Aldrich asking for analytical standards. Responding to their requests, we introduced the following four standards in 2003:

Cat. No.	Brand	Name	Synonym	Pack Size
33347	Riedel-de Haën	3-Amino-2-oxazolidinone	AOZ	50 mg
33349	Riedel-de Haën	5-Methylmorpholino-3-amino-2-oxazolidinone	AMOZ	50 mg
33870	Riedel-de Haën	1-Aminohydantoin hydrochloride	AHD	10 mg
33656	Riedel-de Haën	Semicarbazide hydrochloride	SEM	100 mg

Table 1 Nitrofurans standards

Soon thereafter, we were asked to provide the derivatized and isotope-labelled standards as well. In response, we developed and introduced seven additional nitrofurans standards. Removing the need for the analyst to

derivatize and label the compounds permits a significant reduction in analysis time. The 2-nitrobenzaldehyde derivative of nitrofurans is the most commonly used standard in nitrofurans analysis.

Cat. No.	Brand	Name	Synonym	Pack Size
33869	Riedel-de Haën	5-(Morpholinomethyl)-3-(2-nitrobenzylidenamino)-2-oxazolidinone	2-NP-AMOZ	10 mg
33868	Riedel-de Haën	3-(2-Nitrobenzylidenamino)-2-oxazolidinone	2-NP-AOZ	10 mg
33871	Riedel-de Haën	(2-Nitrobenzaldehyde semicarbazone	2-NP-SCA	10 mg
33870	Riedel-de Haën	3-(2-Nitrobenzylidenamino)-2,4-imidazolidinedione	2-NP-AHD	10 mg

Table 2 Derivatized and isotope labeled nitrofurans standards

For scientists performing nitrofurans analysis by LC-MS, we introduced three unique isotope marked standards:

Cat. No.	Brand	Name	Synonym	Pack Size
33881	Riedel-de Haën	2-Nitrobenzaldehyde semicarbazone	AMOZ-D5	10 mg
33880	Riedel-de Haën	4,4,5,5-Tetradeutero-3-amino-oxazolidinone	AOZ-D4	10 mg
33882	Riedel-de Haën	Semicarbazide-13C-15N ₂ hydrochloride	SCA-HCl (13C, 15N)	10 mg

Table 3 Isotope labeled nitrofurans standards for LC-MS

References

- (1) Determination tissues of the metabolites of nitrofurans antibiotics in animals tissues by HPLC-tandem mass spectrometry, A. Leitner, P. Zvlner, W. Lindner, J. Chromatogr. A, 939 (2001) 49-58.
- (2) Analysis of protein-bound metabolites of furazolidone and furaltadone in pig liver by high performance liquid chromatography and liquid chromatography-mass spectrometry, E. Horne, A. Cadogan, M. O'Keefe and L.A.P. Hoogenboom, The Analyst, 121 (1996) 1463-1468.
- (3) The use of pig hepatocytes to study the nature of protein-bound metabolites of furazolidone; a new analytical method for their detection, L.A.P. Hoogenboom, M. van Kammen, M.C.J. Berghmans, J.H. Koeman and H.A. Kuiper, Food and Chemical Toxicology, 29 (1991), 321-328.

Petroleum Hydrocarbon Standards for SIMDIS Analyses

Simulated distillation (SIMDIS) by gas chromatography is often used in the petrochemical industry to analyze the composition of an oil or gas sample by measuring the boiling point range distribution of the sample components.

By Vicki Yearick

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Understanding the boiling point range distribution of petrochemical products is critical for selection of the proper method to process feed stocks, for process control, for contaminant identification and for development of new test methods. The technique often employed in the petrochemical industry for the quick determination of the boiling point range is simulated distillation (SIMDIS) by gas chromatography (GC).

SIMDIS methods are analytical methods, developed through consensus and prescribed by the American Society of Testing and Materials International (formerly ASTM) to provide standardization in the marketplace. Each method cites specific GC column phase(s) and dimensions, test condition(s) and guidelines for the composition of the hydrocarbon standards to be used. For example, **Figure 1** shows the separation of ASTM D2887 Reference Gas Oil Sample, Lot 2 test mixture on a Supelco Petrocol 2887 GC column. (At the end of this article is a special offer on Supelco GC columns for petrochemical analysis.)

Through its Supelco brand, Sigma-Aldrich offers individual qualitative and quantitative hydrocarbon standards and reference mixes to meet the needs of analysts using SIMDIS-GC. By using Sigma-Aldrich standards, the time and costs associated with preparing your own standards and mixtures are eliminated. Every Supelco SIMDIS standard and reference mixture is prepared in accordance to ASTM recommendations, and in volume/volume or weight/weight formulations. All calibration references are accompanied by documentation.

For additional information request Bulletin 864 "Simulated Distillation of Petroleum Products by Packed Column and Capillary Column GC" or Bulletin 858 "Petroleum/Chemicals Application Guide", using the enclosed reply card. These bulletins can be also requested by phone, or downloaded from our website:

www.sigma-aldrich.com/supelco-library

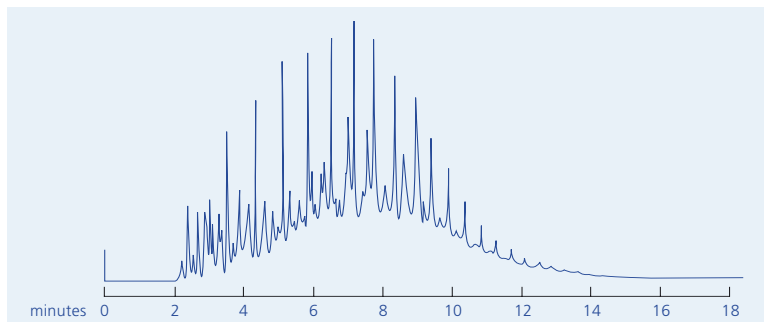


Figure 1: Rapid Analysis of Reference Gas Oil

Column: Petrocol 2887, 5 m x 0.53 mm ID, 5.0 μ m film (Cat. No. 25323)

Col. Temp.: -20°C to 320°C at 20°C/min, hold 5 min

Carrier: nitrogen, 6 mL/min

Det.: FID (32 x 10-10 AFS)

Inj: 0.1 μ L, direct (350°C)

Sample: Reference Gas Oil (Cat. No 48873)

Cat. No.	Description (L x ID, film thickness)
24133-U	Petrocol DH 50.2 (50 m x 0.20 mm, 0.50 μ m)
24155	Petrocol DH 150 (150 m x 0.25 mm, 1 μ m)
24160-U	Petrocol DH 150 (100 m x 0.25 mm, 0.50 μ m)
25323	Petrocol 2887 (5 m x 0.53 mm, 5.0 μ m)
25337	Petrocol Ex2887 (5 m x 0.53 mm, 0.1 μ m)
24282	Petrocol DH Octyl (100 m x 0.25 mm, 0.50 μ m)

Table 1 ASTM D2887 Boiling Range Distribution of Petroleum

Special Offer to Analytix Readers!

35% off Supelco GC columns for petrochemical analysis.

Use Promotion Code 964 when placing your order to receive this offer. Offer good until Dec. 31, 2004

Cat. No.	Brand	Description	Composition (w/w)	Pack Size
48873	Supelco	ASTM D2887 Reference Gas Oil Sample, Lot 2	Petroleum fraction having a boiling range from 250°F to 850°F, evaluated in round-robin studies by the ASTM	pack of 6 ampules of 1 mL each
506419	Supelco	ASTM D2887 Reference Gas Oil Sample, Lot 2	Petroleum fraction having a boiling range from 250°F to 850°F, evaluated in round-robin studies by the ASTM	1 mL ampule
48882	Supelco	ASTM D2887 Quantitative Calibration Mix	n-Decane 11.84 %, n-Dodecane 11.78 %, n-Dotriacontane 0.983 %, n-Eicosane 1.966 %, n-Heptane 5.97 %, n-Hexadecane 9.80 %, Hexane 7.79 %, n-Hexatriacontane 0.983 %, n-nonane 7.84 %, n-Octacosane 0.983 %, n-Octadecane 4.915 %, n-Octane 7.88 %, n-Tetracontane 0.983 %, n-Tetracosane 1.966 %, n-Tetradecane 11.56 %, n-Tetratetracontane 0.983 %, n-Undecane 11.78 %	pack of 6 ampules of 1 mL each

Table 2 ASTM D2887 Boiling Range Distribution of Petroleum

Cat. No.	Brand	Description	Composition (w/w)	Pack Size
48879	Supelco	ASTM D3710 Quantitative Calibration Mix	n-Butylbenzene 3.5 %, n-Decane 3.5 %, 2,4-Dimethylpentane 5.8 %, n-Dodecane 3.5 %, n-Heptane 10.5 %, n-Hexane 5.8 %, 2-Methylbutane 10.5 %, n-Methylpentane 5.8 %, n-Octane 5.8 %, n-Pentadecane 2.3 %, n-Pentane 8.1 %, n-Propylbenzene 4.7 %, n-Tetradecane 2.3 %, Toluene 11.6 %, n-Tridecane 2.3 %, p-Xylene 14.0 %	pack of 6 ampules of 1 mL each
506435	Supelco	ASTM D3710 Quantitative Calibration Mix	n-Butylbenzene 3.5 %, n-Decane 3.5 %, 2,4-Dimethylpentane 5.8 %, n-Dodecane 3.5 %, n-Heptane 10.5 %, n-Hexane 5.8 %, 2-Methylbutane 10.5 %, n-Methylpentane 5.8 %, n-Octane 5.8 %, n-Pentadecane 2.3 %, n-Pentane 8.1 %, n-Propylbenzene 4.7 %, n-Tetradecane 2.3 %, Toluene 11.6 %, n-Tridecane 2.3 %, p-Xylene 14.0 %	1 mL ampule
506427	Supelco	ASTM D3710 Qualitative Calibration Mix	n-Undecane 11.78 %, n-Butane 4.5 %, n-Butylbenzene 3.2 %, n-Decane 3.2 %, 2,4-Dimethylpentane 5.4 %, n-Dodecane 3.2 %, n-Heptane 9.7 %, n-Hexane 5.4 %, 2-Methylbutane 9.7 %, 2-Methylpentane 5.4 %, 2-Methylpropane 1.5 %, n-Octane 5.4 %, n-Pentadecane 2.2 %, n-Pentane 7.6 %, n-Propane 1.5 %, n-Propylbenzene 4.3 %, n-Tetradecane 2.2 %, Toluene 10.8 %, n-Tridecane 2.2 %, p-Xylene 13.0 %	1 mL ampule
48884	Supelco	ASTM D3710 Qualitative Calibration Mix	n-Undecane 11.78 %, n-Butane 4.5 %, n-Butylbenzene 3.2 %, n-Decane 3.2 %, 2,4-Dimethylpentane 5.4 %, n-Dodecane 3.2 %, n-Heptane 9.7 %, n-Hexane 5.4 %, 2-Methylbutane 9.7 %, 2-Methylpentane 5.4 %, 2-Methylpropane 1.5 %, n-Octane 5.4 %, n-Pentadecane 2.2 %, n-Pentane 7.6 %, n-Propane 1.5 %, n-Propylbenzene 4.3 %, n-Tetradecane 2.2 %, Toluene 10.8 %, n-Tridecane 2.2 %, p-Xylene 13.0 %	pack of 6 ampules of 1 mL each

Table 3 ASTM D3710 Boiling Range Distribution of Gasoline

Cat. No.	Brand	Description	Composition (w/w)	Pack Size
48479	Supelco	ASTM D5307 Crude Oil Internal Standard	n-Heptadecane , n-Hexadecane, n-Pentadecane , n-Tetradecane	25 mL ampule
48179	Supelco	ASTM D5307 Crude Oil Quantitative Standard	n-Decane 6.25 %, n-Dodecane 6.25 %, n-Dotriacontane 6.25 %, n-Eicosane, 6.25 % n-Heptadecane 6.25 %, n-Hexadecane 6.25 %, n-Hexatriacontane 6.25 %, n-Octacosane 6.25 %, n-Octadecane 6.25 %, n-Pentadecane 6.25 %, n-Tetracontane 6.25 %, n-tetracosane 6.25 %, n-Tetradecane 6.25 %, n-Tetratetracontane 6.25 %, n-Tridecane 6.25 %, n-Undecane 6.25 %	2 mL ampule
48182	Supelco	ASTM D5307 Crude Oil Qualitative Standard	n-Butane 15 %, n-Heptane 15 %, n-Hexane 15 %, n-Nonane 15 %, n-Octane 15 %, n-Pentane 15 %, n-Propane 10 %	1 mL ampule

Table 4 ASTM D5307 Boiling Range Distribution of Crude Petroleum

Certified Polymer Standards for Gel Permeation Chromatography

Fluka now offers a new dimension of polymer analysis: the first certified polymer standards. Polystyrene (broad and narrow), PEO and polylactic acid standards are available.

By Rainer Walz

rwalz@sial.com

Knowledge of the molecular structure of polymers, including chemical composition, branching, average chain length and chain length distribution, is critical to the manufacture of many commercial products, from plastic bottles to canopies of fighter jets. One analytical difficulty facing polymer scientists is how to obtain polymer standards having a known chain length to calibrate the GPC analysis.

Because of the widespread need for certified polymer standards, and the impact of polymer quality on consumer safety, a government project was initiated. The polymer standards certifying process was coordinated by the German National Institute for Material Science and tested in Berlin by the Federal Institute for Materials Research and Testing (BAM). The certified parameters were carried out by well established round robin experiments done by several independent certified laboratories.

Researchers at Fluka set out to provide polymer analysts with reliable, certified polymer standards that meet the rigorous requirements of the BAM tests. These standards are characterized using a battery of analytical tests, including GPC, GPC with light scattering detection (on-line and off-line) and GPC-viscometry (on-line and off-line). To further characterize our polymer standards, we also use MALDI-TOF, NMR, DSC and rheological testing when appropriate.

The data provided by this extensive Quality Assurance testing appears in the Certificate of Analysis for each lot of Fluka polymer standards. Some critical results presented in the Certificate of Analysis include the average molecular weight (M_w and M_n), polydispersity (D) and other information about the dimension of polymer solutions like intrinsic viscosity (η).

Fluka Certified Polymer Standards can be used for calibration of the following analytical instruments: GPC, light scattering, viscometry, rheology, MALDI-TOF, spectroscopy, surface analysis.

They offer the benefits of:

- Well established and standardized characterization using different, independent analytical methods
- Round robin experiments that guarantee reliable parameters like M_w and M_n
- High accuracy
- Extensive quality documentation

The Certified Polymer Standards are supplied in 100 mg quantities, in glass vessels packed in aluminium bags to protect them from air and moisture. The lot-dependent average molecular weight is printed on the label.

Figure Gel permeation chromatography

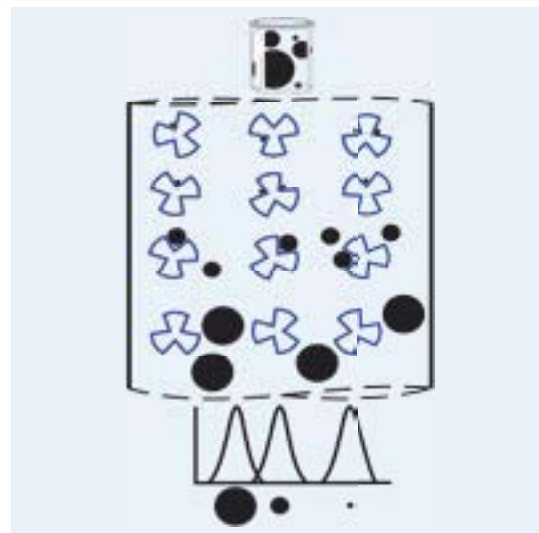


Table Certified Polymer Standards

Cat. No.	Brand	Polymer	M_w in Da	M_w/M_n	Packaging Size
44101	Fluka	Polyethylenoxide	10 160	1.01	100 mg
51876	Fluka	Polystyrene	87 600	1.08	100 mg
53397	Fluka	Polystyrene broad	205 600	2.26	100 mg
53395	Fluka	Polystyrene broad	349 800	2.25	100 mg
50243	Fluka	Polylactic Acid	77 450	1.68	100 mg
78464	Fluka	Polylactic Acid	225 200	1.98	100 mg

Kit for the determination of hydrocarbon oil index according to DIN EN ISO 9377-2/2000 (H53) This kit contains not only the dual layer Florisil®/Na₂SO₄ SPE tube, but also all the reagents and standards employed in “water quality-determination of hydrocarbon oil index” (DIN EN ISO 9377-2/2000).

By Rainer Walz

rwalz@sial.com

ISO 9377 specifies a method for the determination of the hydrocarbon oil index in water by means of gas chromatography. It is suitable for surface water, waste water and water from sewage treatment plants and allows the determination of hydrocarbon oil index in concentrations above 0.1 mg/L.

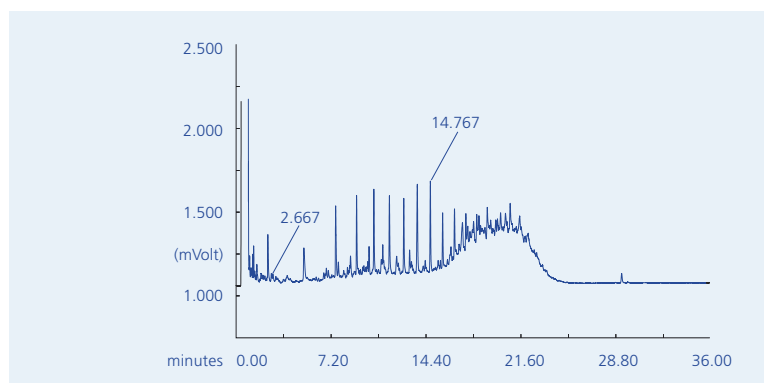
The water sample is extracted with an hydrocarbon solvent. Polar substances are removed by clean up on Florisil®. The purified aliquot is analyzed by capillary chromatography using a non-polar column and a flame ionization detector. The total peak area between n-decane and n-tetracontane is measured. The concentration of mineral oil is quantified against an external standard consisting of two specified mineral oils, and

the hydrocarbon oil index is calculated [Method principle extracted from the European Standard DIN EN ISO 9377-2/2000. To request a copy of “water quality-determination of hydrocarbon oil index” (DIN EN ISO 9377-2 /2000), please contact the DIN institute in Berlin, Germany, www2.din.de].

Inside the kit, you can find the new dual layer glass Florisil®/Na₂SO₄ SPE Tube. These tubes have Na₂SO₄ in the upper layer and Florisil® (magnesium silicate) in the lower layer, separated and packed with PTFE frits. The upper Na₂SO₄ layer aids in removing aqueous sample residues that may hinder Florisil® performance and/or subsequent GC analysis.

Table 1 Kit for determination of hydrocarbon oil index (Cat. No. 68172) content

Cat. No.	Description
67444	Alkane standard mixture C10-C40
52582-U	Dual Layer Florisil®/Na ₂ SO ₄ SPE Tube, 2 g/2 g/6 mL, pk 48
18602	Mineral oil standard mixture Type A and B for DIN EN ISO 9377-2/2000 made of standards from BAM (German Federal Institute for Materials Research and Testing), 10 mg/mL each
51706	Quality control standard solution for DIN EN ISO 9377-2/2000, 10 mg/mL
49574	Extraction solvent stock solution according to DIN EN ISO 9377-2



Mineral oil sample after Florisil® absorption

GC chromatogram done as recommended by DIN EN ISO 9377-2/2000 (H53)
 Column: Supelco HT-5 6m x 0.53 mm ID, fused silica, D_f 0.1 µm (Cat. No. 25004)
 Injection temp.: 40°C to 300°C
 Injection volume: 2 µl
 Carrier gas: helium
 Temp. programm: 40°C/5 min, 10°C/min to 300°C, 300 °C/5 min
 Detector: FID

Table 2 Ordering information

Cat. No.	Brand	Description	Note
68172	Fluka	Kit for the determination of the hydrocarbon oil index acc. to DIN EN ISO 9377-2/2000 (H53)	The kit allows users to extract and analyze 2 series of 10 water samples in duplicate, including 1 blank for each series (total of 48 determinations)

AAS and ICP Calibration Standards High quality ion standards and concentrates to meet the claims of accuracy and reliability for quantitative trace analysis by AAS, ICP-OES and ICP-MS.

By Friederike Jäkle

fjaekle@sial.com



Picture Analytix Notes: AAS and ICP Standards

For detailed information about this product line, use the enclosed Reply Card or visit www.sigma-aldrich.com/aas_icp_standards to get the new AnalytixNotes: ICP and AAS Calibration Standards brochure today.

The AAS and ICP single element standard solutions and concentrates as well as the ICP multi-element standard solutions are produced according to ISO 9001 using highest purity salts and acids or bases. The ready-to-use solutions are traceable to certified reference standard solutions (EMP/BAM or NIST). The calibration of the instruments in our Quality Control Department is carried out using original standards of international institutions such as NIST. All calibration standards are supplied with a Certificate of Analysis stating composition, traceability, release date and expiry date. For the ICP master solutions and concentrates, the levels of known impurities are also determined.

The range of calibration standards includes:

- AAS single element standard solutions with element concentrations of 1000 mg/kg and 10 000 mg/kg
- AAS single element concentrates available with 1 g and 10 g of metal
- ICP master single element standard solutions with element concentrations of 1000 mg/kg
- ICP master single element standard concentrates containing 1 g of metal
- ICP multi-element standard solutions with element combinations and concentrations for common environmental analysis, contaminants in ground and drinking water, for the analysis of sewage sludge and for tuning instruments

Picture AAS and ICP Calibration Standards

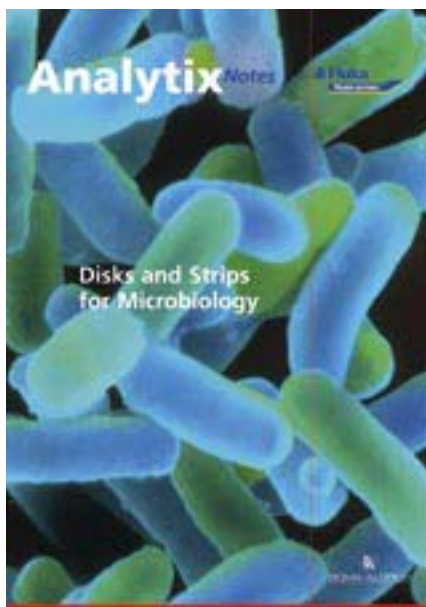


www.sigma-aldrich.com/aas_icp_standards

Disks and Test Strips for Microbiology Disks and strips are an economical, fast and easy-to-use method to identify microorganisms.

By Jvo Siegrist

isiegris@eurnotes.sial.com



Picture Analytix Notes: Disks and Strips for Microbiology (HBN)

Application: Oxidase Test Disks

Few analytical tests have more immediate impact on human health than those that indicate the presence of microbial contamination. The Oxidase Strip Test is an important differential procedure to identify gram-negative bacteria. The cytochrome oxidase present in most gram-negative bacteria triggers the reaction of N,N-dimethyl-p-phenylenediamine with α -naphthol, forming indophenol blue. This test is mainly used to differentiate:

1. Oxidase positive *Neisseria* from other gram-negative *diplococci*
2. Oxidase positive *Aeromonas hydrophila* from *Escherichia coli* (gram-negative)
3. Oxidase positive *Plesiomonas shigelloids* from *Shigella sonnei* (gram-negative)

If oxidase-negative bacteria are absent, no reaction occurs and no color change occurs. However, when these bacteria are present, a dark blue or black spot develops. The Pictures on the right describe the procedure.

www.sigma-aldrich.com/microbiology

Table Ordering information

Cat. No.	Brand	Description	Pack Size
40560	Fluka	Oxidase Strips	100 test strips

Learn about Fluka's Microbiological Discs and Strips in this instructional manual. It includes a description of the methods, theoretical background, test organisms and other important information. Pictures and helpful graphics guide the user toward maximizing their results using the disks and strips.

To obtain a copy of the manual, complete the enclosed reply card or by visiting our website:

www.sigma-aldrich.com/disks_strips_request



Picture 1 Wipe off a suspect colony from a Petri dish using the paper zone of the oxidase strip



Picture 2 Incubate 1 minute



Picture 3 This strip shows the blue color indicating a bacteria with oxidase activity.

New! HYDRANAL® Multimedia Guide Version 2.0 Get a free copy by completing the attached form or visiting www.sigma-aldrich.com/hydranal_cd

Moisture determination is a ubiquitous analytical need, and Karl Fischer titration is one of the most commonly-employed techniques. Sigma-Aldrich introduced the HYDRANAL® product line nearly 25 years ago and since then it has become the industry standard for quality, capacity, speed, safety and reliability in moisture determination. Now you can learn about HYDRANAL® and the theory behind it our new HYDRANAL® Multimedia Guide. In it you'll find a description of the technology, product overview and a database with over 500 moisture analysis applications.

The HYDRANAL® Multimedia Guide contains:

- Theory behind volumetric and coulometric Karl Fischer titration
- Overview of the HYDRANAL® product line and a complete product listing
- Introduction on how to prepare and analyze samples for moisture analysis using HYDRANAL®
- Database of more than 500 HYDRANAL® applications
- Bibliography of HYDRANAL® citations in the scientific literature
- How to request Laboratory Reports, MSDS and other literature, and get technical help from our expert staff

The HYDRANAL® Multimedia Guide version 2.0 is currently available in German, English and Spanish.

To obtain your copy of the HYDRANAL® Multimedia Guide, please complete the Reply Card in this issue of Analytix, or go to our website:
www.sigma-aldrich.com/hydranal_cd



Picture
HYDRANAL® Multimedia Guide (GVJ)

Complexometric titration reagents Sigma-Aldrich offers the most important aminopolycarboxylic acids in the IDRANAL® product line.

By Michael Jeitziner
mjeitziner@sial.com

Reliable complexometric titrations require reagents with controlled, consistent composition and concentration, high purity and activity. The IDRANAL® product line comprises the most commonly used complexation reagents: EDTA, NTA, DTPA, DCTA, and EGTA.

IDRANAL® I (NTA, Nitrilotriacetic acid)

As stability constants of NTA chelates are lower than that of EDTA, NTA is used as a

masking reagent or for industrial purposes. It is a versatile and less expensive chelating agent than EDTA.

IDRANAL® II (EDTA – free acid) and IDRANAL® III (Disodium-EDTA)

EDTA (ethylenediamine tetraacetic acid) is widely used for in complexometric titration. It is also a common stabilizing agent and flavor enhancer in food and cosmetics industries, among many other applications. The free acid of EDTA is a colorless crystalline material which is only slightly soluble in water. However, the sodium salts are quite soluble.

Cat. No.	Brand		Metal						
			Ag (I)	Ba (II)	Ca (II)	Cd (II)	Co (II)	Cr (III)	Cu (II)
34539	Riedel-de Haën	IDRANAL® I: NTA	5.2	4.8	6.1	9.5	10.4	> 10.0	13
34540 and 34549	Riedel-de Haën	IDRANAL® II / III: EDTA	7.3	7.8	11	16.5	16.3	23.4	18.8
34588	Riedel-de Haën	IDRANAL® IV: DCTA	8.2	8.4	12.5	19.2	19		21.3
34589	Riedel-de Haën	IDRANAL® V: DTPA	8.7		10.7	19.3	18.4		21.5
34596	Riedel-de Haën	IDRANAL® VI: EGTA	6.9	8.4	11	16.7	12.5		17.8

Table 1 Formation constant of IDRANAL® complexing agents

www.sigma-aldrich.com/idranal

IDRANAL® A and B (EDTA) for measurement of water hardness

The IDRANAL® A and B solutions are EDTA-Na₂ solutions used for determining water hardness. 1 mL volume of IDRANAL® A corresponds exactly to 5.6° German degrees of hardness (56 mg CaO/L) in 100 mL of water. 1 mL of IDRANAL® B corresponds to 1 German degrees of hardness in 100 mL of water. For all other cations capable of forming chelate complexes (e.g. Ni(II), Fe(III), etc.) the IDRANAL® III-solution is preferred.

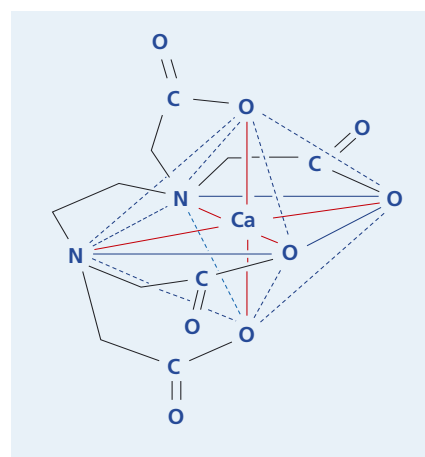
IDRANAL® IV

(DCTA, 1,2-Cyclohexanediaminetetraacetic acid)

CDTA (1,2-cyclohexanediaminetetraacetic acid), the complexometric reagent in IDRANAL® IV, has complexing properties similar to EDTA. However, DCTA complexes have better stability than EDTA complexes, making them advantageous for determination of alkaline earth salts. Aluminum-DCTA complexes are more readily formed, making the otherwise obligatory boiling process in back-titrations unnecessary. The Ni-DCTA complex is so stable that it is virtually inert to cyanide reaction and it is therefore possible to separate Ni(II) from Zn(II), Cd(II) and Cu(II) ions using this reagent.

IDRANAL® V (DTPA, Diethylenetriamine pentaacetic acid)

IDRANAL® V (DTPA) has special applications. It facilitates the determination of thorium as well as rare earth elements. It also provides a reliable titration of barium and strontium. DTPA is also used in electrochemical applications as a strong complexing agent for the Hg(II), Cr(III) and Cr(IV) ions.



Picture Nitrilotriacetic acid

IDRANAL® VI (EGTA, Ethyleneglycol-O,O'-bis (2-aminoethyl)-N,N,N',N'-tetraacetic acid)

IDRANAL® VI is ideal for the determination of Ca and Mg ions as their stability constants are widely different. It is also used for DNA synthesis and microbiological applications.



Picture VOLPAC® packages

IDRANAL® solutions

VOLPAC® Package

IDRANAL® complexometric solutions are available in convenient 10 L containers. These ready-to-use solutions are ideal for online and automated titration. The VOLPAC® container consists of flexible polyethylene bag with an outlet tap housed within a strong cardboard cube and can be easily adapted to most commercially available titration instruments. The 5 L pack will be available soon.

Cat. No.	Brand	Product	Pack size
34544	Riedel-de Haën	IDRANAL® B	10 L
34550	Riedel-de Haën	IDRANAL® III 0.1 mol/L	10 L
35102	Riedel-de Haën	IDRANAL® III 0.2 mol/L	10 L
35103	Riedel-de Haën	IDRANAL® IV 0.1 mol/L	10 L

Table 2 IDRANAL® VOLPAC® Packages

Fe (II)	Fe (III)	Hg (II)	La (III)	Li (I)	Mg (II)	Mn (II)	Na (I)	Ni (II)	Pb (II)	Sn (II)	Sr (II)	Tl (III)	Zn (II)
8.8	15.9	14.6	10.5	2.2	5.5	7.4	2.2	11.5	11.4		5	18	10.7
14.3	25.1	21.8	15.5	2.8	8.7	14	1.7	18.6	18	18.3	8.6	22.5	16.5
16.3	28.1	24.3	16.9	4.1	10.3	16.8	2.7	19.4	19.7		10.5	38.3	18.7
16.5	28.6	27	19.6		9.3	15.5		20.3	18.8			48	18.8
11.9	20.5	23.1	15.8	1.8	5.2	12.3	1.4	13.6	14.7	23.9	8.5		14.5

Sample Preparation for Trace Analysis: TraceSelectUltra and TraceSelect

Ultra-pure acids, bases and salts for smelting and wet digestion in environmental, water and food analysis.

By Friederike Jäkle

fjaekle@sial.com



TraceSelectUltra acids and bases are intended for the most demanding trace analysis requirements. All metals are supplied in the ppt (< 10 ng/kg) trace range. TraceSelectUltra acids and bases are purified by sub-boiling distillation and packed in specially manufactured and cleaned Teflon PFA (fluoropolymer) bottles. Water and ortho-phosphoric acid are supplied in specially pre-leached HDPE bottles. The TraceSelectUltra product series was recently expanded by eight new products.

The acids, bases and salts in the TraceSelect series were developed for sample preparation and analysis in the ppb ($\mu\text{g}/\text{kg}$) trace range. Purity and composition are guaranteed by 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 TraceSelectUltra and TraceSelect products. They are produced and filled under clean room conditions and delivered with a Certificate of Analysis.

Table 1 TraceSelectUltra reagents

Cat. No.	Brand	Product Name	Specification
02658	Fluka	Hydrofluoric acid	TraceSelectUltra for trace analysis ≥ 49 % (acidimetric)
02650	Fluka	Nitric acid	TraceSelectUltra for trace analysis ~ 65 % (T)
07692	Fluka	Acetic acid	TraceSelectUltra for trace analysis ≥ 99.0 % (T)
14211	Fluka	Water	TraceSelectUltra for trace analysis
64957	Fluka	Phosphoric acid	TraceSelectUltra for trace analysis ≥ 85 % (T)
77239	Fluka	Sulfuric acid	TraceSelectUltra ≥ 95 % (T)
96208	Fluka	Hydrochloric acid	TraceSelectUltra for trace analysis ≥ 30 % (T)
23828	Fluka	Hydrobromic acid	TraceSelectUltra for trace analysis ≥ 44 % (T)
16748	Fluka	Ammonium hydroxide solution	TraceSelectUltra for trace analysis ≥ 25 % (T)
16911	Fluka	Hydrogen peroxide solution	TraceSelectUltra ≥ 30 % (RT) for trace analysis

Note: T: Titration; RT: Redox titration.

Table 2 TraceSelect reagents

Cat. No.	Brand	Product Name	Specification
45727	Fluka	Acetic acid	TraceSelect for trace analysis ≥ 99.0 % (T)
09857	Fluka	Ammonium hydroxide solution	TraceSelect for trace analysis ≥ 25 % NH_3 in water (T)
84415	Fluka	Hydrochloric acid	TraceSelect for trace analysis fuming 37 % (T)
47559	Fluka	Hydrofluoric acid	TraceSelect for trace analysis 47-51 % (acidimetric)
95321	Fluka	Hydrogen peroxide solution	TraceSelect ≥ 30 % (RT)
84385	Fluka	Nitric acid	TraceSelect for trace analysis ≥ 69.5 % (T)
79614	Fluka	Phosphoric acid	TraceSelect for trace analysis ≥ 85 % (T)
77227	Fluka	Perchloric acid	TraceSelect for trace analysis 67-71 %
84716	Fluka	Sulfuric acid	TraceSelect for trace analysis ≥ 95 % (T)
09725	Fluka	Hydrofluoric acid	TraceSelect for trace analysis 47-51 % (acidimetric)
09979	Fluka	Ammonium sulfate	TraceSelect ≥ 99.5 % (T)
60347	Fluka	Potassium phosphate dibasic	TraceSelect anhydrous ≥ 99.5 % (T)
71629	Fluka	Hydrofluoric acid	TraceSelect for trace analysis 47-51 % (acidimetric)
60348	Fluka	Potassium bisulfate	TraceSelect ≥ 99.0 % (T)
60371	Fluka	Potassium hydroxide hydrate	TraceSelect ≥ 65 % (T)
60429	Fluka	Potassium nitrate	TraceSelect ≥ 99.5 % (T)
71347	Fluka	Sodium carbonate (soda ash)	TraceSelect anhydrous ≥ 99.5 % (T)
71492	Fluka	Sodium phosphate monobasic (sodium dihydrogen phosphate, monosodium phosphate)	TraceSelect anhydrous ≥ 99.0 % (T)
71752	Fluka	Sodium nitrate	TraceSelect ≥ 99.0 % (T)
13171	Fluka	Sodium hydroxide solution	TraceSelect ≥ 30 % in water (T)

Note: T: Titration; RT: Redox titration.

HYDRANAL® Application: Cosmetic Industry

Cosmetic products often contain a very high water content that must be exactly determined for the product to have acceptable texture and shelf-life, and for it to be able to form stable emulsions.

By Michael Jeitziner

mjeitziner@sial.com

Introduction

Accurate moisture determination requires precise sampling, weighing and testing of the product. Typically, the cosmetic samples are acquired using a syringe which is weighed before and after the sample is delivered into the testing vessel (differential weighing). For these types of samples, volumetric Karl Fischer determination is preferred over coulometry since the latter requires very small sample amounts. Oil-in-water emulsions, which are common among cosmetic products, often require the addition of chloroform and even elevated temperatures to ensure complete solubilization of any waxy substances as well as release and homogenous distribution of trapped water.

Hand Balm (Laboratory Report L492*)

Most hand creams are formulations containing water and oil stabilized with an emulsifier. Reliable determination of moisture in emulsions is difficult under standard working conditions because the water is not evenly distributed within the emulsion. An additional complication is that undissolved sample components, like waxes and oils, can collect around the indicator electrode and interfere with the measurement. Successful moisture determination of emulsions can be achieved by heating the sample in the titration vessel to distribute the sample more evenly, and by adding formamide to prevent the deposition of waxes and oils on the indicator electrode.

Moisture level in a sample of hand cream is measured by filling the titration vessel with 30 mL HYDRANAL® Methanol (e.g. HYDRANAL® Methanol Rapid or HYDRANAL®-CompoSolver E) and 10 mL HYDRANAL® Formamide Dry, heating it to 50°C, and titrating it to dryness with HYDRANAL® Composite 5. A 40 mg sample is then weighed using the differential weighing procedure, and the water content is titrated with HYDRANAL® Composite 5. Complete instructions and results can be found in the Laboratory Report L492*.

*Please contact our HYDRANAL® Laboratories (h.hoffman@europe.sial.com). We will be glad to send you our Laboratory Reports by fax or e-mail. You can also find the full list on our website www.sigma-aldrich.com/hydranal

Cat. No.	Brand	Product	Used as....
34805	Riedel-de Haën	HYDRANAL®-Composite 5	Titrant (one component-reagent)
37817	Riedel-de Haën	HYDRANAL®-Methanol Rapid	Solvent for fast titration
34741	Riedel-de Haën	HYDRANAL®-Methanol dry	Solvent
34724	Riedel-de Haën	HYDRANAL®-Formamide dry	Solubilizing agent

Table HYDRANAL®-Reagents used for this application



Figure 1 Moisture levels of some cosmetic products determined using HYDRANAL®-reagents. (Note: * Laboratory Reports)

HYDRANAL® Application: Food Industry Accurate determination of the moisture content of fresh and processed foods such as meat, fish and dairy products is important from both quality and commercial value standpoints.

By Michael Jeitziner
mjeitziner@sial.com

Introduction

Moisture plays a critical role in the quality and shelf-life of many fresh and processed food products. HYDRANAL® reagents are ideally suited for this application. When performing moisture analysis on food samples, it is important that no chemical side reactions occur during sample preparation that can actually generate water. Also, interference from other substances, such as mercaptans and aldehydes, is typically small because of their low levels compared to the water content of the food product. Another complication is that the water content is often not distributed evenly throughout most food samples. The sample preparation techniques must ensure a homogenous suspension.

Water Determination in White Pepper (Laboratory Report L 433*)

Pepper provides an example of a moisture analysis application of a food substance. Classical Karl Fischer titration methods prove difficult; pepper does not dissolve or disperse evenly in the alcoholic medium of the Karl Fischer titration in the titration vessel. The water from the pepper sample is released only very slowly in a volumetric titration. Even adding formamide or increasing the temperature to 50°C does not significantly improve the extraction rate. External extraction, where the extraction of the sample occurs in an Erlenmeyer flask and not directly in the titration vessel, is required. In this procedure, after a certain extraction time an aliquot is taken using a syringe and injected into the titration vessel. The advantages of external extraction are twofold: the titration vessel is not contaminated with the insoluble material, and the extraction and titration solvents can be different. External extraction was carried out on the pepper sample. Experiments showed a water content of 13.6 % after one hour, 14.0 % after 2 hours, 14.3 % after 3 hours and 14.4 % after 24 hours extraction. Extraction was consistent, with at least three hours recommended extraction time.

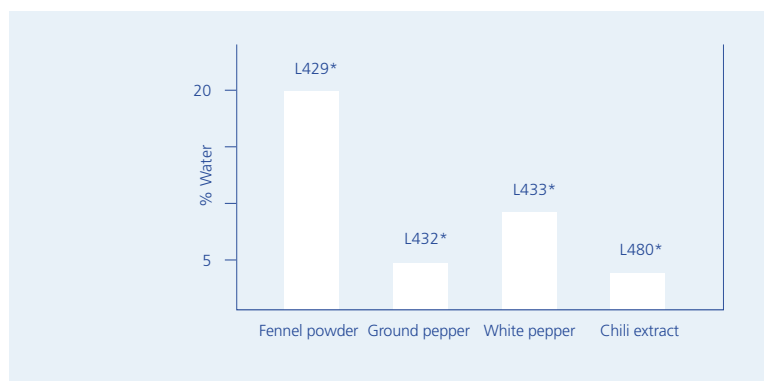


Figure Moisture levels of spices samples determined using HYDRANAL®-Reagents.
(Note: * Laboratory Reports)



Picture 1 Ground pepper

The procedure followed for the pepper samples is as follows:

- Weigh approximately 2 g of white pepper into an Erlenmeyer or other stoppered flask.
- Add 100 mL HYDRANAL®-Methanol Dry via pipette.
- Determine the total weight of sample plus reagent.
- Add 30 mL HYDRANAL®-Methanol Dry or 30 mL HYDRANAL®-CompoSolver E to the titration vessel.
- Titrate to dryness using HYDRANAL®-Composite 5.
- After an extraction time of at least 5 hours, remove 10 mL of the extract solution using a syringe and inject it into the titration vessel. Determine the exact weight of the sample by back-weighing.
- The water content of the extracted sample is titrated using HYDRANAL®-Composite 5.

A blank value should be analyzed by treating HYDRANAL®-Methanol dry in exactly the same way as the sample, being certain to use a dry pipette and syringe. The two-component reagent HYDRANAL®-Titrant 5/ Solvent or HYDRANAL®-Titrant 5E HYDRANAL®-Solvent E can also be used.

*Please contact our HYDRANAL® Laboratories (h hoffman@europe.sial.com). We will be glad to send you our Laboratory Reports by fax or e-mail. You can also find the full list on our website www.sigma-aldrich.com/hydranal

Cat. No.	Description	Product	Used as....
34741	Riedel-de Haën	HYDRANAL®-Methanol-dry	Solvent (one-component reagent for volumetry)
34734	Riedel-de Haën	HYDRANAL®-CompoSolver E	Solvent (one-component reagent for volumetry)
34805	Riedel-de Haën	HYDRANAL®-Composite 5	Titrant (one-component reagent for volumetry)
34801	Riedel-de Haën	HYDRANAL®-Titrant 5	Titrant (two-component reagent for volumetry)
34800	Riedel-de Haën	HYDRANAL®-Solvent	Solvent (two-component reagent for volumetry)
34732	Riedel-de Haën	HYDRANAL®-Titrant 5E	Titrant (two-component reagent for volumetry)
34730	Riedel-de Haën	HYDRANAL®-Solvent E	Solvent (two-component reagent for volumetry)

Table HYDRANAL®-Reagents used for this application

Upcoming Events HYDRANAL® Seminars

When	Where
October 1 st	Malaysia
October 4 th	Bangkok, Thailand
October 6 th – 8 th	Taiwan
October 11 th – 14 th	Korea
October 20 th	Egerkingen, Switzerland
November 3 rd – 4 th	Seelze, Germany
November 17 th	Paris, France

For registration and additional information, please contact

Ms. Helga Hoffmann

E-mail: h hoffman@europe.sial.com

Survey on The Analytix

We're seeking your opinion on the new Analytix design!

The Analytix newsletter is designed for you, to meet your needs in current analytical chemistry and related techniques. In 2004, we launched a new version of the Analytix with completely new layout and enhanced content.

We are now soliciting your input on this new design. By completing a short, on-line survey you will help us determine the impact of the new design, understand your needs and see how we can improve quality of



our service. As a token of our thanks, we will send you a free Compass Key Fob. To complete the on-line survey and receive your gift, please visit:

www.sigma-aldrich.com/analytix_survey

New Product Corner

Unique fatty acid tryptamides derived from cocoa shells.



Fatty acid tryptamides (FAT) are shell indicators in cocoa products and are used to determine the quality of chocolate. High quality chocolate should be devoid of any residual shell materials. Chemists at Sigma-Aldrich's Fluka facility have developed an innovative method to extract five unique FAT compounds from cocoa shells and provide them in pure form.

Picture Chocolate

Table FAT compounds

Cat. No.	Brand	Product Name	Acronym	Package Sizes
12094	Fluka	Behenic acid tryptamide	BAT	25 mg, 100 mg
00864	Fluka	Cerotic acid tryptamide	CAT	25 mg, 100 mg
7347	Fluka	Lignoceric acid tryptamide	LAT	25 mg, 100 mg
41905	Fluka	Heptadecanoic acid tryptamide	MAT	25 mg, 100 mg
56924	Fluka	Tricosanic acid tryptamide	TAT	25 mg, 100 mg

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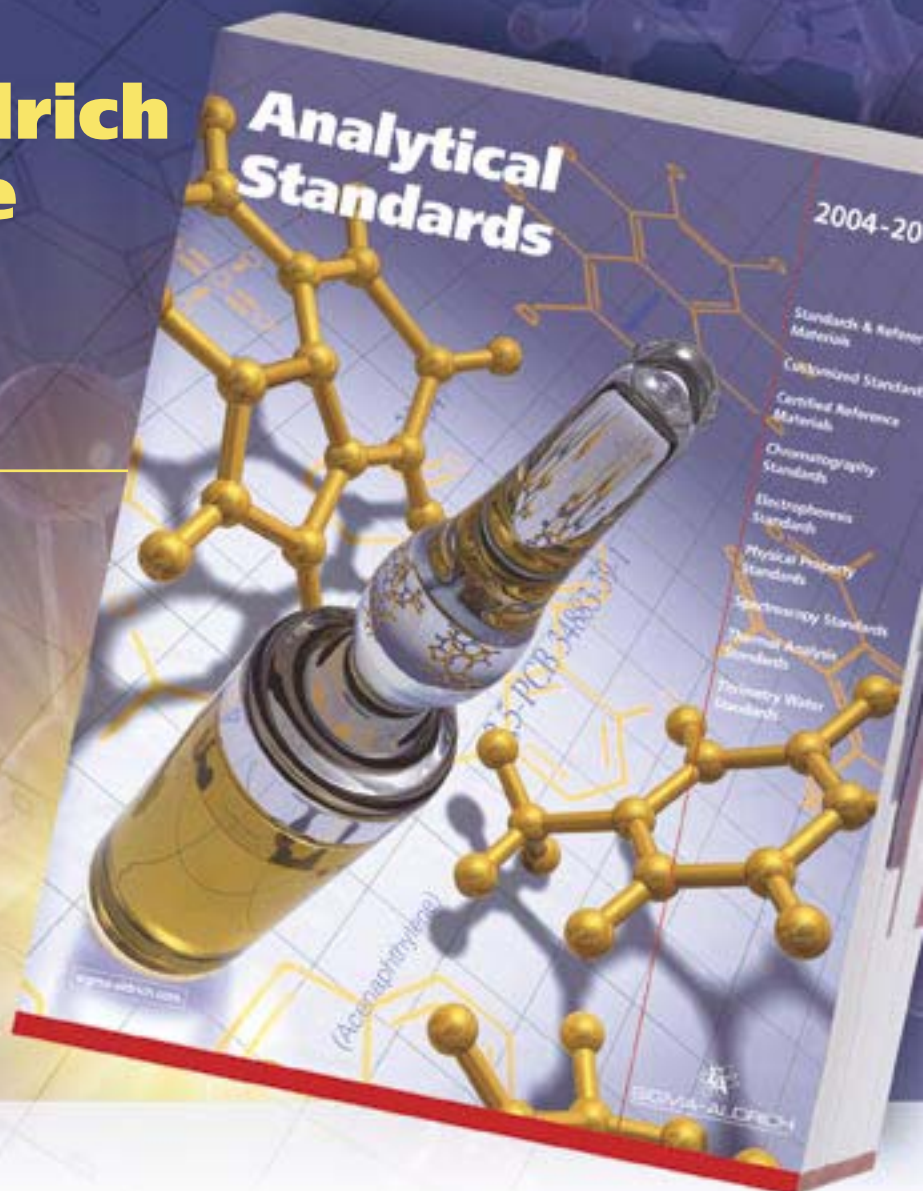
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