Analytix

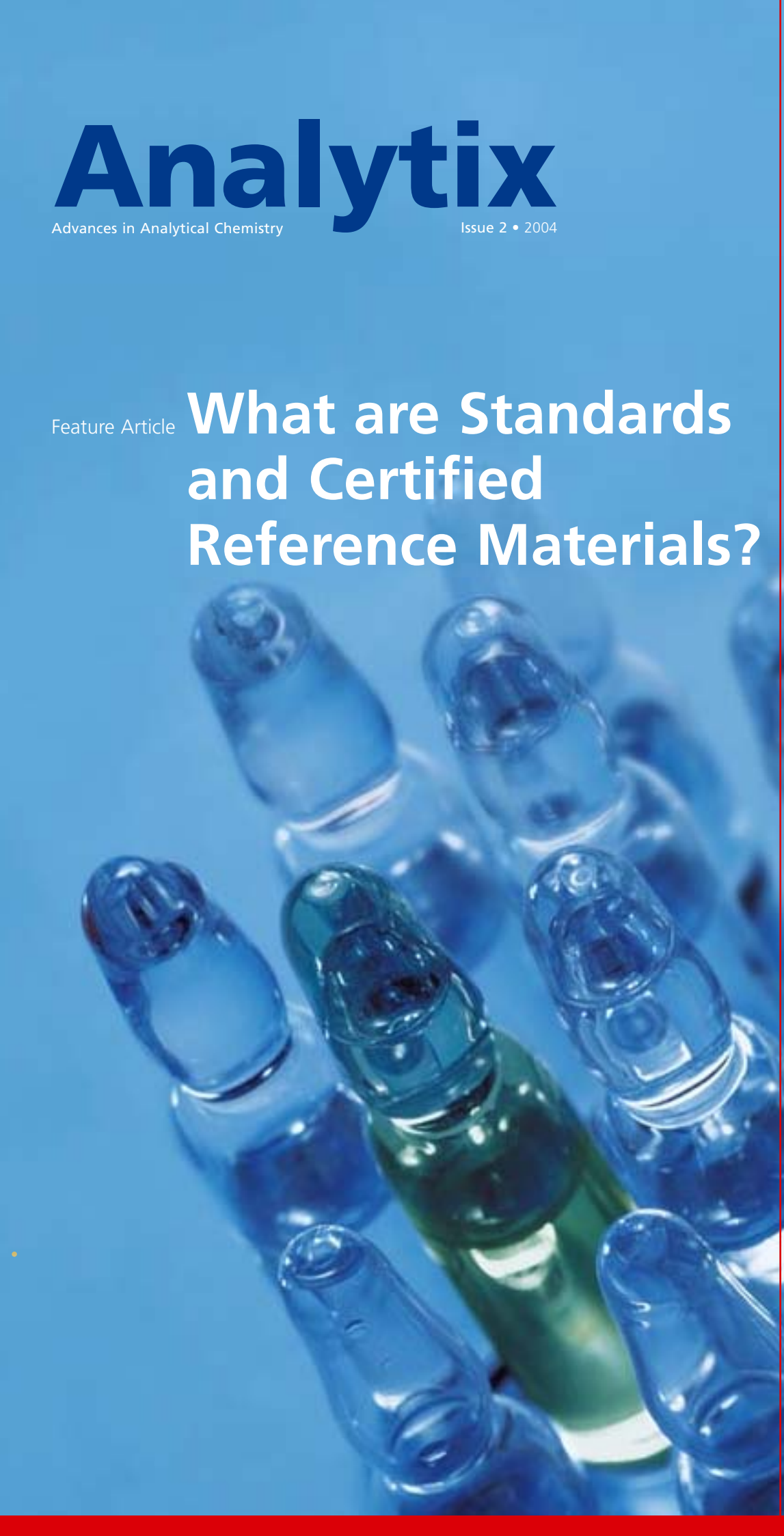
Advances in Analytical Chemistry

Issue 2 • 2004

Fluka
Riedel-de Haën®

Feature Article

What are Standards and Certified Reference Materials?



Derivatization reagents
Silylating Reagents

Analytical Reagents
Solvents for LC-MS

IPC Reagents

Solvents for Spectroscopy

HYDRANAL©
Laboratory Report

Water Standards

Application: Petrochemical Industry

Microbiology
Ready Prepared Plates for Water Analysis

New Chromogenic Media

Selective Media for *Bacillus anthracis*

Sensoric Application
Magnesium Ionophore IV

SIGMA-ALDRICH

Analytical Standards Catalog ..... Sigma-Aldrich launches its first comprehensive Analytical Standards Catalog. It's devoted exclusively to chemical standards and reference materials.

Picture ..... Rainer Walz, PhD
Analytical Standards and Reagents
Product Manager

Analytical Standards Catalog

Dear Colleague,
Sigma-Aldrich launches its first comprehensive Analytical Standards Catalog. It's devoted exclusively to chemical standards and reference materials. This new catalog brings together more than 7 000 products from across all relevant Sigma-Aldrich brands including Fluka, Riedel-de Haën, Supelco, Isotec, Sigma and Aldrich.

As a comprehensive guide to Sigma-Aldrich's Analytical Standards, the new Catalog will facilitate easier sourcing and ordering of products ranging from Chromatography to Spectroscopy and from Certified Standards to Special Packaging and Customization Services.

This new Catalog will help you find what you need much quicker and easier, by combining the products across all of our analytical standards brands.

What can we do for you?
If you like to find out if we also have a solution for your problem, please call the Technical Service Team or contact us via e-mail (see contact data on the back cover of this magazine).

We look forward hearing from you.
Kind regards

Rainer Walz, PhD
Analytical Standards and Reagents Product Manager

rwalz@sial.com

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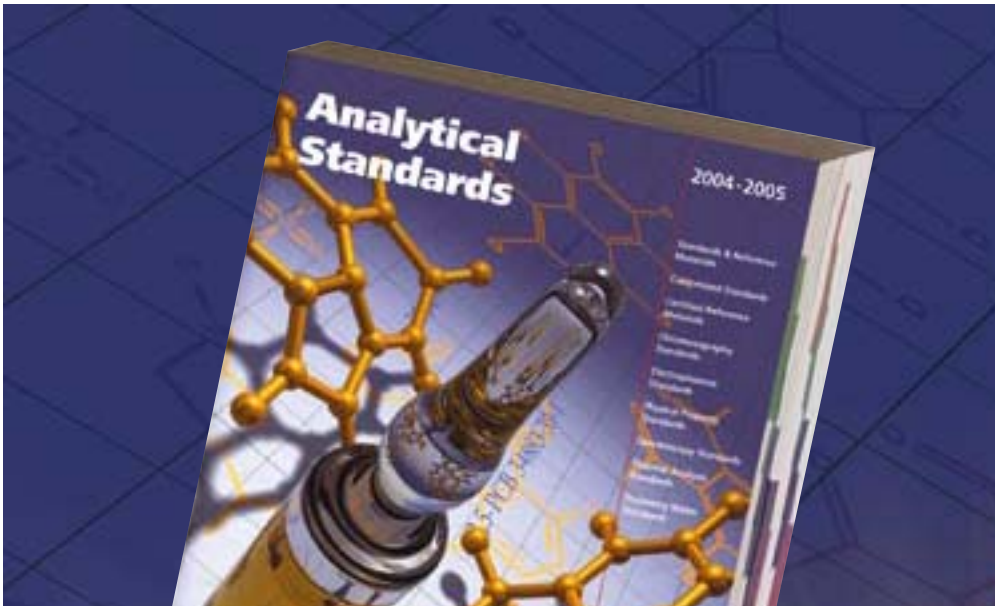
Picture .....
Analytical Standards Catalog



Standards, Certified Reference Materials and Reference Materials .....

Depending on the application that standards are used in, either chemical/physical quality control or pharmaceutical quality control/GMP production, different definitions will apply.

By Rainer Walz, rwalz@sial.com ....
Fluka Chemie GmbH, Buchs SG, Switzerland

Definition of Standards

Certified Reference Material meets the requirements of ISO guides 30-35

A Certified Reference Material must carry an SI (Système International d'Unités) traceable value and an attached uncertainty. Each Certified Reference Material is accompanied by a certificate stating the SI traceable value and uncertainty. The measurement procedure from sampling to calculation of the result must be fully validated. The term „Certified Reference Material" is used in chemical/physical application.

Reference Material

According to ISO guides 30-35 a substance whose property values are sufficiently homogenous and well established to be used for measurements. The term „reference material" is used in chemical/physical application.

Primary Standard

The requirements for a Primary Standard are the same as for Certified Reference Material. The term „primary standard" is used in pharmaceutical application.

Secondary Standard

A substance whose value refers to a primary standard. The term „secondary standard" is used in pharmaceutical application.

Tertiary Standard

A substance whose value refers to a secondary standard. The term „tertiary standard" is used in pharmaceutical application.

Pharmaceutical and GMP Application

Base	**Pharmacopoeia institutions, FDA-guideline, ICH-guide, PIC-guide**			
Divided into	**Primary standard**		**Secondary standard/ working standards**	**Tertiary standards/ working standards**
Substructure	In-house primary standards	Official standards of pharmacopoeias or official authorized institutions (e.g., EMPA, NIST, LGC, BAM)		
Certification procedure	• Characterization by assay determination, identification test, tests of impurities and by-products	• Collaborative testing • Assay determination	• Assay determination referring to a primary standard	• Assay determination referring to a secondary standard
Labeling and marking	**In accordance with official requirements, e.g., guidelines of pharmacopoeia: CRS = chemical reference substance (EP, BP); BRS = biological reference substance; RS = reference substance = primary standard (USP)**			
Use	• Identification • Assay determination • System suitability test • Instrument calibration • Impurity limits • Method validation • Purity		• System suitability test	• System suitability test

Table 1 ..... Definition of Certified Reference Materials, Reference Materials and Standards by Pharmaceutical and GMP Application

Chemical and Physical Application

Base	**ISO guide 30**	
Divided into	**Certified Reference Material (CRM)**	**Reference Material (RM)**
Substructure	Primary standard	
Certification procedure	• Each certified value is accompanied by an uncertainty at a stated level of confidence procedure • Certificate is included with the product • Certificate issued by a certifying body	• Properties sufficiently well established for the used methods
Labeling and marking	**In accordance with ISO guidelines**	
Use	• Identification • Assay determination • System suitability test • Instrument calibration • Impurity limits • Method validation • Purity	• System suitability test

Table 2 ..... Definition of Certified Reference Materials, Reference Materials and Standards by Chemical and Physical Application

		Certified Reference Material (CRM)		Reference Material (RM)	Primary Standard		Secondary Standard/ Working Standard	Tertiary Standard/ Working Standard
Group	**Subgroup**	**Primary Standard**	**Secondary Standard**		**In-House Primary Standard**	**Official Standards of Pharmacopoeia**		
CRMs from	Titrimetry	+	+	-	-	+	-	-
	Ion Chromatography	-	+	-	-	+	-	-
EMPA/BAM	Spectroscopy	-	+	-	-	+	-	-
	GPC/SEC	-	+	-	-	+	-	-
CRMs from IRMM	All	+	+	-	-	+	-	-
Electrophoresis Chromatography		-	-	-	-	-	-	+
	Environmental Standards	-	-	+	-	-	-	+
	Forensic & Veterinary Standards	-	-	+	-	-	-	+
	Ion Chromatography	-	-	+	-	-	-	-
	Life Science, Food & Beverage Standards	-	-	+	-	-	-	+
	Petroleum Standards	-	-	+	-	-	-	+
	Pharmaceutical Standards	-	-	+	-	-	-	-
	Polymer Standards	-	-	+	-	-	-	-
Physical Properties		-	-	+	-	-	-	+
	Color Reference Solutions	-	-		-	-	+	-
	Conductivity Standards	-	-	+	-	-	+	-
	Melting & Boiling Point	-	-	+	-	-	+	-
	MS Markers	-	-	+	-	-	-	+
	Particle Size Standards	-	-	+	-	-	+	-
	pH Calibration	-	-	+	-	-	+	-
	Turbidimetry	-	-	+	-	-	+	-
Spectroscopy		-	-	+	-	-	+	-
Titrimetry		-	-	+	-	-	+	-
Thermal Analysis		-	-	+	-	-	-	+

Table 3 ..... Overview of different standards and their categorization into table 1 and 2

Silylating Reagents for Gas Chromatographic Analysis ..... Silylation is the most widely used derivatization reaction for gas chromatography. Nearly all functional groups that present separation challenges, such as hydroxyl, carboxylic acid, amine, thiol or phosphate, can be silylanized.

By Rainer Walz and Vicki Yearick.....
rwalz@sial, vyearick@sial.com

A silylation reaction consists of the introduction of a silyl group into a molecule, usually in substitution for active hydrogen. Replacement of active hydrogen by a silyl group reduces the polarity of the compound and reduces hydrogen bonding. The silylated derivative thus is more volatile and more stable. Gas chromatographic detection is enhanced. The trimethylsilyl (TMS) group, $Si(CH_3)_3$ is the most popular and versatile silyl group for these purposes. A variety of trimethylsilylating agents with different derivatization properties are commercially available. Silylating mixtures have been developed to increase the silylating potential using catalysts and/or to reduce undesirable side reactions. For example, activated MSTFA mixtures are the most powerful silylating reagents available. Mixtures can save analysts time in preparing derivatives while reducing the number of chemicals to be inventoried. These, too, are readily available.

Cat. No.	Brand	Package Size	Product Name	Chemical Name
33018	Supelco	20 x 1mL	BSA + TMCS (5:1)	N,O-Bis(trimethylsilyl)acetamide + Trimethylchlorosilane (5:1)
33031-U	Supelco	25mL	BSA + TMCS + TMSI (3:2:3)	N,O-Bis(trimethylsilyl)acetamide + Trimethylchlorosilane +Trimethylsilylimidazole (3:2:3)
33155-U	Supelco	25mL	BSTFA + TMCS (99:1)	Bis(trimethylsilyl)trifluoroacetamide + Trimethylchlorosilane (99:1)
33046	Supelco	20 x 1mL	HMDS + TMCS (3:1)	1,1,1,3,3,3,-Hexamethyldisilazane + Trimethylchlorosilane (3:1)
394610-25ML	Supelco	25mL	Reacta-Sil Concentrate	1,1,1,3,3,3,-Hexamethyldisilazane + Trimethylchlorosilane (2:1)
33039	Supelco	25mL	HMDS + TMCS + Pyridine (3:1:9)	1,1,1,3,3,3,-Hexamethyldisilazane + Trimethylchlorosilane + Pyridine (3:1:9)
394882-5ML	Supelco	5mL	MTBSTFA	N-tert-Butyldimethylsilyl)-N-methyltrifluoroacetamide
375934-5ML	Supelco	5mL	MTBSTFA + TBDMCS (99:1)	N-tert-Butyldimethylsilyl)-N-methyltrifluoroacetamide + t-Butyldimethylchlorosilane (99:1)
33156-U	Supelco	25mL	TMSI + Pyridine (1:4)	Trimethylsilylimidazole + Pyridine (1:4)
33092-U	Supelco	10 x 1mL	T-Butyldimethylsilylimidazole-dimethylformamide	T-Butyldimethylsilylimidazole-dimethylformamide

www.sigma-aldrich.com/supelco-gc-derivatization

Cat. No.	Brand	Package Size	Product Name	Chemical Name
15222	Fluka	1mL, 5mL, 25mL	N,O-Bis(trimethylsilyl)acetamide	N,O-Bis(trimethylsilyl)acetamide
15243	Fluka	10mL, 50mL	N,O-Bis(trimethylsilyl) trifluoroacetamide	N,O-Bis(trimethylsilyl) trifluoroacetamide
52619	Fluka	10mL, 50mL, 250mL	Hexamethyldisilazane	Hexamethyldisilazane
69479	Fluka	1mL, 5mL, 25mL	N-Trimethylsilyl-N-methyltri-fluoroacetamide	N-Trimethylsilyl-N-methyltrifluoroacetamide

www.sigma-aldrich.com/fluka-gc-derivatization

Cat. No.	Brand	Package Size	Product Name	Chemical Name
69478	Fluka	1mL, 5mL	MSTFA + 1 % TMCS	N-Methyl-N-(trimethylsilyl)trifluoroacetamide with 1% trimethylchlorosilane
50992	Fluka	5mL, 25mL	MSTFA + Ethanethiol + Ammoniumiodide	N-Methyl-N-trimethylsilyltrifluoroacetamide activated with ethanethiol and ammoniumiodide
44156	Fluka	5mL, 25mL	MSTFA + Trimethylsilyl + Ethanethiol	N-Methyl-N-trimeth
12124	Fluka	5mL, 25mL	MSTFA + Imidazole	N-Methyl-N-trimethyl

www.sigma-aldrich.com/fluka-gc-derivatization

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Please send us back the enclosed reply card or download it from www.sigma-aldrich.com/supelco-library or www.sigma-aldrich.com/ analytix

Literature Code	Name
BGL	Guide to Derivatization Reagents for GC (Technical Bulletin)
AWH	BSA + TMCS (Product Specification Sheet)
AWI	BSA + TMCS + TMSI (Product Specification Sheet)
AWK	BSTFA + TMCS (Product Specification Sheet)
AWO	HMDS + TMCS (Product Specification Sheet)
AWP	HMDS + TMCS + Pyridine (Product Specification Sheet)
AWR	TMSI + Pyridine (Product Specification Sheet)
BFF	N-t-Butyldimethylsilylimidazole (Product Specification Sheet)
FGB	Analytix on Derivatizing Reagents

Table 1 ..... Available Literature

Introductory offer
Are Supelco and Fluka Silylating reagents new to you? 35% off when you order the Silylating Reagents listed on Tables 2, 3 and 4.

This offer is valid until 15th September 2004. Please quote Promotion Code 978 when placing your order.

Synonym	Special Advantages	Acids	Alcohols	Amines	Phenols	Thiols
Sylon BT	enhanced reactivity for amides, secondary amines, & hindered alcohols	++	++	++	++	+
Sylon BTZ	highly reactive, simultaneously derivatizes amino and hydroxyl groups	++	++	++	++	+
Sylon BFT	TMCS increases BSTFA reactivity	+++	+++	+++	+++	+
Sylon HT	greater silylating potential than HMDS alone	++	++	++	++	+
HMDS:TMCS (2:1)	TMCS increases reactvity rate	++	++	++	++	+
Sylon HTP	fast, versatile reagent	++	++	++	++	+
-	mild silylating reagent, derivatives very stable to hydrolysis	+++	++	+++	++	++
-	TBDMCS enhances amine derivatization	++	++	++	++	++
Sylon TP	useful for hindered steroids; chocie reagent for sugars with trace moisture	+++	++	-	++	+
-	useful for mass spectrometry, provides high-mass ions	+	+	-	+	-

Table 2 ..... Silylating Agents

Synonym	Special Advantages	Acids	Alcohols	Amines	Phenols	Thiols
BSA	Reactive, universal	++	++	++	++	+
BSTFA	Highly reactive, more volatile than BSA	++	++	++	++	+
HMDS	Gaseous by-products	+	++	+	++	+
MSTFA	Highly reactive, even more than BSTFA	++	++	++	++	+

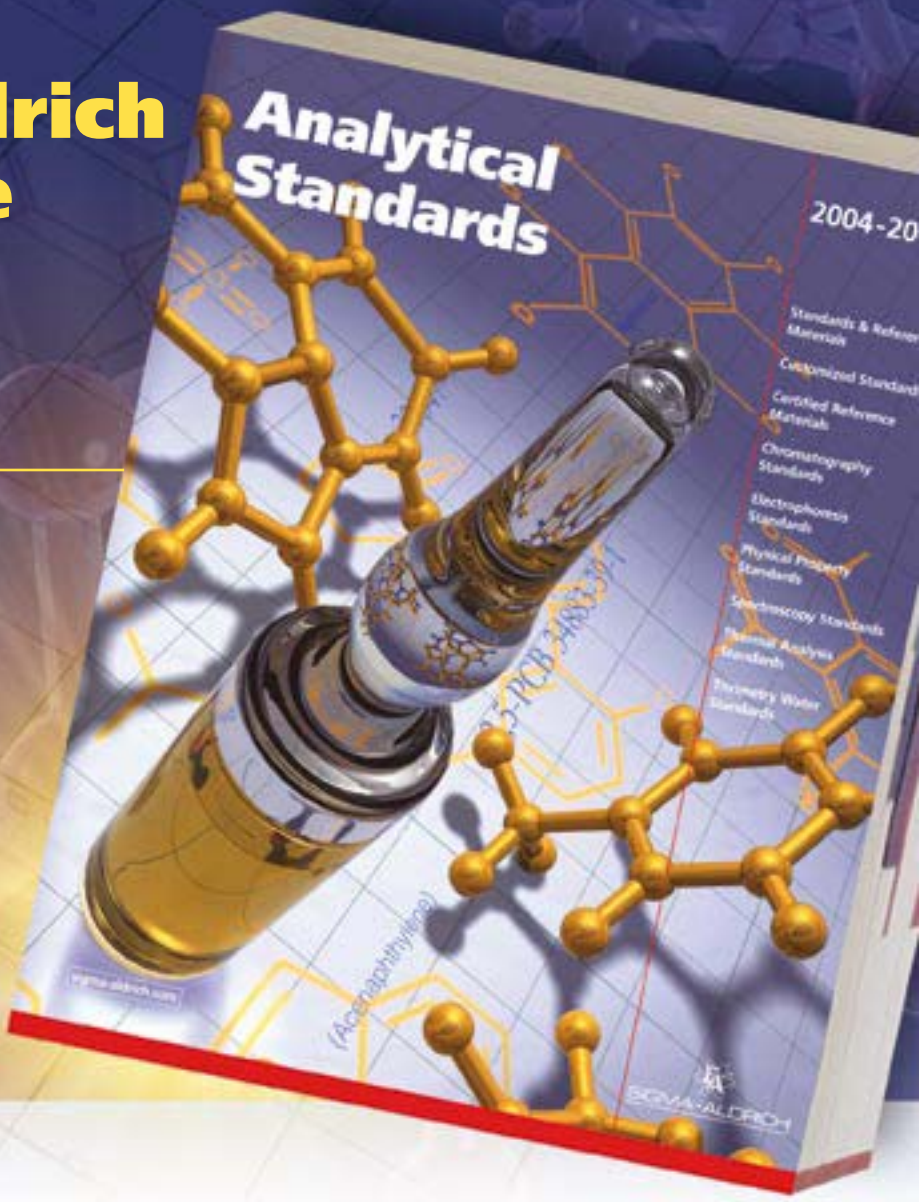
Table 3 ..... Trimethylsilylating Agents

Synonym	Special Advantages
MSTFA / TMCS	Sylilation
MSTFA activated I	Very powerful mixture for trimethylsilylation of -OH and -NH groups
MSTFA	Very powerful
MSTFA	Very powerful

Table 4 ..... MSTFA Mixtures

Looking for Analytical Standards?

Let Sigma-Aldrich Be Your Guide



ANNOUNCING

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LC-MS CHROMASOLV® Solvents ..... Riedel-de Haën has developed a new generation of LC-MS solvents that will allow you to get best possible performance out from your system. Make sure you will only get the TRUE peaks!

By Frederik Pillong ...fpillong@sial.com

LC-MS CHROMASOLV® Solvents

- Accurately tested for LC-MS (analysis based on the reserpine test of many suppliers)
- Very low amount of metal ions, such as sodium and potassium
- High UV-transmittance for combination with UV and diode array detection
- Excellent gradient baseline
- Particle tested

Specifications

Solvent content (GC) >99.9% (with the exception of ethyl acetate: 99.7%); LC gradient testing in UV and MS; metal impurities (Na, K, Mg, Ca: 1ppm); UV-transmittance and fluorescence; particle tested.

Picture .....
Only the TRUE PEAKS are worth to explore

Cat. No	Brand	Product Name	Pack Sizes
39253	Riedel-de Haën	CHROMASOLV® Water	1 L
34967	Riedel-de Haën	CHROMASOLV® Acetonitrile	1 L / 2.5 L
34966	Riedel-de Haën	CHROMASOLV® Methanol	1 L / 2.5 L
34965	Riedel-de Haën	CHROMASOLV® 2-Propanol	1 L / 2.5 L
34972	Riedel-de Haën	CHROMASOLV® Ethylacetate	1 L / 2.5 L

Table ..... Product range

Are you looking for an LC-MS column?
Please visit www.sigma-aldrich.com/lc-ms-columns

www.sigma-aldrich.com/lc-ms-solvents

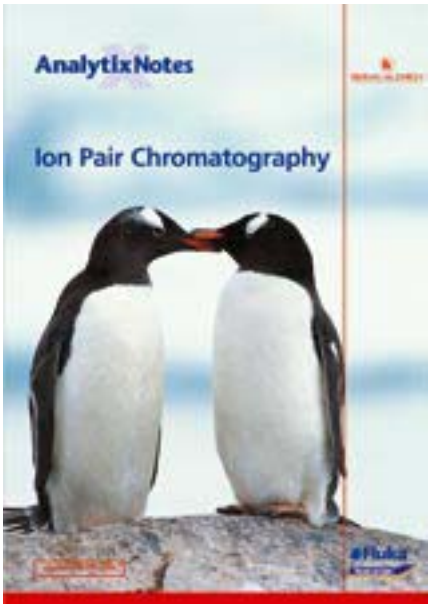
How to Select the Right Ion Pair Chromatography Reagent .....

The key to resolving complex mixtures of polar and ionic molecules is to select the right Ion Pair Chromatography (IPC) reagent.

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

The key to resolving complex mixtures of polar and ionic molecules is to select the right Ion Pair Chromatography (IPC) reagent. Alkyl sulfonates are a good first choice for basic solutes, whereas quaternary amines are useful for the acidic ones. Once this is done, the method can be further optimised by adjusting the pH and the concentration. Tables 1 and 2 give you an overview of IPC reagents for the separation of cations and Table 3 shows IPC reagents suitable for the separation of anions.

For a complete list of products, please visit
www.sigma-aldrich.com/ipc



Picture .....
Analytix Notes on Ion Pair Chromatography (FZI)

Do you want to know more about IPC? Please ask us for the Analytix Notes on Ion Pair Chromatography (FZI). Send back the enclosed reply card or download it from www.sigma-aldrich.com/analytix

Special Offer

Are these IPC reagents new to you? You can try them with our special Starter Kit offer. Get 40% discount on all recommended IPC reagents listed on Tables 1, 2 and 3.

This offer is valid until 15tu September 2004. Please quote Promotion Quote 976 when placing your order. This offer only applies to smaller pack sizes.

Cat. No.	Compound (solid)	Carbon length
52862	1-Hexanesulfonic acid sodium salt monohydrate	C6
51832	1-Heptanesulfonic acid sodium salt monohydrate	C7
74882	1-Octanesulfonic acid sodium salt monohydrate	C8

Table 1 ..... Selection of solid IPC reagents suitable for cation separation sorted by carbon chain length.

Cat. No.	Compound (concentrate)	Carbon length
52864	1-Hexanesulfonic acid sodium salt concentrate (~0.33M)	C6
51834	1-Heptanesulfonic acid sodium salt concentrate (~0.33M)	C7

Table 2 ..... Selection of IPC reagent concentrates suitable for cation separation sorted by carbon chain length. Concentrates are available in packages with 6 ampoules. Dilute to 1-liter with HPLC grade water (Cat. No. 95304) to obtain a 0.005M eluent solution.

Cat. No.	Compound (solid)	Carbon length (longest chain)
88106	Tetrapropylammonium hydrogensulfate	C3
86847	Tetrabutylammonium hydrogensulfate concentrate	C4
86853	Tetrabutylammonium hydrogensulfate	C4
87299	Tetrahexylammonium hydrogen sulfate	C6

Table 3 ..... Overview of solid IPC reagents suitable for anionic separation sorted by carbon chain length.

Did You Know .....

Riedel-de Haën offers you a special line of solvents for spectroscopy?

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

The SPECTRANAL® solvent line has been developed for UV-, IR-, and fluorescent spectroscopy. In addition to high chemical purity, guaranteed high UV- transmittance, and the absence of foreign band absorption in the IR-spectrum, these solvents are UV- and IR spectroscopy tested. A guaranteed low content of fluorescent substances ensures suitability for fluorescence detection. Proton-free solvents, such as carbon disulfide or carbon tetrachloride are additionally tested for the absence of interfering peaks in 1H-NMR spectroscopy. Solvents for hydrocarbon analysis are generally tested at high layer thickness.

Picture ..... Spectroscopy

Offer

35% off all spectroscopy solvents in this list.
Please quote Promotion Code 979 when placing your order.
Offer valid untill 15th September 2004.

Cat. No.	Brand	Grade and specification	Product	Pack Size
34905	Riedel-de Haën	SPECTRANAL®, >= 99.9 % GC	Carbon tetrachloride	1L, 2,5L
34906	Riedel-de Haën	SPECTRANAL®, >= 99.9 % GC	Methanol	2,5L
34908	Riedel-de Haën	SPECTRANAL®, >= 99.8 % GC	Dichloromethane	1L, 2,5L
34933	Riedel-de Haën	SPECTRANAL®, >= 99.9 % GC	Carbon disulfide	1L
34904	Riedel-de Haën	SPECTRANAL®, >= 99.8 % GC	2-Propanol	1L, 2,5L
34900	Riedel-de Haën	SPECTRANAL®, Reag. ACS, >= 99.8 % GC	Acetone	1L, 2,5L
34902	Riedel-de Haën	SPECTRANAL® , Reag. ACS, >= 99 %	Chloroform	1L, 2,5L
34915	Riedel-de Haën	SPECTRANAL®, >= 99.7 % GC	Dimethyl sulfoxide	1L
34903	Riedel-de Haën	SPECTRANAL®, >= 99.9 % GC	N,N-Dimethylformamide	1L, 2,5L
34907	Riedel-de Haën	SPECTRANAL®, >= 99.8 % GC	Ethylene glycol	1L
34929	Riedel-de Haën	SPECTRANAL®, 99.9 % GC	Toluene	1L, 2,5L
34913	Riedel-de Haën	SPECTRANAL®, >= 97.0 % GC	Hexane	1L, 2,5L
34911	Riedel-de Haën	SPECTRANAL®, Reag. ACS, Reag. Ph. Eur., >= 99.7 % GC	Cyclohexane	1L
34936	Riedel-de Haën	SPECTRANAL®, >= 99 % GC	Heptane	1L, 2,5L
34917	Riedel-de Haën	SPECTRANAL®, Reag. ACS, >= 99.7 % GC	Ethyl acetate	1L
34918	Riedel-de Haën	SPECTRANAL®, >= 99.5 % GC	2,2,4-Trimethylpentane	2,5L

Table ..... Product Range

Do You Want to Know the Latest Developments in Spectroscopy?

Sigma-Aldrich's SciBookSelect is a selected library of more than 2,000 items that helps you to keep pace with new technologies, while updating your protocols and knowledge. 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 SciBookSelect.

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HYDRANAL® Laboratory Report: Water Determination in Peppermint Oil and Spearmint Oil ..... The volumetric determination of water content is no problem for both oils when HYDRANAL®-CompoSolver E is added to the titration vessel and titrated with HYDRANAL®-Composite.

By Helga Hoffman .....
hhoffman@europe.sial.com

Picture .....
Peppermint

The volumetric determination of water content is no problem for both oils when HYDRANAL®-CompoSolver E is added to the titration vessel and titrated with HYDRANAL®-Composite. When methanol is used as solvent, it originates a weak by-reaction leading to a high water content determination. In spearmint oil, we found 0.083% H_2O in methanol, and only 0.073% in HYDRANAL®-CompoSolver E. In the coulometric determination, the weak by-reaction causes delays in the occurrence of the end point. In this instance, it is also advisable to use a reagent having ethanol instead of methanol. If you can adjust the device parameters, you should program a stop drift of 20 µg/min or an end level of 0.50µg/sec. We examined various oils. A peppermint oil contained 0.12% water; another 1.21%. In the spearmint oil we found 0.074%, and 0.69% H_2O in another sample.

Cat. No.	Brand	Product
Volumetry		
34806	Riedel-de Haën	HYDRANAL®-Composite 2
34734	Riedel-de Haën	HYDRANAL®-CompoSolver E
Coulometry		
34726	Riedel-de Haën	HYDRANAL®-Coulomat E

Table ..... Reagents used for this application

Volumetric method
In a titration vessel, 30mL HYDRANAL®-CompoSolver E are added and titrated to dryness with HYDRANAL®-Composite 5. Next, 1 g of the sample is weighed into the vessel by accurately back-weighing and its water content titrated with HYDRANAL®-Composite 2.

Coulometric method
5mL HYDRANAL®-Coulomat E are added in the cathode compartment in a coulometry cell with diaphragm cells; the anode compartment is also filled up to the same fill level with HYDRANAL®-Coulomat E. A cell without diaphragm only requires 100mL HYDRANAL®-Coulomat E. The device is turned on and, once the drift is low and stable, some 0.5mL of the sample are accurately back-weighed and injected.

HYDRANAL®- Water Standards traceable to NIST SRM 2890 ..... Calibration, validation and control of analytical instruments and reagents is a central requirement in regular Quality Management Systems. In the case of Karl Fischer titrations, the most widely used method for water determination, a known amount of water is required.

By Michael Jeitziner .....
mjeitziner@sial.com

Calibration, validation and control of analytical instruments and reagents is a central requirement in regular Quality Management systems. In the case of Karl Fischer titrations, the most widely used method for water determination, a known amount of water is required. Pure water can be used, but the amounts needed (10mg for volumetry, and 100µg – 1mg for coulometry) are difficult to weigh.

We recommend using water standards with an exact confirmed water content
- For titre determination
- For monitoring precision and accuracy
- For validation and inspection of KF titrators according to ISO 9000, GMP, GLP and FDA guidelines

If you have further questions concerning HYDRANAL® products, please contact us

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Traceability to a national standard or to a SI unit is often required in these guidelines. Three of our HYDRANAL®-Water Standards, one for volumetry and two for coulometry, are traceable to the NIST standard reference material SRM 2890, Water Saturated Octanol. „NIST" stands for the National Institute of Standards and Technology of the USA.

Liquid HYDRANAL®-Water Standards are currently used for the volumetric titre determination of Karl Fischer reagents, to control coulometric instruments and, most importantly, for the quality control of Karl-Fischer instruments in general as it relates to ISO 9001-9003 and other standard procedures. Three different water standards are currently available. Liquid HYDRANAL®-Water Standards consist of a solvent mixture with specific composition and a precisely determined water content. The exact water amount can be found on a Certificate of Analysis which is enclosed in each product package. HYDRANAL®-Water Standards are sealed under Argon in 4mL or 8mL glass ampoules. The humidity-proof packaging allows for storage up to five years. Each box contains 10 ampoules.

Picture ..... HYDRANAL® Water Standards

Advantages of HYDRANAL®-Water Standards

- Packaged in glass ampoules under argon
- Each ampoule only for one use
- Shelf life 5 years
- Contains instruction for use according to guide lines like ISO 9001-9004
- Manufactured by a company certified ISO 9001
- Certificate of Analysis enclosed in each pack
- Traceable to NIST SRM 2890

Cat. No.	Brand	Product	Description	Pack Size
34849	Riedel-de Haën	HYDRANAL®-Water Standard 10.0	Standard for volumetric KF titration, 1g (1mL) contains 10.0mg = 1.00% water at 20°C contains 10 glass ampoules of 8mL traceable to NIST SRM 2890	80mL
34828	Riedel-de Haën	HYDRANAL®-Water Standard 1.00	Standard for coulometric KF titration, 1g (1mL) contains 1.00mg = 0.10% water at 20°C contains 10 glass ampoules of 4mL traceable to NIST SRM 2890	40mL
34847	Riedel-de Haën	HYDRANAL®-Water Standard 0.10	Standard for coulometric KF titration, 1g contains 0.10mg = 0.01 % water at 20°C contains 10glass ampoules of 4mL traceable to NIST SRM 2890	40mL
34748	Riedel-de Haën	HYDRANAL®-Water Standard KF-Oven	Solid standard for control of KF ovens, assay of water: 5.55 ± 0.05%	10g
34803	Riedel-de Haën	HYDRANAL®-Standard Sodium Tartrate-2-hydrate	Solid Standard for titre determination of volumetric reagents. Water content 15.66±0.05%	25g, 100g
34813	Riedel-de Haën	HYDRANAL®-Standard 5.00	Liquid Standard for titre determination of volumetric reagents. Water content 5.00±0.02mg/mL at 20°C	100mL, 500mL

Table ..... Product Range

www.sigma-aldrich.com/hydranal

HYDRANAL® Application: Petrochemical Industry .....

Moisture in oils is one of the most serious contaminants and its presence must be carefully monitored in mineral oils. The knowledge of water content of mineral oil is important in refining, dealing and transfer (corrosion, viscosity).

By Michael Jeitziner .....
mjeitziner@sial.com

Introduction

Mineral oils and their products mainly consist of longer chained hydrocarbons, and as such are only soluble in methanol to a limited extend. A suitable addition to the solvent is often chloroform in order to improve the solubility, though this is not always successful as the problems encountered are often more complex. Water contents in the range from 0.2 to 20g/L are determined by volumetric Karl Fischer titration, as in ASTM (=American Society for Testing and Materials) method D 4377-00 „Standard Test Method for Water in Crude Oils by Potentiometric Karl Fischer Titration".

For the determination of water contents in the low ppm range, coulometry should be the method of choice. Besides its high precision, this method has distinguished itself due to the ease of introduction of liquid samples with a syringe. With the publication of the ASTM method, D4928-96 „Standard Test Methods for Water in Crude Oils by Coulometric" the coulometric determination of water in crude oils was established as an accepted procedure. The method is gaining increasing acceptance in the petroleum industry, where it is used by production engineers, transporters, and processors.

In practice many oils consisting of additives have to be investigated. Most of the additives as mercaptanes, thiophosphoric esters or overbased calciumsulfonates cause a side reaction which results into too high water contents. These oils have to be analyzed by the indirect method using a KF oven. Reagents and procedures are described for oils which do not cause side reactions and can therefore be done directly.

HYDRANAL® offers the broadest range of Karl Fischer reagents available while also being the safest reagents available on the market. The products are of the highest quality offering the largest water capacities, fastest titration times, stable end points, excellent reproducibility, and longest storage stability. When you purchase HYDRANAL® Karl Fischer reagents, you not only receive the products, but also the years of experience in this field developed by our technical support laboratories around the world.

Special Products for Oil analysis

34749 HYDRANAL-Solvent Oil for volumetric water determination in fats and oils

HYDRANAL®-Solvent Oil is a new solvent component which dissolves long-chain aliphatic molecule. It contains a long-chain alcohol as the main solvent along with the components required for the Karl Fischer reaction – sulfur dioxide, imidazole and methanol.

HYDRANAL®-Solvent Oil has a water capacity of approx. 3mg water per mL, i.e. in 30mL HYDRANAL®-Solvent Oil up to 90mg water can be titrated. This capacity is sufficient because oily materials contain only small amounts of water. HYDRANAL®-Titrant is used as titration reagent. In case chloroform is the preferred solubilizer, 34812 HYDRANAL®-Solvent CM can be used instead. Since the water content in fats and oils is low, 34811 HYDRANAL®-Titrant 2 or the ethanol-based 34723 HYDRANAL®-Titrant 2 E is recommended.

34868 HYDRANAL®-Coulomat Oil for the coulometric water determination in oils

The new reagent is based on methanol, to which defined quantities of aromatic and halogenated hydrocarbons have been added as solubilizers to increase its solubilizing properties. Also in HYDRANAL®-Coulomat Oil, samples of oil in suspension may be carried out. Nevertheless, the determination of water contents can be performed without any difficulties. The addition of xylene results in a higher solubility of especially tar, relevant amounts of which are contained in crude oils. Thereby the formation of tar layers on the indicator electrode can be inhibited and no disturbances are to be anticipated for the identification of the end points. The right portion of chloroform ensures a fine dispersion of all types of oil.

The optimally aligned formulation meets all requirements of customary coulometers with regard to the conductivity of the reagents of used. This new reagent is intended for use as an anolyte for coulometric cells with diaphragm in combination with the catholyte 34840 HYDRANAL®-Coulomat CG.

Picture .....
Petrochemical plant

We are offering a few more applications for the petrochemical industry: Diesel L427*, Engine Oil L201*, Fuel oil, heavy L111*, Greases L412*, Hydraulic Oil L107*, Insulating Oil L462*, Petrol unleaded L428*, Polybutene L188*, Used Hardness-Oil L381*.

*Laboratory Applications
Please contact our HYDRANAL-Laboratories (hhoffman@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

Crude Oil

(Laboratory Application L108*)

Crude oil is basically a mixture of hydrocarbons containing tar. The water in such a mixture is not at all homogeneously distributed, and as such, particular demands are placed on the handling of the sample. An addition of toluene to the working medium proved particularly helpful in the analysis of crude oils like Iran Heavy and Bachaquero grades.

Kerosene

(Laboratory Application L112*)

As kerosene contains only a few ppm of water, a determination of its water content by coulometry means using the most suitable method. To improve the solubility of kerosene, a mixture of 34807 HYDRANAL®-Coulomat A and chloroform (70 + 30 parts by volume) should be used. 40mL of kerosene will dissolve in 100mL of this mixture. The cathodic compartment is filled with 34840 HYDRANAL®-Coulomat CG.

Rape Seed Oil (Bio-diesel)

(Laboratory Application L319*)

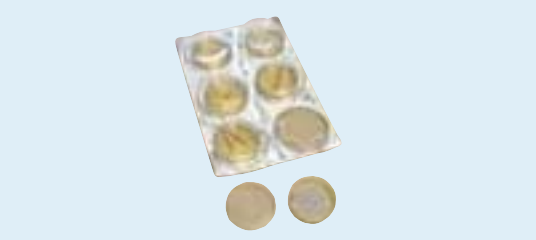
Rape seed oil is a substitute diesel made from renewable, non-fossil, organic materials and therefore enjoys great popularity. Rape seed oil does not dissolve in the methanolic working medium of the KF titration. Addition of solvents such as chloroform or 1-decanol improve the solubility of the rape seed oil, however, the limit is quickly reached. Chloroform can be avoided, if 37856 HYDRANAL®-LipoSolver MH is used in the titration vessel. In 30mL 2-3g sample can be dissolved. 34827 HYDRANAL®-Composite 1 or 34806 HYDRANAL®-Composite 2 are recommended as titrant reagents. For coulometric determination the solubility also has a limit. The addition of chloroform helps finely distribute the undissolved sample. It is important to choose a delay time of 30 seconds to ensure each injection of oil has fully titrated out.

Very well suitable is a special reagent for oils, 34749 HYDRANAL®-Coulomat Oil. This reagent contains as solubilizer chloroform and xylene as well.

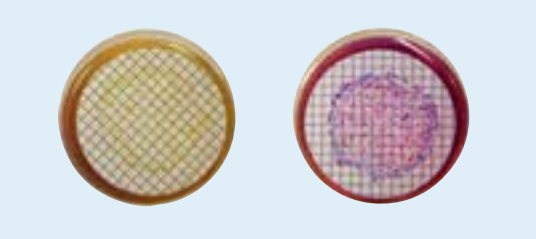
Ready Prepared Plates for Water Analysis ..... Prepared filtration plates with a 55mm diameter, presented in a blister pack and individually protected with a separate plastic bag. Avoiding fast dehydration and contamination enhances the shelf life of the media.

By Jvo Siegrist .....
isiegris@eurnotes.sial.com

The use of agar plates is described in several directives related to potable water control. Such solid media allows the precise enumeration of microorganisms in water by simply incubating for a few hours. When grown on plates, the microorganisms are characterized by a typical morphology. Also, it is easy to isolate a single colony for further confirmation. These plates were specially designed to accommodate 47mm diameter water filter membranes. Save time in the preparation of your assays by using these reliable, ready to use plates.



Picture 1 ..... Filtration plates in blister package



Picture 2 ..... m-CP Agar Plate with *Clostridium perfringens* before (left) and after (right) development

Picture 3 ..... Enterococcus sp. on Enterococcus Selective Agar Plate

Product Name	Brand	Cat. No.
Detection of Total and Fecal Coliforms		
For the detection of total coliforms incubate at 37°C, for fecal coliforms at 44°C mFC Agar Plates	Fluka	19958
Detection of Fecal Enterococci		
Enterococcus Selective Agar Plates (Slanetz-Bartley Agar; ISO 7899-2:2001)	Fluka	38259
Total Enumeration of Aerobic Microorganisms		
Nutrient Agar Plates	Fluka	44776
Plate Count Agar Plates	Fluka	00464
Tryptic Soy Agar Plates	Fluka	57994
Detection of Clostridia		
m-CP Agar Plates	Fluka	43047
Detection of Pseudomonas		
Cetrimide Agar Plates	Fluka	14521
Detection of Staphylococci		
Mannitol Salt Agar Plates	Fluka	9166
Detection of Moulds and Yeasts		
Sabouraud 4% Glucose Agar Plates	Fluka	40376

Table ..... Product range of plates for water control (Pack size: each box contains 30 plates; Cat. No.43047 contains 12 plates).

New Chromogenic Media .....

The determination of organisms is easier and more reliable using media with chromogenic substrates. Why not use these state of the art media instead of traditional ones?

By Jvo Siegrist .....
isiegris@eurnotes.sial.com

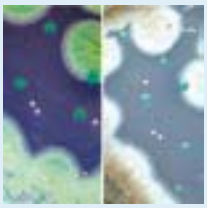
HiCrome Aureus Agar - Fluka Cat. No. 05662
(To be supplemented with Egg Yolk Tellurite Emulsion – Fluka Cat. No. 75208) This selective medium is used to isolate, enumerate and identify ***Staphylococci*** from environmental samples. Coagulase positive ***Staphylococcus aureus*** produces brown black colonies (see Picture 1) whereas ***Staphylococcus epidermidis*** gives yellow- slightly brownish colonies. Other organisms give rise to either colourless or blueish colonies due to the presence of a chromogen. For example, ***Listeria monocytogenes*** colonies are blueish while the ones from ***Bacillus spp, Escherichia coli*** and ***Micrococcus spp*** are colourless.

Picture 1 ..... HiCrome Aureus Agar; ***Staphylococcus aureus*** (black)

References

(1) A.C. Baird Parker, An improved diagnostic and selective medium for isolating coagulase-positive staphylococci. J. Appl. Bacteriol. 25, 12 (1962)

HiCrome OGYE Agar - Fluka Cat. No. 66481
(To be supplemented with Oxytetra Selective Supplement; Fluka Cat. No. 51239) This is a selective and differential medium which facilitates rapid isolation of yeasts and moulds from milk and milk products. Oxytetracycline makes the medium more selective by inhibiting the growth of ***Lactobacilli. Aspergillius niger*** appears as light blue coloured colonies with black spores due to presence of chromogenic substrate. ***Candida albicans*** shows green coloured colonies and ***Saccharomyces cerevisiae*** gives colourless colonies (see Picture 2).



Picture 2 .....
HiCrome OGYE Agar; ***Aspergillus niger*** (light blue), ***Candida albicans*** (green), ***Saccharomyces cerevisiae*** (colourless)

References

(1) D.A.A. Mossel, et al., Oxytetracycline-Glucose-Yeast Extract Agar for selective enumeration of moulds and yeasts in foods and clinical material, J. appl. Bact. 33, 454 (1970)
(2) D.A.A. Mossel, M. Visser, W.H.J. Mengerink, A comparison of media for the enumeration of moulds and yeasts in foods and beverages, Lab. Pract., 11, 109 (1962)

A Selective Media for *Bacillus anthracis* ..... The threat of bioterrorism, war and natural outbreaks require the exhaustive of control for insidious organisms. *Bacillus anthracis* is one of the most notorious controlled species.

By Jvo Siegrist .....
isiegris@eurnotes.sial.com

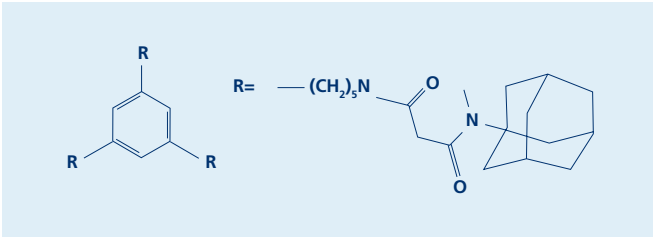
Fluka is now introducing a medium, recommended by the WHO for determining ***Bacillus anthracis*** (Fluka Cat. No. 55678). This medium is called PLET Agar after the initials of Polymyxin B - Lysozym - EDTA and Thallous acetate. In order to make it selective, an Anthracis Selective Supplement (Fluka Cat. No. 72659) must be added. Organisms such as ***Bacillus cereus*** are inhibited. ***Bacillus anthracis*** appears after 36-40 hours incubation at 37°C as a circular, creamy-white colony with a ground glass texture.

References

(1) R.F. Knisley J. Bacteriol., 92, 784 (1966)
(2) J.R. Norris, C.W. Berkley, N.A. Logan, A.G. O'Donnell, In M.P. Starr et al (ed), The Prokaryotes: a Handbook on Habitats, Isolation and Identification of Bacteria, vol. 2, Springer Verlag, Berlin (1981)
(3) J.M. Parry P.C.B. Turnbull, J.R. Gibson, A Colour Atlas of Bacillus species, Wolfe Medical Publications, London, UK (1983)

Magnesium Ionophore VI ..... The ion-selective electrodes based on Magnesium Ionophore VI show excellent Magnesium selectivity over calcium and potassium.

By Michael Jeitziner .....
mjeitzine@sial.com



Recommended Cell Assembly

Hg, Hg_2Cl_2 l KCl(sat.) l 3M KCl l l sample solution l l liquid membrane l 0.1 M $MgCl_2$ l AgCl, Ag

Magnesium Ionophore VI .....
(ETH 5506; 1,3,5-Tris-[10-(1-adamantyl)-7,9-doxo-6,10-diazaundecyl]benzol)
$C_{63}H_{96}N_6O_6$ M_r = 1033.49 [151058-38-7] (Fluka Cat. No 63112, Selectophore®)

Recommended Membrane Composition	Compound	Cat. No.	Brand
1.00 wt%	Magnesium Ionophore VI	63112	Fluka
0.3 wt%	Potassium tetrakis (4-chlorophenyl)borate	60591	Fluka
65.00 wt%	2-Nitrophenyl octyl ether	73732	Fluka
33.00 wt%	Poly(vinyl chloride) high molecular weight	81392	Fluka

Table ..... Reagents used for this application

Electrode Characteristics and Function

$\log K^{Pot}_{MgLi}$	-4.7	$\log K^{Pot}_{MgCa}$	-1.7
$\log K^{Pot}_{MgNa}$	-4.4	$\log K^{Pot}_{MgRb}$	-1.6
$\log K^{Pot}_{MgSr}$	-2.8	$\log K^{Pot}_{MgH}$	0.1
$\log K^{Pot}_{MgK}$	-2.7		

Slope of linear regression: 29.2±0.53mV.

Selectivity Coefficients $\log K^{Pot}_{MgLi}$ as obtained by the separate solution method (0.1 M solution of the corresponding chloride).

Table ..... Electrode Characteristics and Function

Literature

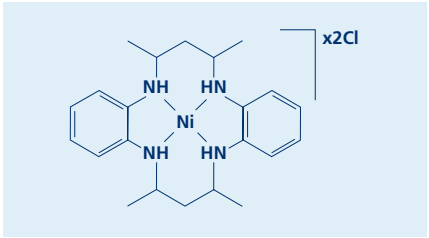
(1) W. Simon et.al. Anal. Chim. Acta 281, 129 (1993)

New Product Corner ..... Nickel Ionophor I

5,7,8,14-Tetramethyldibenzo[bi]-1,4,8,11-tetrazacyclotetradecannickel(II)chloride (Cat. No 30155).

A novel membrane sensor using Ni-Ionophor I, sodium tetraphenylborate (NaTPB) as anion discriminator and dibutyl butylphosphonate (DBBP) as plasticizer in a PVC matrix in the ration 2,5:1:100:100 (Ni-Ionophor I: NaTPB:DBBP:PVC) (w/w) works well in the concentration range 7.0x10-6-1.0x10-1M with a slope 29.8±0.2 mV/decade of activity. The response time is fast as 12s and has a good selectivity over a number of cations. The useful pH range of the electrode is 2.0-7.6. The sensor can work satisfactorily in 40%(v/v) water-methanol, ethanol and acetone mixtures, and can be used over a period of 5 months without any drift in potentials.

The electrode can be used as an indicator electrode in the potentiometric titration of Ni^{2+} against EDTA and for the direct determination of Ni^{2+} in samples like electroplating waste and chocolate.



Cat. No 30155: Nickel Ionophor I .....
5,7,8,14-Tetramethyl-dibenzo[bi]-1,4,8,11-tetrazacy-clotetradecannickel(II)chloride
(Cat. No 30155)

Literature

(1) Vinod K. Gupta, Rajendra Prasad and Azad Kumar, Sensors 2002, 2, 384-396.

Cat. No.	Brand	Description	Pack Size
30155	Fluka	Nickel Ionophor I	50mg, 250mg
38479	Fluka	Dibutyl butylphosphonate Selectophore® grade	1mL, 5mL, 25mL
72018	Fluka	Sodium tetraphenylborate Selectophore® grade	100mg, 1g
81392	Fluka	Poly(vinyl chloride) Selectophore® grade, high molecular weight	1g, 10g, 50g

Table ..... Reagents used for novel membrane sensor

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Upcoming Events .....

Hydranal® Seminars

When		Where
June	22th	Germany, Frankfurt
September	15th	Italy, Padova
October	20th	Switzerland, Egerkingen
November	3rd + 4th	Germany, Seelze
	17th	France, Paris

For registration and additional information, please contact
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SUPELCO — Chromatography Products for Analysis and Purification

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