

# Analytix

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## IRMM Certified Reference Materials



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Institute for Reference  
Materials and Measurements

- CRMs from the IRMM
- CRMs for Inorganic Trace Analysis
- Detection of Yeasts
- Standards for POPs
- Polymeric Adsorbent Media
- Water in Aldehydes and Ketones

**SIGMA-ALDRICH®**

## Certified Reference Materials



Michael Weber  
Manager Research &  
Development/Innovation Europe

### Dear Colleague,

As you may already know, Sigma-Aldrich has made significant progress over the last few years to become the world's No. 1 supplier of analytical chemistry products: about a quarter of our total portfolio of 100,000 chemical products are tailor-made for analytical techniques and applications. Within this very broad product offering, analytical standards and reference materials have a unique position, since they directly affect any analytical measurement result. It could be said that buying an analytical standard is a matter of trust.

Conscious of this fact, Sigma-Aldrich's analytical division has significantly improved internal skills and infrastructure, and intensified collaborations with selected partners. Among these partners there are several National Metrological Institutes (NMIs) representing the highest level of metrological competence in the world.

With the EMPA (former Swiss National Institute for Chemical Metrology) many successful projects have been realised and a series of Certified Reference Materials (CRMs) for titrimetry and IC have been introduced to the market. As a result Sigma-Aldrich constructed a new lab for CRM production in Switzerland, which is now operating under double accreditation following both ISO/ IEC 17025 and ISO Guide 34.

In addition, we have developed a long-lasting collaboration with the German NMI BAM (Federal Institute for Materials Research and Testing).

The Institute for Reference Materials and Measurements (IRMM) is one of seven institutes at the European Joint Research Centre (JRC) and represents the biggest NMI in Europe. IRMM has a long track record in the production and certification of CRMs and also operates under double accreditation. Several hundred IRMM CRMs are available and new products are added every year.

Sigma-Aldrich signed a distribution agreement with IRMM a few years ago, which means that all existing and new IRMM Reference Materials are available through Sigma-Aldrich's outstanding worldwide supply chain. This distribution agreement has recently been extended for another ten years and we are proud to be able to continue to bring the highest quality CRMs to our customers.

Buying analytical standards is a matter of trust. Why trust anyone else?

Best regards,

Michael Weber  
Manager Research & Development/Innovation Europe  
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Determination of Water Content in Samples Containing Aldehydes and Ketones

## Certified Reference Materials from the IRMM

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The IRMM (Institute for Reference Materials and Measurements), situated in Geel (Belgium), is one of the seven institutes of the Joint Research Centre (JRC), a Directorate-General of the European Commission (EC). It was founded in 1957 as the Central Bureau for Nuclear Measurements (CBNM) but the name was changed in 1993 to IRMM to reflect its new, broader remit.

The IRMM's mission is to promote a common and reliable European measurement system in support of EU policies. As a part of this, the IRMM supplies certified reference materials (pure and matrix materials) for various applications including environmental analysis, food analysis, clinical chemistry, physical properties or industrial applications.

In the late nineties, Sigma-Aldrich and the IRMM entered into a collaboration to develop reference materials for genetically modified organisms (GMOs). In addition but independent of this collaboration, Sigma-Aldrich and IRMM signed a distribution contract for CRMs. This successful distribution contract has now been renewed for another ten years. Sigma-Aldrich is proud to continue as an authorised distributor of IRMM reference materials.

The different Certified Reference Material (CRM) trademarks provided by the IRMM are:

**IRMM** certified reference materials: produced by the EC-JRC-IRMM.

**BCR**<sup>®</sup> certified reference material that was supported by funding via several other programmes of the European Commission.

**ERM**<sup>®</sup> certified reference materials: a new brand, launched through the ERM initiative, a collaboration between the Federal Institute for Materials Research and Testing (BAM) Germany, LGC Standards (UK) and the IRMM.

All these CRMs are produced according to the specific guidelines of the European Commission, which take into account the relevant ISO Guides 34 and 35. Each product is delivered with a certificate containing a certified value (including uncertainty), which is traceable to an SI unit or an internationally accepted reference. The certificate also states the intended use for each CRM. Stability of the CRMs is ensured by storage under controlled conditions and by monitoring programmes that have been set up to control CRM stability during shelf life.

Cat. No.	Product	Details	Pack Size
BCR-385R	Peanut Butter	Aflatoxin High Level	100 g
BCR-401R	Peanut Butter	Aflatoxin Low Level	100 g
ERM-BE376	Food and Feeding stuff	Aflatoxins	2 x 75 g
IRMM-351	Escherichia Coli 0157 (NCTC 12900)	1 lyophilised sphere	1 vial
IRMM-352	Salmonella enteritidis (NCTC 12694)	1 lyophilised sphere	1 vial
IRMM-354	Candida Albicans (NCPF 3179)	in material spheres	1 vial
ERM-BB124	Pork Muscle	Veterinary Drugs	10 g
IRMM-355	Enterococcus Faecalis (CIP 106877)	in material spheres	1 vial
ERM-BF425a	Soya 356043 (blank)	containing 0 % GMO material	1 g
ERM-BF425b	Soya 356043 (level 1)	containing 0.1 % GMO material	1 g
ERM-BF425c	Soya 356043 (level 2)	containing 1 % GMO material	1 g
ERM-BF425d	Soya 356043 (level 3)	containing 10 % GMO material	1 g
99999	GMO Set Soya 356043	Set containing one bottle of ERM-BF425 a, b, c and d	4 x 1 g

**Table 1** Some examples of food and feeding stuff CRMs

In the following paragraphs are listed some typical products and some which have been recently added to the portfolio. For a full understanding of the broad spectrum of different applications covered by the IRMM reference materials, and a complete product list, please visit our homepage at [sigma-aldrich.com/irmm](http://sigma-aldrich.com/irmm). There you will also be able to download the catalogue of IRMM reference materials and you will find a link to the IRMM homepage, where certificates and certification reports of all the products are directly accessible.

### Certified Reference Materials for Environmental Analysis

This product group comprises CRMs for pure materials and synthetic mixtures of organic pollutants (PAHs, PCBs, PCDDs, PCDFs etc.) as well as matrix materials (soils, ground- and wastewater etc.) with certified contents of elements or organic pollutants. Some examples are listed in **Table 2**.

Cat. No.	Product	Details	Pack Size
BCR-343	3-Hydroxybenzo(a)pyrene	Pure Material	10 mg
BCR-146R	Sewage Sludge (Industrial Origin)	Element Content	40 g
BCR-524	Industrial Soil (PAHs)	Organic pollutants	40 g
BCR-535	Freshwater Harbour Sediment	Organic pollutants	40 g

**Table 2** Examples of environmental CRMs

### Certified Reference Materials for the Analysis of Food and Feeding Stuff

Pure materials and synthetic mixtures of the CRMs for food and feeding stuff analysis include ethanol, sugar and syn-

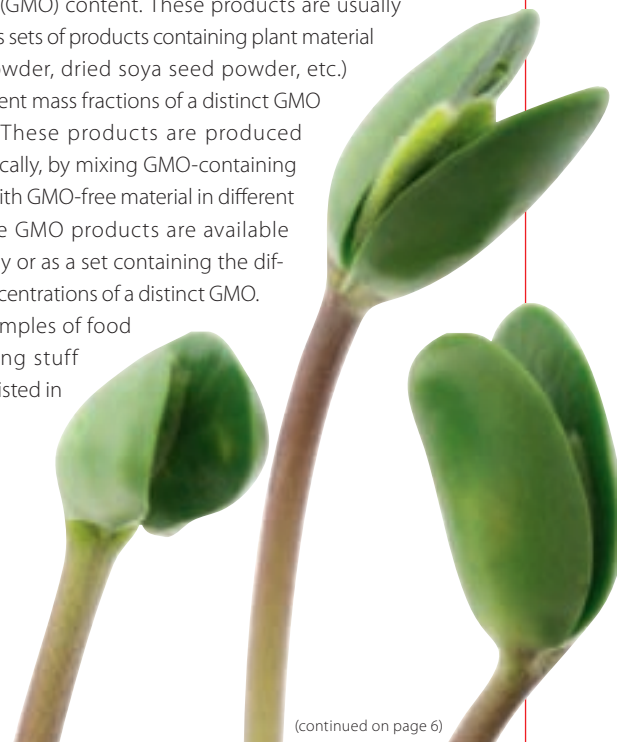
thetic wine, Tetramethylurea for SNIF-NMR and several certified solutions of natural toxins.

Food matrix standards comprise a very broad range of products, certified for natural toxins (e.g. BCR-385R, BCR-401R, ERM-BE376), xenobiotics, total element content, proximates and conventional properties, microbiological properties (e.g. IRMM-351, IRMM-352, IRMM-354 and IRMM-355), veterinary drugs (e.g. ERM-BB124) and identity.

A big product group are the CRMs for genetically modified organism (GMO) content. These products are usually available as sets of products containing plant material (maize powder, dried soya seed powder, etc.) with different mass fractions of a distinct GMO material. These products are produced gravimetrically, by mixing GMO-containing material with GMO-free material in different ratios. The GMO products are available individually or as a set containing the different concentrations of a distinct GMO.

Some examples of food and feeding stuff CRMs are listed in

**Table 1.**



(continued on page 6)



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### Certified Reference Materials for Clinical Chemistry

In addition to pure materials, matrix CRMs are available for hormone content, total element content, protein content, total element content, protein content (e.g. ERM-DA472/IFCC), catalytic activity (e.g. ERM-AD457/IFCC) and DNA sequences. Two examples are listed in **Table 3**.

Cat. No.	Product	Details	Pack Size
ERM-DA472/IFCC	Human Serum	Protein Content (CRP)	1 ampoule
ERM-AD457/IFCC	Aspartate Transaminase (AST)	Protein Activity	1 vial

**Table 3** Some examples of clinical chemistry CRMs

### Certified Reference Materials for Physical Properties

CRMs for physical properties are available for thermal, mechanical, morphological and optical properties. The recently added IRMM-304 is not a CRM but an example of a QCM (quality control material) for morphological properties. It consists of silica nanoparticles, suspended in an aqueous solution, and its intended use is to check the performance of instruments or methods that characterise the particle size distribution of nanoparticles suspended in a liquid medium.

Cat. No.	Product	Details	Pack Size
IRMM-304	Colloidal Silica	suspension in aqueous solution	1 ampoule

**Table 4** Example of a quality control material for morphological properties

### Certified Reference Materials for Industrial Applications

These products include industrial materials certified for the composition or for the content of trace elements or organic pollutants. Two examples are the recently added products ERM-EC590 polyethylene and ERM-EC591 polypropylene, with certified levels of several polybrominated diphenylethers (flame retardants).

Cat. No.	Product	Details	Pack Size
ERM-EC590	Polyethylene (LDPE)	Organic pollutants	20 g
ERM-EC591	Polypropylene (PP)	Organic pollutants	20 g

**Table 5** Examples of Industrial Applications CRMs

### Certified Reference Materials for Isotopic Measurements

There are two types of materials related to isotopic measurements: materials that are certified for the isotope abundance ratio (e.g. IRMM-010) and materials that are certified for the isotope amount content (e.g. IRMM-643). The former are suitable for calibration of measurements of isotope abundance ratios. The latter can be directly used in isotope dilution as “spikes” against which an unknown amount of an isotope or an element can be measured. These examples are listed in **Table 6**.

Cat. No.	Product	Details	Pack Size
IRMM-010	Platinum isotopic wire	Isotope abundance ratio	30 mg
IRMM-643	<sup>32</sup> S in 2.8M HNO <sub>3</sub>	Isotope amount content	5 mL

**Table 6** Examples of Isotopic Measurements CRMs



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## Yeasts

*A versatile organism with a diversified range of species ...*

Yeast is one of the most important microorganisms known and utilised by mankind. Ancient Middle Eastern civilisations used the organism to bake bread and to produce mead, beer and wine.

Yeast is one of the best studied eukaryotic organisms and is often used in molecular biology as well as diverse other applications. It is the most frequently used organism for fermentation. Yeasts are unicellular fungi, although they sometimes build multicellular constructs known as pseudohyphae, or false hyphae. Most yeasts are not pathogenic; however there are certain strains that can cause infections in humans. In current technology, yeasts have an important role in the production of biofuels. The biomass from yeast is also used as a nutritional supplement or as a component of microbiological media. It is also an important source of vitamin B<sub>12</sub>.

The natural sources of yeast vary broadly. They may be found in the ocean, on the skin of humans and animals, and on the skins of fruit.

### Reproduction:

Yeasts have asexual and sexual reproductive cycles; however, the most common method for reproduction is asexual by budding or fission. The nucleus of the parent cell splits and a daughter cell is built out of the parent cell and is separated.

The sexual reproduction method (meiosis) entails division of a diploid chromosome set and production of haploid spores, which then may conjugate with another cell to reform a diploid cell. However, haploid cells will generally die under stress, while spores can survive harsh conditions.

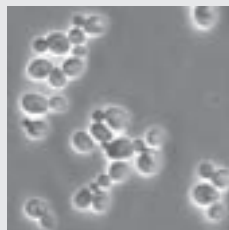
<b>Domain:</b>	Eukaryota	
<b>Kingdom:</b>	Fungi	
<b>Typical divisions:</b>		
Ascomycota	Saccharomycotina (true yeasts: e.g. <i>Saccharomyces</i> , <i>Candida</i> and <i>Pichia</i> species)	
	Taphrinomycotina	Schizosaccharomycetes (fission yeasts)
Basidiomycota	Urediniomycetes	Sporidiales

**Table 1** Scientific classification and typical divisions (not complete)

There are diverse species of yeasts with a broad range of applications. While the majority of yeasts are beneficial to humans and provide benefits such as biotechnological advancements, other unpleasant organisms may lead to infections or cause spoilage of fruit juices and alcoholic beverages. Some of the best-known yeast species with their respective descriptions are listed in the info box on the right-hand side.

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

### Did you know?



Under normal conditions, a yeast cell can live up to two days and produce about 20 offspring? But in this time it will have more than a 10<sup>20</sup> descendants.

- *Saccharomyces cerevisiae*: also called budding yeast or baker's yeast. *Saccharomyces* comes from the Greek term for sugar mould. The organism is also used as top-fermenting yeast in the brewery. It can ferment glucose, fructose, mannose, galactose, maltose, maltotriose, raffinose, and trehalose, but not lactose and cellobiose. The cells are round to ovoid and have a diameter of 5–10 micrometres. The organism reproduces by asexual reproduction called budding.
- *Saccharomyces carlsbergensis*, *Saccharomyces uvarum*: bottom-fermenting yeasts, used for the production of lager beer; also ferments many sugars at low temperatures.
- *Candida albicans*: an opportunistic pathogen that causes oral, intestinal and vaginal infections.
- *Candida utilis*: used in the production of Kefir
- *Brettanomyces bruxellensis* (*Dekkera bruxellensis*): spoilage organism in wine
- *Pichia pastoris*: organism for biotechnological production of proteins (high growth rate and non-fastidious)
- *Malassezia furfur*: responsible for skin problem (scurf)



**Figure 1** Several baker's yeast colonies on a microbiological medium

(continued on page 8)

Media	Brand	Cat. No.	Organisms/remarks
CaCO <sub>3</sub> Agar	Fluka	40545	yeast differentiation based on acid production from glucose
BiGGY Agar	Fluka	73608	<i>Candida albicans</i> , <i>Candida tropicalis</i> .
Corn Meal Agar	Fluka	42347	yeasts, fungal stock cultures, chlamyospore production by <i>Candida albicans</i> , mycel/pseudo-mycelium forming
Czapek Dox Agar	Fluka	70185	fungi and yeasts, defined C-source and inorg. N-source
Dichloran Rose bengal Agar (Base)	Fluka	17147	yeasts and moulds
EMB Agar	Fluka	70186	rapid identification of <i>Candida albicans</i> , differentiation and identification of bacteria ( <i>E. coli</i> , <i>Aerobacter aerogenes</i> , Staphylococci)
Levine EMB Agar	Fluka	62087	rapid identification of <i>Candida albicans</i> , differentiation and identification of bacteria ( <i>E. coli</i> , <i>Aerobacter aerogenes</i> , Staphylococci)
Lysine Medium	Sigma	L5910	wild yeasts in pitching yeasts, defined medium
Malt Agar	Sigma	M9802	yeasts and moulds
Malt Extract Agar	Fluka	70145	yeasts and moulds
Malt Extract Agar modified, Vegitone	Fluka	38954	yeasts and moulds
Malt Extract Broth	Fluka	70146	yeasts and moulds
OGY Agar	Fluka	75310	yeasts and moulds
Orange-serum Agar	Fluka	75405	acid-tolerant spoilage microorganisms
Peptone Yeast Extract Agar	Fluka	77196	yeasts and moulds
Potato Glucose Agar	Fluka	70139 51684	yeasts and moulds
Potato Glucose Rose bengal Agar (Base)	Fluka	17204	For promoting ascospore production
Potato Glucose Sucrose Agar	Fluka	17205	<i>Zygosaccharomyces rouxii</i>
Rice Extract Agar	Fluka	83551	yeasts, differentiation by chlamyospores and morphology
Rose Bengal Agar Base	Fluka	R1273	yeasts and moulds
Rose bengal Chloramphenicol Agar	Fluka	17211	yeasts and moulds
Sabouraud 2 % Glucose Agar	Fluka	84086	dermatophytes, fungi and yeasts
Sabouraud 4 % Glucose Agar	Fluka	84088 40376 55277	cultivation of pathogenic and non-pathogenic fungi, especially dermatophytes
Sabouraud Glucose Agar with Chloramphenicol	Fluka	89579	fungi and yeasts
Sabouraud Glucose Broth	Fluka	S3306	yeasts, moulds and aciduric bacteria
Sabouraud Maltose Agar	Fluka	S4181	dermatophytes, fungi and yeasts
SD Agar	Fluka	84605	yeast
Tomato Juice Broth	Fluka	17218	yeasts and other aciduric microorganisms

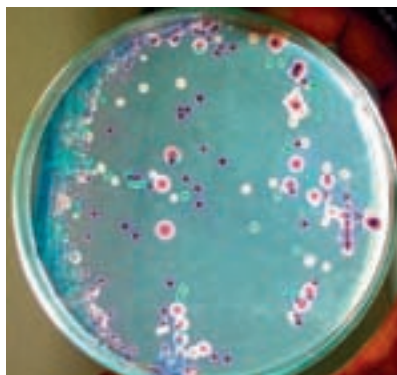
**Table 2** Most common media for yeasts

For the detection, enumeration and cultivation of yeasts, the classical culture method is still needed. The media are in many cases very simple, as yeast is non-fastidious. Regarding selectivity, oxytetracyclin, gentamicin, chloramphenicol, rose bengal, chlortetracycline, and bismuth ammonium citrate at low pH conditions are often used. For differentiating the morphology of the colony, the microscopic image (budding, spore production, mycel/pseudo-mycelium forming) and biochemical reactions are used. An overview of some common media is given in **Tables 2** and **3**.

Selective chromogenic media	Brand	Cat. No.	Organisms/remarks
HiCrome™ OGYE Agar Base	Fluka	66481	yeasts and moulds; <i>C. albicans</i> shows green and <i>Sacch. cerevisiae</i> colourless colonies
Candida Ident Agar	Fluka	94382	<i>Candida albicans</i> (green), other yeasts (white, pink, beige), Gonococci, moulds

**Table 3** Chromogenic media for yeasts and moulds

Chromogenic media, one of the cleverest systems, provide a perfect solution for the selective isolation, differentiation and identification of moulds and yeasts. With the aid of chromogenic substrates, certain characteristic enzymes are detected, and antibiotics inhibit bacterial growth. Differentiation is easy because of the different colours of the colonies, depending on the species, caused by the ability to cleave different chromogenic substrates (see **Table 3** and **Figure 2**).



**Figure 2** *Candida* Ident Agar, modified (*Candida albicans* = green-coloured smooth colonies, *Candida tropicalis* = blue to metallic blue-coloured raised colonies, *Candida glabrata* = cream to white, *Candida krusei* = purple coloured fuzzy colonies)

### New Detection Kits for Yeasts

The HybriScan® Test Kit is a new technology based on a rRNA probes sandwich method. It is possible to detect diverse species or a group of organisms at the same time if the appropriate probes are chosen. The test needs no PCR and is as simple as the ELISA test. It is a very robust

system and detects only living cells, unlike the PCR. It is also possible to quantify the cell numbers with a colour reaction and microplate reader. The HybriScan®**D** Yeast kit was designed with a specific probe for yeasts of the family of Saccharomycetaceae. The HybriScan®**D** Drink kit detects additional beverage-spoiling bacteria.

More information about the theoretical background of the system, additional kits available, and much more, can be found under [sigma-aldrich.com/hybriscan](http://sigma-aldrich.com/hybriscan)

Product Description	Tests	Brand	Cat. No.
HybriScan® <b>D</b> Yeast	96	Fluka	61397
<b>Specificity:</b> yeasts including genera <i>Zygosaccharomyces</i> , <i>Saccharomyces</i> , <i>Candida</i> , <i>Dekkera</i> , <i>Torulaspota</i> and <i>Pichia</i>			
HybriScan® <b>D</b> Drinks	96	Fluka	68301
<b>Specificity:</b> amongst other yeasts of the genera <i>Saccharomyces</i> , <i>Zygosaccharomyces</i> , <i>Brettanomyces</i> , <i>Torulaspota</i> , <i>Pichia</i> , <i>Candida</i> and bacteria of the genera <i>Lactobacillus</i> , <i>Acetobacteraceae</i> and <i>Alicyclobacillus</i>			

**Table 4** HybriScan Kits for detection of yeast species

### References

- [1] Balasubramanian M.K., Bi. E., Glotzer M. "Comparative Analysis of Cytokinesis in Budding Yeast, Fission Yeast and Animal Cells". *Curr. Biol.* 14 (18) (2004).
- [2] Yeong F.M., "Severing All Ties Between Mother and Daughter: Cell Separation in Budding Yeast". *Mol. Microbiol.* 55 (5), p. 1325–31 (2005).
- [3] Neiman A.M., "Ascospore Formation in the Yeast *Saccharomyces cerevisiae*". *Microbiol. Mol. Biol. Rev.* 69 (4), p. 565–84 (2005).

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## Analytical Standards for Persistent Organic Pollutants

Matthias Nold, Product Manager Analytical Standards [matthias.nold@sial.com](mailto:matthias.nold@sial.com)

Persistent organic pollutants (POPs) are a threat to human health and to the environment. Since they are very stable and hydrophobic molecules, they bio-accumulate through the food web and pose significant health risks such as cancer, birth defects, reproductive disorders, and dysfunctional nervous and immune systems in humans and in wildlife. Polar regions are most exposed, since many toxins are swept to the Arctic by ocean or air currents; no individual in any global region, however, is free from measurable traces of POPs.

With the aim to eradicate the most dangerous of these chemicals, the Stockholm Convention on Persistent Organic Pollutants was organised under the guidance of the United Nations Environment Programme (UNEP) in 2001. The Convention, which entered into force in May 2004, listed the 12 most dangerous POPs (called the “dirty dozen”) that shall be either outlawed or strictly limited. These 12 initial chemicals included a range of chlorinated pesticides (DDT, Aldrin, Chlordane, Dieldrin, Endrin, Heptachlor, Hexachlorobenzene, Mirex and Toxaphene) as well as dioxins and polychlorinated biphenyls (PCBs).

In May 2009, a follow-up conference was held in Geneva to update the list of hazardous chemicals. Parties agreed to



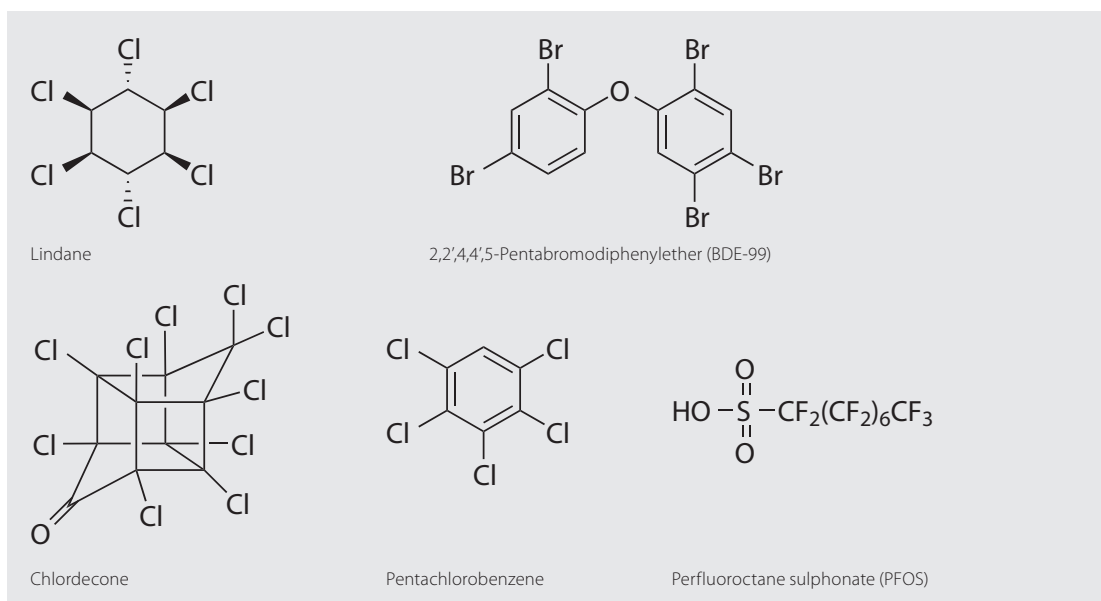
expand the list by nine more chemicals, including the insecticide Lindane,  $\alpha$ - and  $\beta$ -hexachlorocyclohexane, the pesticide chlordecone, perfluorooctane sulphonic acid, pentachlorobenzene as well as hexabromobiphenyl, and two bromodiphenylether congeners (BDE) which are used as flame retardants.

The goal of the convention, to protect human health and the global community by reducing and eventually eradicating

Product Name	Prod. No.	Brand	Pack Size
Aldrin	36666	Fluka	250 mg
Aldrin solution 100 ng/ $\mu$ L in acetonitrile	36664	Fluka	2 mL; 10 mL
Chlordane (technical mixture)	45378	Fluka	250 mg
$\alpha$ -Chlordane solution 10 ng/ $\mu$ L in cyclohexane	31197	Fluka	2 mL
Dieldrin	33491	Fluka	100 mg
Dieldrin solution 100 ng/ $\mu$ L in acetonitrile	36660	Fluka	2 mL
Endrin	32014	Fluka	250 mg
Endrin solution 200 ng/ $\mu$ L in isooctane	48976	Supelco	10 mL
Heptachlor	49041	Supelco	1000 mg
Heptachlor solution 100 ng/ $\mu$ L in methanol	31211	Fluka	2 mL; 10 mL
Hexachlorobenzene	45522	Fluka	250 mg
Hexachlorobenzene solution 1000 ng/ $\mu$ L in acetone	40008	Supelco	1 mL
Hexachlorobenzene- $^{13}\text{C}_6$	606332	Aldrich	10 mg
Mirex	36170	Fluka	100 mg
Mirex solution 100ng/ $\mu$ L in acetonitrile	45887	Fluka	10 mL
Toxaphene	PS79	Supelco	1000 mg
Toxaphene solution 500ng/ $\mu$ L in methanol	48243	Supelco	1 mL
4,4'-DDT	31041	Fluka	100 mg
4,4'-DDT 100ng/ $\mu$ L in methanol	36662	Fluka	2 mL
4,4'-DDT- $d_8$	34021	Fluka	10 mg
Polychlorinated dibenzodioxins (“dioxins”)	For a complete list of Dioxin standards available at Sigma- Aldrich please see: <a href="http://sigmaaldrich.com/dioxinstandards">sigmaaldrich.com/dioxinstandards</a>		
Polychlorinated biphenyls (PCBs)	For a complete list of PCB standards available at Sigma-Aldrich please see: <a href="http://sigmaaldrich.com/PCBstandards">sigmaaldrich.com/PCBstandards</a>		

**Table 1** Standards for the first series of banned persistent organic pollutants (“dirty dozen”)

(continued on page 12)



**Figure 1** Structures of some of the persistent organic pollutants recently added to the list of banned chemicals

these dangerous substances, is a very challenging one. Analytical chemistry can supply an important contribution by measuring the levels of persistent organic pollutants in food and in the environment. This not only protects consumers from the exposure and uptake of these toxins, but also helps monitor the progress of the worldwide elimination of POPs.

Sigma-Aldrich provides analytical standards for the hazardous chemicals targeted by the Stockholm Convention.

**Tables 1** and **2** list standards (neats and solutions) and, if available, isotopically marked standards suitable for the analysis of persistent organic pollutants.

#### Literature and Links:

- Official page of the Stockholm Convention: [www.pops.int](http://www.pops.int)
- [sigma-aldrich.com/dioxin](http://sigma-aldrich.com/dioxin)
- Supelco brochure "Dioxin and PCB analysis"
- [sigma-aldrich.com/etc/medialib/docs/Supelco/General\\_Information/dioxin-brochure-jxb.Par.0001.File.tmp/dioxin-brochure-jxb.pdf](http://sigma-aldrich.com/etc/medialib/docs/Supelco/General_Information/dioxin-brochure-jxb.Par.0001.File.tmp/dioxin-brochure-jxb.pdf)

Product Name	Prod. No.	Brand	Pack Size
Lindane	45548	Fluka	250 mg
Lindane solution 200 ng/μL in isoctane	48960-U	Supelco	10 mL
Lindane - <sup>13</sup> C <sub>6</sub>	606715	Aldrich	on demand
α-Hexachlorocyclohexane	33856	Fluka	100 mg
α-Hexachlorocyclohexane solution 100 ng/μL in methanol	36658	Fluka	2 mL
β-Hexachlorocyclohexane	33376	Fluka	100 mg
β-Hexachlorocyclohexane solution 100 ng/μL in methanol	36584	Fluka	2 mL
*2,2',4,4',5-Pentabromodiphenylether (BDE-99) solution 50 ng/μL in isoctane	33676	Fluka	1 mL
Chlordecone	45379	Fluka	250 mg
Pentachlorobenzene	35886	Fluka	1 g
Pentachlorobenzene solution 200 ng/μL in methanol	48386	Supelco	1 mL
Perfluorooctane sulphonate	33827	Fluka	100 mg
Perfluorooctane sulphonate solution 100 ng/μL in methanol	33607	Fluka	1 mL

\* For a complete list of BDEs or other flame retardant standards, please visit: [sigmaaldrich.com/flameretardants](http://sigmaaldrich.com/flameretardants)

**Table 2** Standards for the second series of banned persistent organic pollutants

## TraceCERT® Inorganic Standards for AAS, ICP and IC

Matthias Nold, Product Manager Analytical Standards [matthias.nold@sial.com](mailto:matthias.nold@sial.com)



With our Fluka-branded **TraceCERT** products, we offer a product line of certified reference materials that meet the highest metrological standards.

**TraceCERT** standards are produced in a double-accredited laboratory, fulfilling ISO/IEC 17025 and ISO Guide 34. This is the highest achievable quality standard for producers of certified reference materials.

**TraceCERT** products are characterised by:

- Unique (metrological) level of accuracy and lot-specific values and low uncertainties
- Traceability to at least two independent references (i.e. NIST, BAM or SI unit kg)
- Produced and certified according to ISO/IEC 17025 and ISO Guide 34
- Highest-purity starting materials available
- Comprehensive documentation according to ISO Guide 31
- Certificates of ICP standards list up to 70 trace impurities
- ICP standards packaged in light- and gas-tight aluminium foil bags
- Competitive price

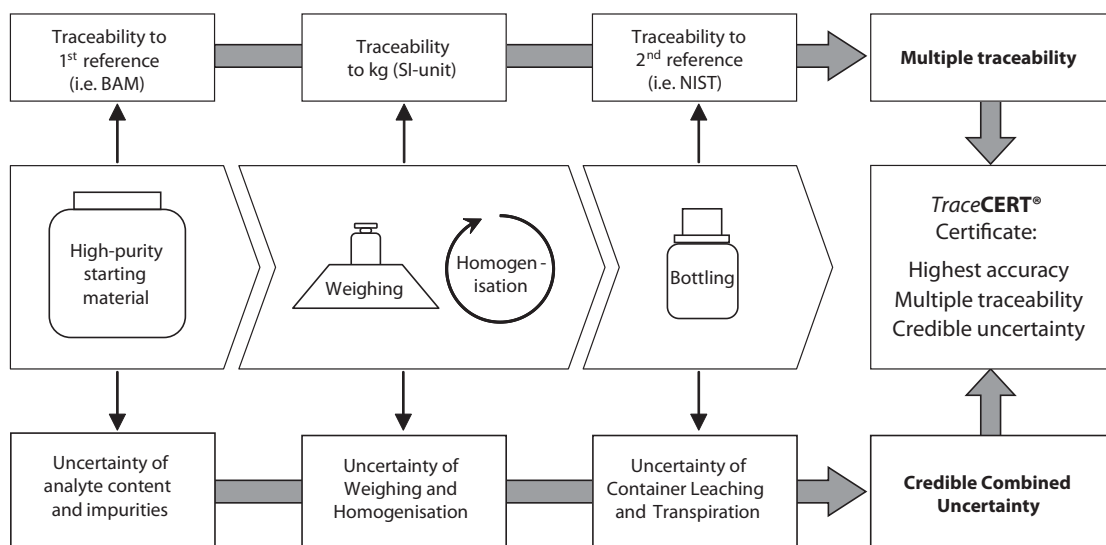
### CRM production at Sigma-Aldrich

In response to the increased importance placed on certified reference materials (CRM) in recent years, Sigma-Aldrich has established partnerships with several metrological institutes that serve as certifying bodies. For certified elemental and anion calibration solutions, Sigma-Aldrich has maintained a long-standing partnership with Swiss Federal Institute for Materials Science and Technology (EMPA). When EMPA terminated their activities in chemical metrology in 2004, Sigma-Aldrich decided to acquire the metrological competency from EMPA and transfer it to its Buchs site in eastern Switzerland. This transfer included not only unique equipment of the highest metrological level, but also the acquisition of technical capabilities and experience by hiring the veteran leadership of EMPA's Metrological Reference and Certification Labs. These investments enabled Sigma-Aldrich to build an entirely new product line of high-quality certified reference materials in a very short time.

### Production process of the TraceCERT products

To produce CRMs that meet all the criteria listed above, the entire development process must be highly sophisticated, beginning from the selection of an appropriate starting mate-

(continued on page 14)



**Figure 1** Schematic representation of the production design and certification of Sigma-Aldrich TraceCERT® products. This approach was established for the preparation of intercomparison samples at the highest metrological stage.

rial to the choice of a purpose-specific packaging. **Figure 1** shows an overview of the production and certification process we employ.

**TraceCERT® starting materials (Figure 2)** are **characterized** by two different approaches:

- (1) direct measurement of the purity by the most accurate method (e.g. titrimetry)
- (2) comparison to an internationally accepted reference material (NIST, BAM, etc); the purity is assigned as 100 % minus impurities.

The combination of these two purity assignments leads to two independent traceability chains, and results in higher reliability of the CRM.

The starting material is **pretreated** (surface etching in case of metals, drying in case of salts) before weighing.



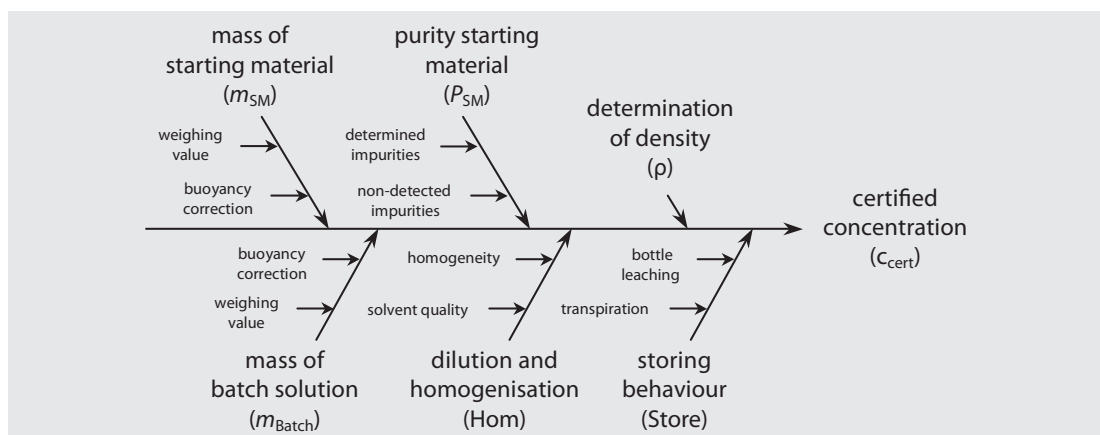
**Figure 2** Only well-characterised and high-purity starting materials are used for TraceCERT™ calibration standards (clockwise from upper left: copper, zinc, nickel and gold).

The **weighing** operation is a key step of production, leading to direct traceability to the SI unit kilogram. Gravimetric preparation using pure materials is a practical and, in most cases, the most accurate calculation of concentration units, through conversion of mass and mole fraction to mass fraction.

The weighted starting material is **dissolved** in high-purity acid and the stock solution is diluted with high-purity water until the calculated total mass of the final solution is reached. This gravimetric approach allows a highly precise adjustment of the final concentration of the calibration solution. **Homogenisation** of the solution is achieved by overhead tumbling of the container for several hours. In the final **bottling** step, contamination is prevented by working under clean room conditions. The bottled solution is then compared to a second, independent reference material.

### Certification

For each **TraceCERT** product, a detailed and comprehensive Certificate is supplied, either delivered with the product (for the ICP standards) or downloadable from the internet using product number and lot number (for the AAS and IC standards). An example of a certificate can be downloaded from the internet for every product ([sigma-aldrich.com/tracecert](http://sigma-aldrich.com/tracecert)). The certificates are in accordance with ISO Guide 31 and contain lot-specific information such as exact content, expiration date, traceability measurements, values of up to 70 impurities (only for the ICP standards) and a proper uncertainty calculation. The uncertainties of the TraceCERT standards are obtained by summing up all the contributing influence parameters (“bottom-up” approach). The influence parameters are shown in **Figure 3** (see next page).

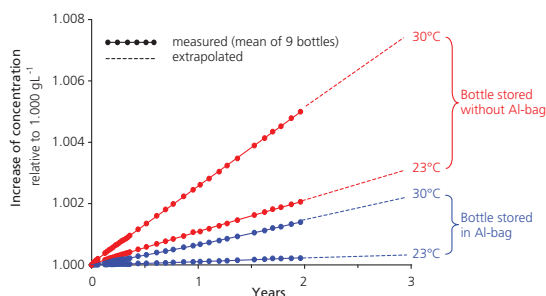


**Figure 3** Cause effect diagram of the preparation procedure of *TraceCERT*® reference materials. Only first and second order influence parameters are shown.

### Storage and stability

The ideal container for standard solutions is totally inert, will not absorb analyte, does not leach impurities into the solution, is impermeable towards the solvent and atmosphere and is easy to handle and store.

We found the most suitable material available today was high-density polyethylene (HDPE). Leaching of impurities into the solution and transpiration of the solvent through the container wall have been investigated comprehensively for this material (**Figure 4**).



**Figure 4** Transpiration behaviour of an aqueous calibration solution in a 100 mL HDPE bottle at different temperatures

The bottles of the *TraceCERT*® ICP standards are packed in sealed aluminium bags, preventing further transpiration of the solvent. Owing to this packaging technique, Sigma-Aldrich can guarantee the ambitious specification of 0.2 % uncertainty for the certified value over the entire shelf life of the standard.

### Product portfolio and custom standards

Only a few years after the introduction of the *TraceCERT* product line, it already comprises more than 120 products. The current portfolio includes an extensive range of single-element standard solutions for AAS and ICP (1000 mg/L), multi-element standards for ICP and standard solutions for ion chromatography (1000 mg/L).

We are continuously expanding the product range. The most recent product introductions are listed in **Table 1**. For the complete portfolio and for more information about the products please consult our website at [sigma-aldrich.com/tracecert](http://sigma-aldrich.com/tracecert). If you don't find the standard you need in our product list, you can solicit a quotation for a customised standard solution using our new online custom standards platform at [sigmaldrich.hil.ch](http://sigmaldrich.hil.ch)

Cat. No.	Description	Pack Size
43678	Lanthanum Standard for AAS	100 mL
42887	Niobium Standard for AAS	100 mL
53378	Cerium Standard for AAS	250 mL
68418	Scandium Standard for AAS	100 mL
75159	Thallium Standard for AAS	100 mL
04689	Titanium Standard for AAS	100 mL
73574	Zirconium Standard for AAS	250 mL
11523	Lanthanum Standard for ICP	100 mL
67913	Niobium Standard for ICP	100 mL
16734	Cerium Standard for ICP	100 mL
92279	Scandium Standard for ICP	100 mL
51873	Thallium Standard for ICP	100 mL
12237	Titanium Standard for ICP	100 mL
51244	Zirconium Standard for ICP	100 mL

**Table 1** Recently added Fluka-branded *TraceCERT* standard solutions (1000 mg/L)

# Monthly Savings Programme

## SAVE 30 %



### Get 30 % Discount for LC-MS Solvents

The LC-MS solvents are specifically formulated to have a low content of alkaline impurities, such as calcium, magnesium, potassium and sodium, which can interfere in the analysis by forming artefacts with the analyte.

The CHROMASOLV® LC-MS solvents are also run through specific UV-spectroscopic quality control tests to guarantee the traditional CHROMASOLV® specification.

More information is available on our website [sigma-aldrich.com/lc-ms](http://sigma-aldrich.com/lc-ms)

The following LC-MS solvents are available with a 30 % OFF discount.

Part No.	Brand	Description
34967	<b>Fluka</b>	Acetonitrile LC-MS CHROMASOLV®, ≥ 99.9 %
34966	<b>Fluka</b>	Methanol LC-MS CHROMASOLV®, ≥ 99.9 %
39253	<b>Fluka</b>	Wasser LC-MS CHROMASOLV®
34965	<b>Fluka</b>	2-Propanol LC-MS CHROMASOLV®, ≥ 99.9 %
34972	<b>Fluka</b>	Ethylacetate LC-MS CHROMASOLV®, ≥ 99.7 %
34986	<b>Fluka</b>	n-Hexane LC-MS CHROMASOLV®, ≥ 97 %
34999	<b>Fluka</b>	n-Heptane LC-MS CHROMASOLV®, ≥ 99 %

To take advantage of this monthly savings offer, please use promotion code **985**.  
Offer is valid until **December 31, 2009**.

## Sigma-Aldrich Introduces 1 Litre Bottles of Ultra-high Purity Acids

Michael Jeitziner, Market Segment Manager Analytical Reagents & Standards [mjeitziner@sial.com](mailto:mjeitziner@sial.com)

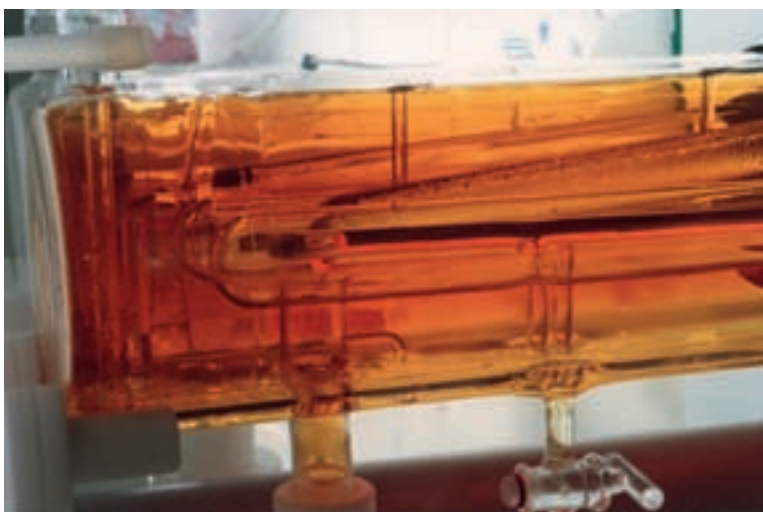
Sigma-Aldrich has recently introduced 1 L pack sizes in order to meet the requirements of routine analysis labs with a high throughput of samples. The new pack sizes have been extensively tested in order to avoid any leaching or matrix effects from the packaging material.

To maintain their high purity, **TraceSELECT**® Ultra products are supplied in Teflon® PFA (fluoropolymer) bottles. Water, hydrogen peroxide solution and ortho-phosphoric acid are supplied in specially pre-leached HDPE bottles. Recent process improvements have allowed us to reduce our impurity specifications in order to guarantee the lowest levels of trace impurities in our **TraceSELECT** Ultra products.

[sigma-aldrich.com/traceselect](http://sigma-aldrich.com/traceselect)

### Product table **TraceSELECT** Ultra reagents

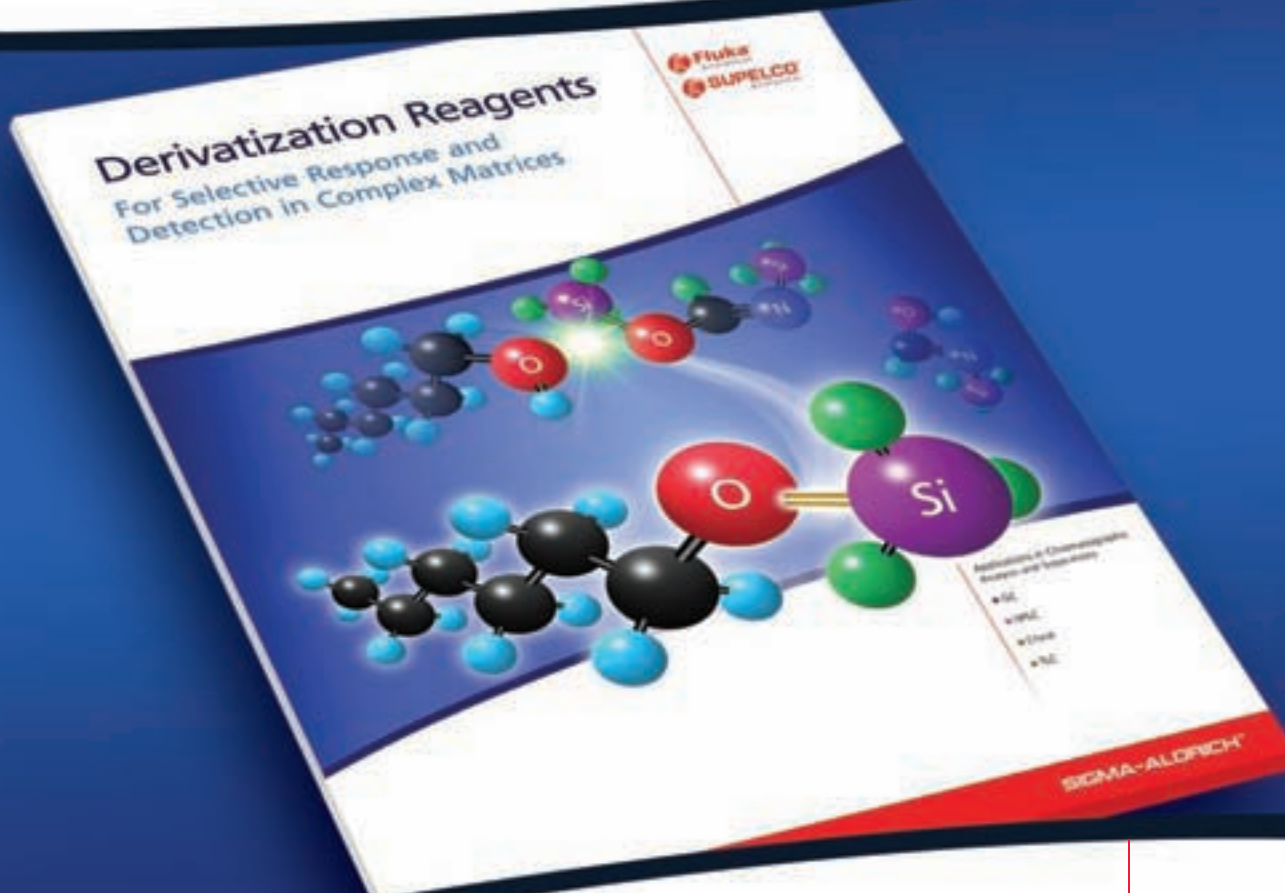
Cat. No.	Brand	Product Name	Specification	Pack Sizes
07692	Fluka	Acetic acid	≥ 99.0 %	250 mL, 1 L
16748	Fluka	Ammonium hydroxide solution	≥ 25 %	250 mL, 1 L
23828	Fluka	Hydrobromic acid	≥ 44 %	250 mL, 1 L
96208	Fluka	Hydrochloric acid	≥ 30 %	250 mL, 1 L
02658	Fluka	Hydrofluoric acid	≥ 49 %	250 mL, 1 L
16911	Fluka	Hydrogen peroxide solution	≥ 30 %	250 mL, 1 L, 5 L
02650	Fluka	Nitric acid	~ 65 %	250 mL, 1 L
12415	Fluka	Perchloric acid	67–72 %	250 mL, 1 L
64957	Fluka	Phosphoric acid	≥ 85 %	250 mL, 1 L
77239	Fluka	Sulphuric acid	≥ 95 %	250 mL, 1 L
14211	Fluka	Water		1 L



# Derivatisation Reagents

 **Fluka**  
Analytical

 **SUPELCO**  
Analytical



Many compounds must be derivatised to improve or even permit their analysis, in particular for GC, HPLC and TLC. Derivatisation reagents for nearly every type of compound and application can be found in our comprehensive product offering, which includes chiral derivatisation reagents for enantiomer analysis. Visit our website to view our products and obtain a copy of our new Derivatisation Reagents brochure.

[sigma-aldrich.com/derivatization](http://sigma-aldrich.com/derivatization)



## Polymeric Adsorbent Media

for chromatography and other applications

Shyam Verma, Market Segment Manager, Reagents & Chemicals [shyam.verma@sial.com](mailto:shyam.verma@sial.com)



Synthetic non-ionic polymer adsorbents are used as a chromatographic medium for a variety of separation/purification applications. Selection of a polymeric adsorbent generally depends upon its porosity (or degree of crosslinking), surface functionality and particle size.



These macroporous polymers, also termed as macroreticular polymers, are manufactured by crosslinking the base polymer, such as styrene with divinylbenzene (DVB) during the polymerisation process. The polymeric resins are produced in the form of spherical beads; each bead is made up of an agglomeration of a large number of very small microspheres that are clusters of smaller gel particles. Crosslinking provides suitable surface area, rigidity and mechanical strength. These adsorbents are porous and contain an internal surface that allows adsorption/desorption of a wide variety of species depending on their application environment.

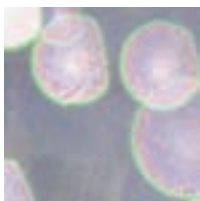


Polymers (especially styrene and acrylic) exhibit hydrophobic surface behaviour that allows effective adsorption and removal of organic impurities. In non-polar solvents, such as hydrocarbons, most adsorbents exhibit some polar or hydrophilic character resulting in adsorption of polar species. The surface of these adsorbent resins can be easily regenerated. The adsorbent resins are tailored for special pharmaceutical applications, removal of organic and aromatic compounds from aqueous streams, treatment of alkanol amines, and other sensitive analytical procedures.



Some common applications are:

- Removal of impurities from analytes, such as organics, bitters in juices, and colorants in aqueous or polar solvents, and sweeteners
- Chromatographic separation and purification
- Air monitoring and sample preparation
- Decoloration and desalting



Sigma-Aldrich offers a variety of polymeric adsorbents that are suitable for a wide spectrum of analytical and industrial applications. Different types of polymeric matrices offered are briefly discussed in the following columns. Additional information on these products can be obtained on our website ([sigma-aldrich.com](http://sigma-aldrich.com))



### 1. Styrenic- and Acrylic-based Adsorbent Resins

#### Products:

- Supelpak™-2 Resins (XAD®-2)
- Supelpak-2
- Supelite™ DAX-8
- Combigel™ XE-305
- Styrene-DVB

#### Application:

- Removal of aromatics (non-polar solute) and hydrophobic organics from water (polar solvent)
- Recovery of airborne semi volatiles, particularly polynuclear aromatic hydrocarbons (PAHs) and dioxins, stack gases, and ambient and indoor air monitoring

**Amberlite® XAD:** Styrene-DVB-based hydrophobic resins with some hydrophilic properties allow adsorption of species with some degree of polarity.

#### Products:

- XAD-2
- XAD-4
- XAD-7HP
- XAD-16 (XAD-16N)
- XAD-16HP
- XAD-1180N
- XAD-761
- XAD-7HP

#### Applications:

- Removal of sparingly soluble organic species from water
- Recovery of antibiotics such as Ceph C from complex fermentation broths in capture/concentration mode
- Decoloration and desalting

**Amberchrom® CG300:** This offers unique advantages in separation/purification procedures for biomolecule. It is ideal for discovery, development and production of biopharmaceutical compounds, especially in the front end capture, purification and desalting operational modes.

#### Applications:

Reversed phase chromatography for purification of proteins, peptides and oligonucleotides and similar molecules, maximising capacity and yield.

(continued on page 20)

## 2. Polyaromatic Resins – crosslinked polystyrenic matrix-based adsorbents.

**Applications:** Extraction of antibiotic intermediates from fermentation broth, separation of peptides or food additives, debittering of citrus juices etc.

**Sepabeads® SP207:** Brominated aromatic matrix exhibits enhanced hydrophobicity, high density and large capacity. It is especially useful with up-flow fluidised bed applications.

**MCI GEL® CHP20P:** This is suitable for reversed-phase chromatography applications, even with non-aqueous solvents.

**Dowex Optipore:** Highly cross-linked styrenic polymer adsorbents, insoluble in strong acid or base, or an organic solvent. Spherical beads with good physical strength offer:

- unique pore size distribution
- high surface area
- hydrophobic surface
- improved capacity for organics
- non-catalytic activity

### Products & applications:

**L493:** This exhibits high capacity and easy regeneration. It is a useful medium for removal of certain organics from water.

**V493:** The surface is hydrophobic and, therefore, suitable for applications with high-humidity air streams. It exhibits high capacity for a variety of volatile organic compounds (VOC) and hazardous air pollutants (HAP), and can be easily regenerated.

**V503:** Spherical beads with low mineral ash content. Recommended for removal of organics from humid air streams.

**SD-2:** An adsorbent with high-specific surface area and excellent mechanical, thermal and chemical stability. Recommended for decoloration and taste/odour removal in sweeteners.

**Diaion/Sepabeads/MCI GEL:** Hydrophobic spherical particles with fine pore structure.

## 3. Methacrylic Resins

**Diaion® HP2MG:** The hydrophilic surface is suitable for adsorption of polyphenols, surfactants and moderately polar compounds, such as antibiotics, colour bodies and aliphatic analytes.

## 4. Hydrophobic Interaction Media

**Sepharose®:** Beaded agarose

### Applications:

- Separation of proteins, especially after concentration by salt precipitation.
- Phenyl Sepharose is useful in low-pressure hydrophobic interaction or ion-exchange applications for proteins. The media with different ligands is used in the HiTrap HIC test kits for protein separation. For these media, the difference in hydrophobicity and ligand density influences binding, resolution, selectivity and analyte recovery.

**Toyopearl®:** Methacrylic polymer with a semi-rigid backbone structure with advantages, such as strong affinity, high capacity and recovery, excellent pressure/flow characteristics, stability and economical purification. It is also offered as a LABPAK Sampler.

## 5. Other Porous Polymers

**Applications:** Trap volatile and semi-volatile compounds up to 350 °C.

**Tenax® TA** – 2,6-Diphenylene oxide polymer with low affinity for water and methanol

**Supelpak-TA** – Refined (screened) Tenax TA

**Tenax GR** – Composite material of 30:70 graphite carbon and Tenax TA, respectively

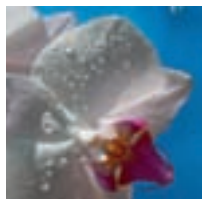
**Porapak™ N** – Divinylbenzene-ethylene glycol dimethacrylate polymer

**Chromosorb® 106** – Styrene/DVB polymer with high surface area

## HYDRANAL® K Reagent Line for Karl Fischer Titration

Determination of Water Content in Samples Containing Aldehydes and Ketones

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 Andrea Felgner, Product Manager Analytical Reagents [andrea.felgner@sial.com](mailto:andrea.felgner@sial.com)

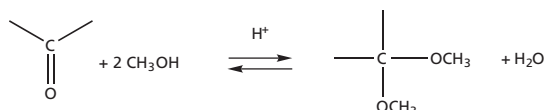


The HYDRANAL® K reagent line is designed to meet the needs of the analytical chemist, providing accurate water content determination by Karl Fischer (KF) titration in samples containing aldehydes and ketones. HYDRANAL Composite 5 K titrating agent prevents side reactions that occur during KF titration in aldehydes or ketones with methanol-containing reagents; these side reactions can compromise the water content determination. HYDRANAL Composite 5 K is a special formulation consisting of imidazole, sulphur dioxide, and iodine dissolved in diethylene glycol monoethyl ether. Three working media for use with HYDRANAL Composite 5 K are supplied by Sigma-Aldrich:

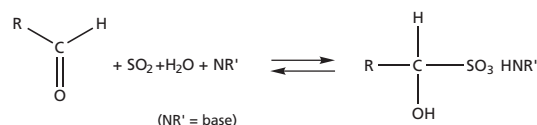
- (1) HYDRANAL KetoSolver – halogen-free
- (2) HYDRANAL Medium K – non-toxic
- (3) HYDRANAL Working Medium K – excellent dissolving properties

### Suppression of Side Reactions that lead to Erroneous Water Content Determinations

This line of reagents does not contain the conventional methanol component. Methanol has been omitted in order to prevent nucleophilic addition with aldehydes and ketones which leads to the formation of water (**Figure 1**). Water produced by this side reaction would result in elevated water content determinations and vanishing end points. The HYDRANAL K reagents are also designed to guard against bisulphite addition to aldehydes (**Figure 2**). This second side reaction consumes a portion of the water content in the neat specimen and results in erroneously low water content measurements. HYDRANAL Composite 5 K is a variant of HYDRANAL Composite 5, with a slightly slower reaction speed to maximise suppression of the bisulphite addition side reaction.



**Figure 1** Aldehydes and ketones undergo nucleophilic addition of methanol resulting in formation of acetal or ketal and water



**Figure 2** Bisulphite addition reaction consuming water

HYDRANAL K reagents are also suitable for amines, siloxanes and other titrations in methanol-free working media. HYDRANAL Medium K, HYDRANAL Working Medium K, and HYDRANAL KetoSolver can each act as the solvent for the determination of water in any substance where methanol can interfere with the titration and therefore must be avoided. The general utility of these reagents renders a universal applicability for KF titrations; they are also suited for easy-to-handle samples that do not interfere with the KF reaction.

### Distinctives of the Three HYDRANAL Working Media

The three working media are all free of methanol but each possesses unique properties for optimisation with particular samples. HYDRANAL KetoSolver is primarily 1-methoxy-2-propanol and is entirely free of halogenated hydrocarbons. It is used with HYDRANAL Composite 5 K for the KF titration of aldehydes and highly reactive ketones like cyclohexanone, trifluoroacetone and diacetyl. For certain less reactive ketones, HYDRANAL Composite 5 can be used as the titrating agent.

HYDRANAL Working Medium K has exceptional solvating power but must be designated as very toxic (T<sup>+</sup>) because it contains 2-chloroethanol. This hazardous component is omitted from HYDRANAL Medium K and replaced with less toxic alcohols in order to lower the toxicity rating to harmful (X<sub>n</sub>); this toxicity rating is due to the percentage of chloroform. Not only is the toxicity level lowered, but the revised formulation of HYDRANAL Medium K provides performance advantages with very reactive aldehydes such as propionaldehyde, butyraldehyde and crotonaldehyde. Capacity and accuracy are also improved for other compounds such as salicylaldehyde, acetylacetone, 2,4-dihydroxyacetophenone and 2-benzolpyridine.

Essentially, HYDRANAL Medium K is a full-fledged substitute for HYDRANAL Working Medium K, providing the same sample capacity, speed and accuracy. In addition, HYDRANAL Medium K offers important application, safety and transportation benefits. Because it is non-toxic, it not only improves workplace safety, but also reduces the amount of packaging material required for shipment and the associated handling and disposal of that packaging material.

(continued on page 22)

The Sigma-Aldrich laboratories compared HYDRANAL® Working Medium K and HYDRANAL Medium K and have reported these findings:

- Comparable sample capacity in 30 mL medium
- Comparable results for water content and standard deviation
- Comparable titration speed
- Comparable accuracy of recovery rate of added water after sample titration

#### Benefits of HYDRANAL Medium K over HYDRANAL Working Medium K

- Reduced toxicity for improved workplace safety while providing equal reactivity
- Performance advantages with very reactive aldehydes
- Less waste of packaging material

#### Coulometric Karl Fischer Titration in Ketones

For the coulometric water determination in ketones, Sigma-Aldrich offers specially designed HYDRANAL Coulomat reagents: Coulomat AK and Coulomat CG-K.

HYDRANAL Coulomat AK is an analytic reagent for the coulometric determination of water in ketones. It contains imidazole, sulphur dioxide, and iodide dissolved in a suitable solvent mixture, and has a capacity of approximately 100 mg of water per 100 mL. HYDRANAL Coulomat AK can also be used as a single reagent for coulometry without diaphragm. HYDRANAL Coulomat CG-K is the corresponding catholytic reagent. It does not contain halogenated hydrocarbons. The water capacity of 5 mL HYDRANAL Coulomat CG-K is 100 mg.

The total sample volume used with one charge of coulometric reagents should not exceed 20 mL (as liquid sample or as dissolved solid sample). Because of ongoing side reactions, larger sample amounts can lead to high instrument drift, causing vanishing end points and erroneous results of the titration.

#### Application Example: Determination of Water in Acetone

The water content in acetone can be determined using both volumetric and coulometric KF titration techniques:

##### • Volumetric titration

Add 30 mL HYDRANAL Medium K to the titration vessel and titrate to dryness with HYDRANAL Composite 5 K. Add approximately 5 mL of the acetone sample, weighed by difference, to the vessel. Titrate the water content with HYDRANAL Composite 5 K.

##### • Coulometric titration

Add 100 mL HYDRANAL Coulomat AK to the analyte compartment of the titration vessel and 5 mL HYDRANAL Coulomat CG-K to the catholyte compartment. After starting the titrator, the cell is dried automatically. When the drift is stable, add approximately 0.5 g of the sample, weighed by difference, to the cell.

#### General Recommendations for KF Titration in Aldehydes and Ketones

##### • Aldehydes

Short-chain aldehydes show a strong tendency to form acetals (along with water formation). Aromatic aldehydes tend to undergo the bisulphite addition. To overcome these potential interferences, we recommend using relatively small samples and titrating rapidly to suppress the formation of acetals and bisulphite compounds. Coulometry is not advised, but if it is necessary, the sample volume should be very small. For example, for water determination in aromatic aldehydes, the sample volume must be less than 0.5 mL. Aliphatic aldehydes, like acetaldehyde, are highly reactive and tend to rapidly form acetals. Only volumetric titration with methanol-free K reagents is recommended and the amount of sample should be kept to a minimum.

##### • Ketones

Ketones have a tendency to form ketals while at the same time forming water. Cyclohexanone and acetone react rapidly, while long-chain ketones and aromatic-substituted ketones show slower reaction rates. Reactive ketones are titrated with methanol-free K reagents. The coulometric determination is possible by using HYDRANAL Coulomat AK and Coulomat CG-K reagents. The sample size for coulometric measurements should not exceed 1 mL, for very reactive ketones like cyclohexanone a maximum of 0.5 mL per sample is recommended.

Substance	Application Number
1,2-Cyclohexanedione	306
2-Dimethylaminomethyl cyclohexanone (DMC base)	518
Butanal (N-Butyraldehyde and iso-Butyraldehyde)	248
Crospovidone (Cross-linked homopolymer of 1-vinylpyrrolidone-2-on)	472
Dexpanthenol	507
Diacetyl (2,3-Butanedione)	496
Ethosuximide (3-Ethyl-3-methyl-2,5-pyrrolidindion)	510
Glutardialdehyde 50 %	273
Glyoxal solution 40 %	267
Glyoxylic acid methylester methylhemiacetal	261
Hexafluoroacetone sesquihydrate	154
N-Formylmorpholine (4-Morpholine carboxaldehyde)	392
N,N-Dimethyl formamide	424

**Table 1** Available KF application reports for selected aldehydes and ketones

Cat. No.	Brand	Description	Package Size
34816	Fluka	HYDRANAL Composite 5 K	500 mL, 1 L, 2.5 L
34805	Fluka	HYDRANAL Composite 5	500 mL, 1 L, 2.5 L
34738	Fluka	HYDRANAL KetoSolver	500 mL, 1 L
34698	Fluka	HYDRANAL Medium K	1 L
34817	Fluka	HYDRANAL Working Medium K	1 L
34820	Fluka	HYDRANAL Coulomat AK	500 mL
34821	Fluka	HYDRANAL Coulomat CG-K	50 mL

**Table 2** Product listing HYDRANAL K reagents

To obtain application reports and more information on HYDRANAL® K reagents as well as our other high-quality HYDRANAL reagents for pyridine-free water determination by KF titration, please visit our website [sigma-aldrich.com/hydranal](http://sigma-aldrich.com/hydranal) or contact our HYDRANAL laboratories.

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