

Analytix

Issue 4 • 2010



Properties and Detection of *Clostridium Perfringens*



- Detection of *Clostridium perfringens*
- Phytopharmaceutical Standards
- Ultra-pure Digestion Reagents
- Ion Sensor Materials
- Headspace Grade Solvents
- Water Content in Candies
- Pesticide Residue Analysis

Microbiological Control



Jvo Siegrist
Product Manager Microbiology

Dear Colleague,

How fitting it is that this issue's cover focuses on a microbiology topic, as this is a timely subject comprising one of the major control parameters for food and beverage safety. It starts with the quality of the raw material, such as milk and meat, extends to the various processing steps, and ends in the quality of the final product. Depending on the nature of the sample, the possible pathogens or spoiling contaminants are determined. For example, if water is required in the processing and its source is surface water, it must be checked for the presence of coliforms such as *Salmonella*, *Clostridium perfringens*, *Pseudomonas* and some other germs, to avoid the introduction of microbial contamination. The classical microbiology approach with media, biochemical testing and, microscopy is the base supported today by immunological methods like ELISA and molecular biological methods like PCR and sandwich hybridization techniques.

As usual, we once again present interesting topics in this issue about microbiology and other analytical topics. I hope you enjoy reading it!

Best regards,

A handwritten signature in black ink, appearing to read 'Jvo Siegrist', written over a white background.

Jvo Siegrist
Product Manager Microbiology
ivo.siegrist@sial.com

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Feature Article

4 ***Clostridium perfringens*, their Properties and their Detection**

Role of *Clostridium perfringens* and their detection, identification, and differentiation

Microbiology

6 **Baird Parker Agar**

Detection and differentiation of *Staphylococcus aureus* on Baird Parker Agar

Standards

8 **NEW Analytical Standards for Natural Products**

Spectroscopy

10 **High Purity Digestion Reagents for Ultra-Trace Analysis**

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

Chemical Sensors

11 **NEW SELECTOPHORE® Brochure Available**

Chromatography

12 **Headspace Grade Solvents**

High purity for "OVIs" analysis

13 **LC-MS Solvents and Reagents**

15 **Dispersive SPE for EN15662: 2008 "QuEChERS" Method**

Quick and simple sample cleanup for determination of pesticides in foods of plant origin

Titration

17 **Determination of Water Content in Candies**

HYDRANAL® Applications: Caramel mass, Jelly sweets, Fruit gum, and Fondant

20 **New Tips and Tricks for Volumetric Titration**

Visit our website to find out more!

21 **Increasing Reliability and Value of Analytical Results**

Sigma-Aldrich achieves accreditation according to ISO/IEC 17025 for Water Standards and Karl Fischer Reagents

Monthly Savings Program

22 **45% Off High-quality titration reagents**

New Product Corner

23 **New High-Purity Standards for Pesticide Residue Analysis**

23 **New Veterinary Drug Standards**

Clostridium perfringens, their Properties and their Detection

Role of *Clostridium perfringens* and their detection, identification, and differentiation

Jvo Siegrist, Product Manager Microbiology ivo.siegrist@sial.com

Clostridia are relatively large, Gram-positive, rod-shaped bacteria that can undergo only anaerobic metabolism. Most Clostridia cannot grow under aerobic conditions and can even be killed by exposure to O₂; however, they form endospores that are able to survive long periods of exposure to air and other adverse environmental conditions. The natural sources of clostridia are anaerobic habitats with organic nutrients, particularly soils, aquatic sediments, and the intestinal tracts of animals. Their fermentation of organic compounds, such as sugars, produces large amounts of CO₂ and H₂ as well as volatile organic compounds like acetic and butyric acid, acetone, and butanol. Metabolism of substrates like amino acids and fatty acids results in foul-smelling degradation products. Clostridia also have an extended range of extracellular enzymes that degrade large biological molecules in the environment into fermentable compounds. Although there are non-pathogenic clostridia, this genus produces some of the most potent biological toxins. The three particularly lethal members of this group are *C. perfringens*, which is responsible for cooked meat-associated food poisoning and wound and surgical infections that lead to gas gangrene, *C. tetani*, which is responsible for deadly tetanus infections, and *C. botulinum*, which causes botulism.

Clostridium perfringens is found in undercooked or improperly sterilized canned foods (germination of endospores) and in water (surface water). The natural contamination source is human and animal feces mainly transmitted into food by water. *C. perfringens* produces an extensive range of invasins and exotoxins. The enterotoxins cause the undesirable, mostly meat-associated, food poisoning and wound and surgical infections that lead to gas gangrene.



Figure 1 Scanning electron micrograph of *C. perfringens* grown on a silicon wafer (source: S. Melville, Department of Biological Sciences, Virginia Tech University)

C. perfringens plays a subsidiary role in water examination [4]. Clostridia are spore builders and are resistant to heating, chlorination and other stress factors. In contrast to vegetative cells like coliforms (*E. coli*, enterococci), which are less resistant, *C. perfringens* has the advantage of surviving longer [6]. Therefore, while a fecal contamination is detected mostly by coliforms as an indicator, which could disappear after a processing step, *C. perfringens* remains present. The organism is not a hazard in water; rather, it is problematic when the water comes in contact with food.

In consideration of the aforementioned facts, it is obvious that detection and identification of *C. perfringens* is an important step towards the control and eradication of this potent pathogen. Some characteristic enzymes of *C. perfringens* are hemolysins (β-hemolysis), lecithinase, extracellular proteases, lipases (phospholipase-C), collagenase, hyaluronidase, saccharolytic enzymes and enzymes to reduce sulphite to sulphide. These enzymes are also used as detection and differentiation targets. It is also notable that *C. perfringens* is a non-motile bacterium and it is the most important of the sulphite-reducing clostridia. Also, *C. perfringens* normally grows at 44 °C, whereas some other clostridia are inhibited at this temperature. This property is used in ISO methods to give the medium more selectivity [4].

For detection of *C. Perfringens*, mCP and TSC agar have been recommended [4, 5]. However, there are problems associated with each of these media. The mCP method is very onerous for routine screening and bacterial colonies cannot be used for further biochemical testing. In addition, the recovery of *C. perfringens* was rejected by ISO in favor of methods based on TSC agar [6]. TSC detects all sulphite-reducing clostridia, however, and not only *C. perfringens*; in some cases, exces-

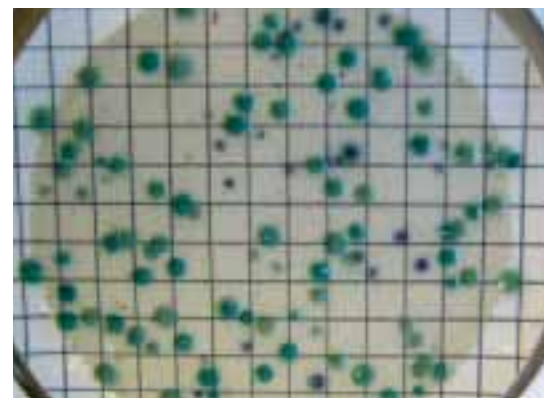


Figure 2 CP ChromoSelect Agar (*C. perfringens* appears as green colonies)

sive blackening of the agar frustrated counting of the lower dilutions. On the other hand, if the contamination was too high, TSC did not consistently produce black colonies and, as a consequence, the colonies' white color provided false negative results.

To prevent this problem, Sigma-Aldrich has introduced a new medium called **CP ChromoSelect Agar**, a chromogenic media from the new generation. Studies have shown that this medium produces counts that are not significantly different from those obtained from other media. The identification of typical and atypical colonies isolated from all media has demonstrated that CP *ChromoSelect* Agar (see **Figure 1**) was more specific to *C. perfringens* in water samples than mCP (see **Figure 2**) and TSCF agar.

To keep *Clostridium perfringens* under control, Sigma-Aldrich has developed a broad range of selective media (**Table 1**), tests (**Tables 2 and 3**) and anaerobic equipment (**Table 4**) for the detection, identification and differentiation of clostridia.

Further details about the media, tests, and equipment for clostridia and many other pathogens can be found on our website sigma-aldrich.com/microbiology

References:

- [1] Sherris Medical Microbiology, 4th ed., K.J. Ryan and C.G. Ray Editors, McGraw Hill, 2004.
- [2] Wells, C. L.; Wilkins, T. D., Clostridia: Sporeforming Anaerobic Bacilli, In *Baron's Medical Microbiology*, 4th ed., S. Baron Editor, Univ. of Texas Medical Branch, 1996.
- [3] Elmer Koneman, W. et al Color Atlas and Textbook of Diagnostic Microbiology, 5th ed., Lippincott Williams & Wilkins, 1997.
- [4] ISO 6461 Part 2. Water Quality – detection and enumeration of *Clostridium perfringens*. (Revision of ISO 6461-2: 1986).
- [5] Council Directive 98/83/EC, Relating to the Quality of Water Intended for Human Consumption, Official Journal of the European Communities, p.L330-32-L330/53 (1998).
- [6] National Standard Method, Enumeration of *Clostridium perfringens* by membrane filtration, issue no 3.1, reference no W5i3.1 (2005).

Test for Clostridia Diagnostics	Cat. No.	Description
Aminopeptidase Test	75554	For the detection of L-alanine-aminopeptidase which is found primarily in Gram-negative microorganisms.
Kovac's Reagent for indoles (isoamyl alcohol as solvent)	67309	For the differentiation of clostridia. In the presence of oxygen, some bacteria are able to split tryptophan into indole and α-aminopropionic acid. <i>Clostridium perfringens</i> is, for example, an indole-negative bacterium.
Kovac's Reagent for indoles (butanol as solvent)	60983	
Mannitol Disks	94438	Used to differentiate bacteria on the basis of mannitol fermentation.
Nitrate Reagent Disks	08086	Used to detect an organism's ability to reduce nitrate.
Tributyryn-Strips	75744	The test principle is hydrolysis of tributyrin. This reaction causes color change of acidobasic indicator.

Table 2 Tests for identification and differentiation of Clostridia

Cat. No.	Description
17170	AEA Sporulation Broth (Base), modified
15997	Brewer Agar
27544	Clostridial Differential Broth
27546	Clostridial Nutrient Medium
12398	CP <i>ChromoSelect</i> Agar
G0289	Gelatin Iron Agar
61348	Lactose Gelatin Medium (Base)
75605	m-CP Agar Base
14305	Motility Nitrate Medium
70151	Nutrient Gelatin
39727	Perfringens Agar Base
91365	Reinforced Clostridial Agar
85627	SPS Agar
17231	SPS Agar, modified
86128	Sulphite Iron Agar
T3938	Tryptone Soya Broth without Dextrose
93745	TSC Agar
93735	TSN Agar

Table 1 Common and new media for *Colostridium perfringens* (a complete list of clostridia media can be found on sigma-aldrich.com/clostridia)

Gram Stain	Cat. No.
Gram Staining Kit	77730
Gram's Crystal Violet Solution	94448
Gram's Decolorizer Solution	75482
Gram's Fuchsin Solution	87794
Gram's Iodine Solution	90107
Gram's Safranin Solution	94635

Table 3 Gram staining kit and single solutions

Anaerobic Equipment	Cat. No.
Anaerobe atmosphere generation bags	68061
Anaerobe indicator test	59886
Anaerobic jar	28029

Table 4 Anaerobic equipment

Baird Parker Agar

Detection and differentiation of *Staphylococcus aureus* on Baird Parker Agar



Figure 1
Ultramicrograph of
Staphylococcus aureus



Figure 2
Microscopic image of
Staphylococcus aureus

Baird Parker Agar is used for the isolation and differentiation of coagulase-positive staphylococci in food and pharmaceuticals according to the Baird Parker method.

As a nitrogen source for the organism, casein peptone and meat extract are added to the medium. Yeast extract provides nitrogen as well as other important nutrients like vitamin B₁₂ complex. The medium contains lithium and tellurite, which inhibit most of the contaminating microflora, while glycine and pyruvate enhance *Staphylococci* growth. *Staphylococci* can reduce tellurite to telluride, which results in grey to black coloration of the colonies. With the addition of egg yolk, the medium becomes yellow, slightly opaque. A clear halo develops around colonies from coagulase positive *Staphylococcus aureus*, and upon further incubation may produce an opaque zone due to an egg yolk – lecithinase reaction (lypolytic activity). Grey-black colonies and a halo on this medium are presumed to be indicative of coagulase positive staphylococci, as a high correlation between coagulase test and lipolytic activity was found. *Staphylococcus aureus* and some strains of *Staphylococcus saprophyticus* (Shaw et al.[5]) may show both of these characteristics, but they are easily distinguished from each other by the different times at which the halo develops.

Baird Parker Agar with Rabbit Plasma Fibrinogen can be used for the detection of coagulase activity. Coagulase positive organisms appear as grey to black colonies because of the tellurite reduction and as an opaque halo, due to the conversion by coagulase from fibrinogen to fibrin. Only plates with less than 100 characteristic colonies should be counted. Use of this medium eliminates the need for an additional coagulase test.

Jvo Siegrist, Product Manager Microbiology ivo.siegrist@sial.com

Composition:

Ingredients	Grams/Litre
Casein Peptone	10.0
Meat Extract	5.0
Yeast Extract	1.0
Lithium Chloride	5.0
Glycine	12.0
Sodium pyruvate	10.0
Agar	15.0
Final pH (at 37 °C) 6.8 ± 0.2	

Description	Cat. No.
Baird Parker Agar	11705
Egg yolk tellurite emulsion	75208
Rabbit Plasma Fibrinogen Supplement	05939

Table 2 Media and supplements

References:

- [1] A.C. Baird Parker, An improved diagnostic and selective medium for isolating coagulase-positive staphylococci. *J. Appl. Bacteriol.* 25, 12 (1962).
- [2] E.H. Lennette, E.H. Spaulding, J.P. Truant, *Manual of Clinical Microbiology*. 2nd ed. Washington D.C.: American Society for Microbiology (1974).
- [3] Jean F. Mac Faddin, *Media for Isolation-Cultivation Identification-Maintenance of Medical Bacteria*. Vol. I, Baltimore, MD.: Williams & Wilkins (1985).
- [4] A. Nisakanen, M. Alto, *Appl. Envir. Microbiol.* 35, 1233 (1978).
- [5] S. Shaw, M. Scott, T. Cowan, *Gen. Microbiol.* 5, 1010-1023 (1957).
- [6] ISO 6888-1:1999; Microbiology of food and animal feeding stuffs – Horizontal method for the enumeration of coagulase-positive staphylococci (*Staphylococcus aureus* and other species) – Part 1: Technique using Baird-Parker agar medium.
- [7] ISO 6888-2:1999; Microbiology of food and animal feeding stuffs – Horizontal method for the enumeration of coagulase-positive staphylococci (*Staphylococcus aureus* and other species) – Part 2: Technique using rabbit plasma fibrinogen agar medium.
- [8] ISO 6888-3:2003; Microbiology of food and animal feeding stuffs – Horizontal method for the enumeration of coagulase-positive staphylococci (*Staphylococcus aureus* and other species) – Part 3: Detection and MPN technique for low numbers.

Organisms (ATCC)	Color of Colony	Growth	Lecithinase	Coagulase
<i>Staphylococcus aureus</i> (25923)	grey-black shiny	+	+	+
<i>Staphylococcus aureus</i> (6538)	grey-black shiny	+	+	+
<i>Staphylococcus epidermidis</i> (12228)	black	+/-	-	-
<i>Micrococcus luteus</i> (10240)	very small in shades of brown-black	+/-	-	-
<i>Bacillus subtilis</i> (6633)	dark brown matt	+/-	-	-
<i>Escherichia coli</i> (25922)	large brown black	+/-	-	-
<i>Proteus mirabilis</i> (12453)	brown-black	+	-	-

Table 1 Culture characteristics after 24 – 48 hours at 35 °C.



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NEW Analytical Standards for Natural Products

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



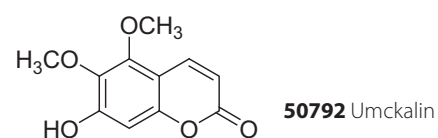
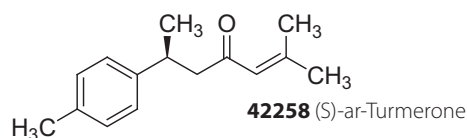
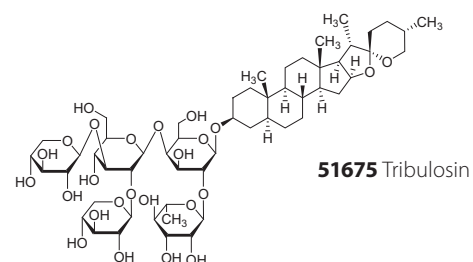
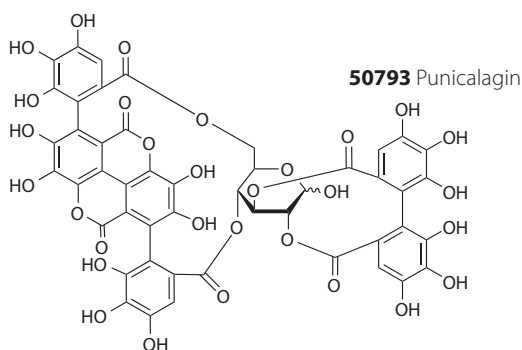
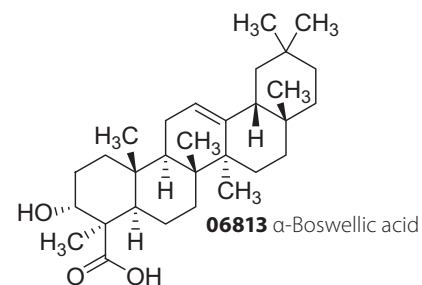
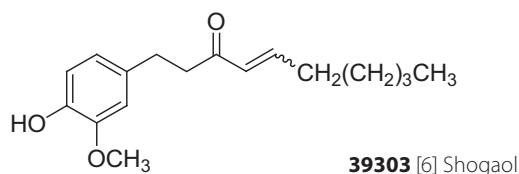
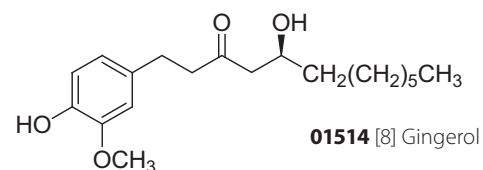
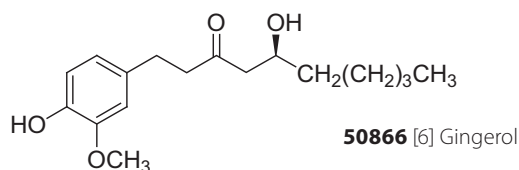
Recently, several new analytical standards for natural products have been added to our portfolio. These substances are constituents of medicinal plants that are commonly used in traditional Chinese and Ayurvedic medicine.

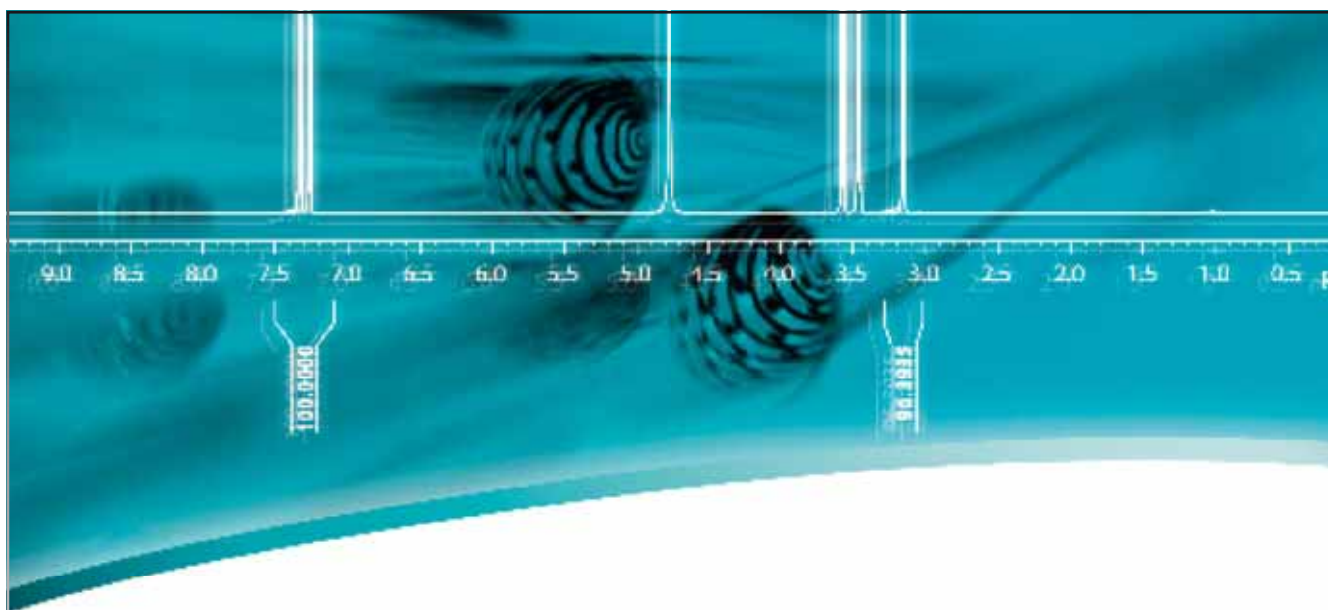
Gingerols are active ingredients and are responsible for the pungent taste of ginger. Shogaol is formed from Gingerol during the drying process. The Shogaol:Gingerol ratio is therefore often used as an indication of product quality.

Among the new standards are furthermore α -Boswellic acid (active ingredient of the *Boswellia* plant), Punicalagin (active ingredient of pomegranate), Tribulosin (active ingredient

of *tribulus terrestris*), ar-Turmerone (active ingredient of turmeric oil, derived from curcuma) and Umckalin (active constituent in root extracts of *Pelargonium* plants).

Brand	Cat. No.	Description	Pack Size
FLUKA®	06813	α -Boswellic acid	10 mg
FLUKA	50866	[6]-Gingerol	10 mg
FLUKA	01514	[8]-Gingerol	10 mg
FLUKA	50793	Punicalagin	10 mg
FLUKA	39303	[6]-Shogaol	10 mg
FLUKA	51675	Tribulosin	10 mg
FLUKA	42258	(S)-ar-Turmerone	10 mg
FLUKA	50792	Umckalin	10 mg





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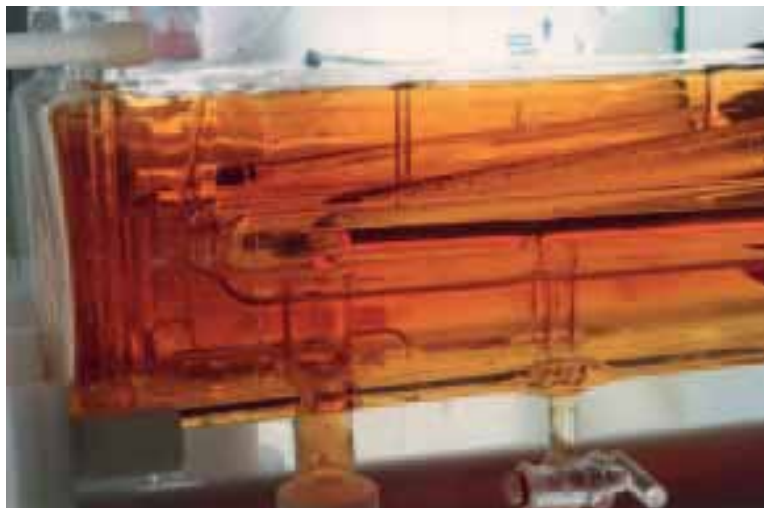


High Purity Digestion Reagents for Ultra-Trace Analysis

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

Sample preparation for trace analysis requires reagents of the highest purity. Our **TraceSELECT®** Ultra acids for ultra-trace analysis at ppb and even ppt levels are produced by sub-boiling distillation. Sub-boiling is recognized as the best way to obtain high-purity acids with the lowest blank values for ultra-trace analysis. The technique is based on the evaporation of liquid by infrared heating at the surface. It avoids violent boiling and the formation of liquid aerosols that can be transported with the distillate.



To maintain their high purity, **TraceSELECT** Ultra products are supplied in Teflon® PFA (fluoropolymer) bottles. Water, hydrogen peroxide solution, TMAOH, 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.

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

Cat. No.	Brand	Product Name	Specification	Pack Size
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
64957	Fluka	Phosphoric acid	≥ 85%	250 mL, 1 L
77239	Fluka	Sulphuric acid	≥ 95%	250 mL, 1 L
14213	Fluka	Tetramethylammonium hydroxide solution (TMAOH)	~ 25% in H ₂ O	250 mL
14211	Fluka	Water		1 L

Product Table **TraceSELECT** Ultra reagents

NEW SELECTOPHORE® Brochure Available

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

Fluka®'s new SELECTOPHORE brochure with 400 citations includes 600 ionophores and auxiliary reagents for the preparation of ion-selective electrodes (ISE). A vast variety of different chemicals, ionophores, and other products required to construct electrodes for electrochemical analyses (e. g. potentiometric, conductance), as well as optodes for optical measurements, can be found in this 184-page handbook.

New ionophores with improved properties

The performance of chemical sensor devices depends on three "S" factors: Selectivity, Sensitivity, and Stability. The ionophore plays the most critical role in optimising these factors. Sigma-Aldrich has successfully developed and introduced many new ionophores with improved characteristics over previously existing ones. The Fluka-branded ionophores are application tested using the electrode body IS-561, Fluka 45137. Two examples of ionophores with improved properties are listed below.

Calcium ionophore V, Selectophore function tested, Fluka 21203 (K23E1; 10,19-Bis[(octadecylcarbamoyl)methoxyacetyl]-1,4,7,13,16-pentaoxa-10,19-diazacycloheptacosane)

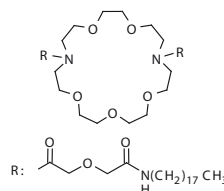


Figure 1 Calcium ionophore V

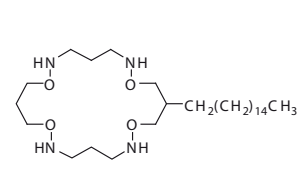


Figure 2 Nitrate ionophore VI

This calcium-selective ionophore obtains superior characteristics over all commercially available calcium ionophores. The PVC membrane stability is improved due to the high lipophilicity of the octadecane groups ($\log P_{TLC} = 14.6$). This ionophore has a linear response between $7 \cdot 10^{-7}$ M and $6 \cdot 10^{-2}$ M calcium ion.

Nitrate ionophore VI, Selectophore function tested, Fluka 07295 (9-Hexadecyl-1,7,11,17-tetraoxa-2,6,12,16-tetraazacycloicosane)

Nitrate ionophore VI is the best nitrate-selective ionophore available. The PVC membrane stability is improved by this ionophore due to the high lipophilicity of the hexadecane group ($\log P = 8.3$ (calc.)). The membrane obtains a linear response between $5 \cdot 10^{-6}$ M and 0.1 M nitrate ion.

Selectophore Brochure 2010

Basic Principles, Technical Instructions, Applications & Product Listings

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High purity for "OVIs" analysis

Shyam Verma, Product Manager Analytical Reagents shyam.verma@sial.com

Michael Kiselewsky, Product Manager Chromatography Reagents michael.kiselewsky@sial.com

Static headspace GC (SH-GC), a commonly used technique in the analysis of organic volatile impurities (OVIs), concentrates volatile analytes to allow their analysis free from sample matrix. Samples to be analyzed by SH-GC are dissolved in a suitable solvent that must allow for sufficient sensitivity of the analytes of interest in the headspace. Also, the vapor pressure of the dissolution solvent itself should be sufficiently low so it will not affect detection of OVI analytes by "flooding" the headspace.

USP <467> and European Pharmacopoeia (EP) methodologies list procedures for both water-soluble and water-insoluble samples [1-2]. For water-insoluble samples, USP <467> designates the use of the solvents dimethylsulphoxide (DMSO) and dimethylformamide (DMF). Other dissolution solvents that have been found to be useful for headspace analysis of water-insoluble samples include dimethylacetamide (DMAC) and 1,3-dimethyl-2-imidazolidinone (DMI); the latter is described for use in EP Method 2.4.24 [2]. These solvents have significantly lower vapor pressure than many other high boiling-point organic compounds and they elute later than most OVI analytes in chromatographic analyses.

Solvent purity is essential to avoid extraneous peaks in the chromatographic analysis, and prevent interference with the analytes of interest. A recent publication [3] on our joint work with Genentech, Inc. reported chromatogram results of comparing the headspace grade to the convention organic synthesis grade solvents. The cleanliness of the headspace grade solvents, as well as their compatibility for use in the analysis of the OVIs listed in United States Pharmacopoeia (USP) Method <467>, European Pharmacopoeia (EP) Method 2.4.24, and the International Conference on Harmonization (ICH) guidelines, were demonstrated. This publication documented the following benefits of Fluka®'s headspace grade solvents.

- Headspace grade solvents produced cleaner blanks.
- Headspace grade solvents did not produce any major interference peaks in the elution range of the target analytes.
- Headspace grade DMSO, DMF, DMAC and DMI solvents evaluated were suitable for the analysis of OVIs by HS-GC.

References

- [1] United States Pharmacopoeia (USP), 31st Edition (2008), <467> Residual Solvents.
- [2] European Pharmacopoeia (EP) 5.0, Vol.1, (2004), 2.4.24 Identification and Control of Residual Solvents.
- [3] A. Quiroga, M. Dong, K. Stenerson, S. Verma, The Utility of Headspace Grade Solvents in the Analysis of Organic Volatile Impurities. Supelco® Publication T409180, November 2009.

Featured Products

Description	Boiling Point	Pack Size	Cat. No.
N,N-Dimethylacetamide	166 °C	1 L	44901
Dimethyl sulphoxide	189 °C	1 L	51779
N,N-Dimethylformamide	153 °C	1 L	51781
1,3-Dimethyl-2-imidazolidinone	225 °C	100 mL, 1 L	67484
Water	100 °C	1 L	53463

All products are Fluka puriss p.a.



LC-MS Solvents and Reagents



Michael Kiselewsky, Product Manager Chromatography Reagents michael.kiselewsky@sial.com



LC-MS is rapidly becoming a routine fixture in today's well-equipped analytical laboratory. Accompanying the increased use of LC-MS are the instrumental, chemical, and database methods aimed at increasing the sensitivity, specificity, and speed of analysis for this invaluable technique. New ion sources, high-resolution LC systems, and rapid mass spectrometers with enhanced ion optics and detectors have lowered the limits of detection, thereby raising the bar on the purity expectations of chemicals used for sample preparation, mobile phases, and post-column additives.

Alkali ions, plasticizers, and surfactants are particularly problematic as they are widespread and interfere strongly with LC-MS by causing higher background noise and the formation of adducts. Because of the integral part that chemistry plays in a successful LC-MS analysis, Sigma-Aldrich has developed and introduced many solvents, additives, and reagents that are specifically designed to meet the requirements of high-purity and consistency.

Solvents

Brand	Product	Name	Description	Pack Size
Fluka®	34967	Acetonitrile	LC-MS CHROMASOLV®, ≥99.9%	250 mL, 1 L, 2.5 L, 6x1 L, 4x2.5 L, 20 L, 45 L
Fluka	34966	Methanol	LC-MS CHROMASOLV, ≥99.9%	1 L, 2.5 L, 6x1 L, 4x2.5 L, 20 L
Fluka	39253	Water	LC-MS CHROMASOLV	1 L, 20 L
Fluka	34965	2-Propanol	LC-MS CHROMASOLV, ≥99.9%	1 L, 2.5 L, 6x1 L, 4x2.5 L
Fluka	34972	Ethyl acetate	LC-MS CHROMASOLV, ≥99.7%	1 L, 2.5 L
Fluka	34986	Hexane	LC-MS CHROMASOLV, ≥97%	1 L, 2.5 L
Fluka	34999	Heptane	LC-MS CHROMASOLV, ≥99%	1 L, 2.5 L

Blends

Brand	Product	Name	Description	Pack Size
Fluka	34978	Water with 0.1% trifluoroacetic acid	LC-MS CHROMSOLV	2.5 L
Fluka	34976	Acetonitrile with 0.1% trifluoroacetic acid	LC-MS CHROMASOLV, ≥99.5%	2.5 L
Fluka	34974	Methanol with 0.1% trifluoroacetic acid	LC-MS CHROMASOLV, ≥99.5%	2.5 L
Fluka	34673	Water with 0.1% formic acid	LC-MS CHROMSOLV	2.5 L
Fluka	34677	Water with 0.1% formic acid and 0.01% trifluoroacetic acid	LC-MS CHROMSOLV, contains 0.093-0.107% (w/w) formic acid as additive	2.5 L
Fluka	34668	Acetonitrile with 0.1% formic acid	LC-MS CHROMASOLV, ≥99.5%	2.5 L
Fluka	34676	Acetonitrile with 0.1% formic acid and 0.01% trifluoroacetic acid	LC-MS CHROMASOLV, ≥99.5%	2.5 L
Fluka	34671	Methanol with 0.1% formic acid	LC-MS CHROMASOLV, ≥99.5%	2.5 L
Fluka	34675	Water with 0.1% acetic acid	LC-MS CHROMSOLV	2.5 L
Fluka	34678	Acetonitrile with 0.1% acetic acid	LC-MS CHROMASOLV, ≥99.5%	2.5 L
Fluka	34672	Methanol with 0.1% acetic acid	LC-MS CHROMASOLV, ≥99.5%	2.5 L
Fluka	34674	Water with 0.1% ammonium acetate	LC-MS CHROMSOLV	2.5 L
Fluka	34669	Acetonitrile with 0.1% ammonium acetate	LC-MS CHROMSOLV, ≥98% acetonitrile basis (GC), 0.093-0.107% (w/v) ammonium acetate	2.5 L
Fluka	34670	Methanol with 0.1% ammonium acetate	LC-MS CHROMASOLV, ≥99.5%	2.5 L
Fluka	19182	Acetonitrile 50%, Water 47.5% and Trifluoroacetic acid 2.5%	LC-MS CHROMASOLV, 50% acetonitrile basis (GC)	250 mL

Eluent Additives

Brand	Product	Name	Description	Pack Size
Fluka	40967	Trifluoroacetic acid	puriss.p.a., eluent additive for LC-MS, ≥99.0%	10x1 mL, 5x10 mL, 1 L
Fluka	56302	Formic acid	puriss.p.a., eluent additive for LC-MS, ~98%	10x1 mL, 50 mL, 1 L
Fluka	49199	Acetic acid	puriss.p.a., eluent additive for LC-MS	50 mL
Fluka	49916	Propionic acid	puriss.p.a., eluent additive for LC-MS, ≥99.5%	50 mL
Fluka	55674	Ammonium formate	puriss.p.a., eluent additive for LC-MS	50 g
Fluka	73594	Ammonium acetate	puriss.p.a., for mass spectrometry, ≥99.0% (calc.on dry substance, T)	25 g, 100 g
Fluka	71402	Sodium citrate tribasic dihydrate	BioUltra, for molecular biology, ≥99.5%	100 g, 250 g, 1 Kg
Fluka	40867	Ammonium bicarbonate	puriss.p.a., eluent additive for LC-MS	50 g
Fluka	44273	Ammonium hydroxide solution	puriss.p.a., eluent additive for LC-MS, ≥25% in H ₂ O	10x1 mL, 100 mL
Fluka	65897	Triethylamine	puriss.p.a., eluent additive for LC-MS, ≥99.5%	50 mL
Fluka	05841	2,2,2-Trifluoroethanol	puriss.p.a., eluent additive for LC-MS, ≥99.8%	10x1 mL, 50 mL



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

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Dispersive SPE for EN15662: 2008 “QuEChERS” Method

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Klaus Buckendahl, Tactical Marketing Sample Preparation, klaus.buckendahl@sial.com

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The core of the method is an adsorbent based on a primary and secondary amine phase (PSA), providing a high capacity for the removal of matrix components such as sugars, organic, and fatty acids as well as polar pigments. The Supelco® QuEChERS centrifuge tubes contain pre-weighed salt and sorbent in proportions exactly as described in the method by M. Anastassiades, one of the developers of the QuEChERS method. This method is formalized as European Standard Method EN 15662:2008 “Foods of plant origin – Determination of pesticide residues using GC-MS and/or LC-MS/MS following acetonitrile extraction/partitioning and cleanup by dispersive SPE – QuEChERS-method”.



The product portfolio covers extraction tubes with salts for adjusting the pH during sample extraction with acetonitrile, cleanup tubes with PSA and $MgSO_4$, and cleanup tubes with PSA and additions of carbon for more pigmented samples or C18 for lipophilic matrices.

To read more about the dispersive SPE products and the QuEChERS method, visit us at sigma-aldrich.com/quechers. Literature is available for download, including the overview chart JWP (see below, available as pdf file) with the complete product listing. For an application example, please see the article “Validation of the EN 15662 Method for the Determination of Pesticide Residues using a Fused-Core™ column and Dispersive SPE (QuEChERS)” (The Reporter Europe 39) in the Technical resource section. To try this method, contact our technical service to request your free sample pack.



nation of Pesticide Residues using a Fused-Core™ column and Dispersive SPE (QuEChERS)” (The Reporter Europe 39) in the Technical resource section. To try this method, contact our technical service to request your free sample pack.

(JWP)



Featured Products

Description	Qty.	Cat. No.
Dispersive SPE (dSPE) Products for EN15662:2008, packed in 15 mL centrifuge tube		
Citrate Extraction Tube 4 g $MgSO_4$, 1 g NaCl, 0.5 g sodium citrate dibasic sesquihydrate, 1 g sodium citrate tribasic dihydrate	50	55227-U
$MgSO_4$ Extraction Tube 6 g magnesium sulphate, 1.5 g sodium acetate	50	55234-U
PSA SPE CleanUp Tube 1 150 mg Supelclean™ PSA, 900 mg $MgSO_4$	50	55228-U
PSA/C18 SPE CleanUp Tube 1 150 mg Supelclean PSA, 150 mg Discovery® DSC-18, 900 mg $MgSO_4$	50	55229-U
PSA/ENVI-Carb SPE CleanUp Tube 1 150 mg Supelclean PSA, 15 mg Supelclean ENVI-Carb, 900 mg $MgSO_4$	50	55230-U
PSA/ENVI-Carb SPE CleanUp Tube 2 150 mg Supelclean PSA, 45 mg Supelclean ENVI-Carb™, 900 mg $MgSO_4$	50	55233-U
Empty Tubes		
50mL Centrifuge Tubes with cap, empty suitable for extraction step in QuEChERS method	50	55248-U
Bulk Adsorbents and Salts		
Supelclean PSA, bulk sorbent	100 g	52738-U
Supelclean ENVI-Carb, bulk sorbent	50 g	57210-U
Discovery DSC-18, bulk sorbent	100 g	52600-U
$MgSO_4$	var.	63135
Sodium citrate dibasic sesquihydrate	var.	71635
Sodium citrate tribasic dihydrate	var.	32320
Sodium chloride	var.	71379
Sodium acetate	var.	241245

Related Information



As the QuEChERS/dispersive SPE approach is getting more popular, users want different salt/adsorbent mixtures for different samples than shown in the EN15662. On our website sigma-aldrich.com/custom-quechers, we offer a service for customization of dSPE products, making application-specific mixes simple and quickly available. For the newest comprehensive listing of our SPE products, download your copy of our most recent SPE brochure at sigma-aldrich.com/spe. This new brochure includes the most recently introduced molecularly imprinted polymer SupelMIP™ phases for PAHs and NSAIDs as well as the Empore® line of SPE Disk filters and cartridges.

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53404	Atto 465-NHS ester	44756	Atto 580 Q NHS ester	07376	Atto 647 NHS ester
41698	Atto 488-NHS ester	79636	Atto 590 NHS ester	76508	Atto 647N NHS ester
41698	Atto 488-NHS ester	08741	Atto 594 NHS ester	76245	Atto 655 NHS ester
00379	Atto 495-NHS ester	93259	Atto 610 NHL ester	75999	Atto 680 NHS ester
77810	Atto 520 NHS ester	18708	Atto 611X NHS ester	16986	Atto 700 NHS-ester
88793	Atto 532 NHS ester	53988	Atto 612 Q NHS ester	93725	Atto 725 NHS ester
61683	Atto 540 Q NHS ester	67351	Atto 620 NHS ester	59808	Atto 740 NHS ester
92835	Atto 550 NHS ester	01464	Atto 633 NHS ester	08741	Atto 594 NHS ester

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Determination of Water Content in Candies

HYDRANAL® Applications: Caramel mass, Jelly sweets, Fruit gum and Fondant

Helga Hoffmann, Technical Service HYDRANAL Manager helga.hoffmann@sial.com
Andrea Felgner, Product Manager Analytical Reagents andrea.felgner@sial.com



Water content determination by Karl Fischer titration is a fast, accurate and reliable method. Especially for food samples, the Karl Fischer (KF) titration shows significant advantages over traditional drying techniques when determining water content. When food samples are heated and dried, the Maillard reaction, a reaction between amino acids and reducing sugars, may occur. During this non-enzymatic browning process, water is formed as a by-product of the reaction. In a drying process, this water would also be detected as weight loss, and the result of the determination would be biased. Furthermore, volatile components contained in the food samples would evaporate and also bias the drying result. KF titration, on the other hand, is not influenced by volatile components. Therefore, KF titration is a broadly applied technique for determining water content, not only in the food industry but also in chemical, pharmaceutical, and petrochemical industries.

In confectionery products, water content affects stability, texture, and flow properties of the product. Consequently, it is an important parameter which must be closely monitored to hold within specifications.

For the titration of the total water content, the sample should dissolve or be dispersed in the working medium. Dissolution can be increased by adding a polar solvent such as HYDRANAL-Formamide, by heating the working medium to about 50 °C using a double-walled titration vessel connected to a circulator, or by introducing a dispersing device

to the titration vessel to homogenize, or suspend the sample in the working medium before starting the titration. Instead of methanol as working medium, the non-toxic HYDRANAL-CompoSolver E can also be used. However, due to the poorer solubility of sugar-containing samples in this medium, titration times can be longer.

Water content determination in Caramel mass

The texture of candies such as soft caramels (toffees) depends on their water content. Soft caramel masses are mainly composed of sucrose, milk, starch syrup, invert sugar, and fat; their average water content is 4–8% [1]. Generally, the relationship between water content and texture is that the higher the water content, the softer the toffee. Flavor and quality-defining components like cream, honey, chocolate, nuts, or vitamins are added to the base mixture just before shaping the toffees.

Caramel masses do not dissolve easily in the methanolic medium of the KF titration at room temperature. Dissolution support is needed here and several possibilities are available:

- Addition of HYDRANAL-Formamide to the working medium
- Titration at 50 °C in a double-walled titration vessel
- Dispersing device introduced to the titration vessel to homogenize or suspend the sample in the working medium

(continued on page 18)

Complete dissolution of such samples may take up to several minutes. The softened caramel mass may stick to the indicator electrode, disrupting indication, and leading to over-titration. This can cause erroneous results, as the titration may be stopped although the water content has not been completely determined. In this case, the electrode must be cleaned and the titration started over with a new sample.

Application: Caramel mass (L413)

20 mL HYDRANAL®-Methanol-dry and 20 mL formamide, or 20 mL HYDRANAL-CompoSolver E and 20 mL formamide are added to the titration vessel, heated up to 50 °C, and titrated to dryness with HYDRANAL-Composite 5.

Sample size: approx. 0.25 g. The sample can be easily handled using a syringe without needle.

If a non-toxic working medium is preferred over methanol, then HYDRANAL-CompoSolver E can be used. The two-component reagent HYDRANAL-Titrant (E)/ Solvent (E) can also be used.



Water content determination in gum and gelatin candies

For production of jelly, gum, and gelatin candies, aromatized sugar solutions are heated with polysaccharides such as agar, pectin, gum arabic etc., and gelatin. They are formed by pouring the mass into starch moulds and removing the pieces after hardening [1]. The most famous gum candies are probably gummy bears, traditionally made with gelatin, but also available in vegetarian variations with starch instead of gelatin. The water content of these candies is relatively high with an average of 14–18%, resulting in the typical gum-like texture [2].

Samples containing high amounts of gelatin should be chopped very finely for this analysis, using a knife or scissors. The HYDRANAL service lab recommends adding formamide to the working medium and heating the titration vessel to 50 °C in order to dissolve the sample. Additionally, the use of a dispersing device is recommended and can reduce the titration time to 1–2 minutes.

Application: Jelly sweets (L402)

20 mL HYDRANAL-Methanol dry and 20 mL formamide are added to the titration vessel, heated to 50 °C, and titrated to dryness with HYDRANAL-Composite 5.

Sample size: approx. 0.2 g, cut into small pieces

The use of a homogenizer reduces dissolving and titration time. The two-component reagent HYDRANAL-Titrant/Solvent can also be used.

Application: Fruit gum with sugar crust (L228)

Fruit gum dissolves very slowly in the alcoholic media of the KF reagents. Again, it is recommended to cut the sample into small pieces with a knife or scissors. Addition of formamide to the working medium is required for sample dissolution. If the titration is carried out at 50 °C, dissolution time is around 3 minutes, with an additional 3–4 minutes for titration.

20 mL of HYDRANAL-Methanol dry or HYDRANAL-Methanol Rapid and 10 mL HYDRANAL-Formamide dry are added to the titration vessel, heated to 50 °C, and titrated to dryness with HYDRANAL-Composite 5.

Sample size: approx. 0.3 g, cut into small pieces

Allow for a dissolution time of around 3 minutes, then titrate with HYDRANAL-Composite 5. The two-component reagent HYDRANAL-Titrant/Solvent can also be used.

Water content determination in Fondant

Fondant is a sugar mixture that can be found in various shapes and colors, e.g. egg-shaped during the Easter season, or scented with peppermint aroma at Christmas. It can have a sugar percentage of up to 90%. This sugar is mainly present as undissolved small crystals (these crystals make fondant appear white), in a highly viscous suspension with saturated sugar solution [2]. The water content of Fondant lies at about 10–15% and the mass is still meltable and pourable on heating [1].

Fondant dissolves very slowly in the methanolic medium of Karl Fischer titration; the addition of formamide to the working medium is required. Titration at 50 °C further increases solubility.

Note: If a dispersing device in the titration vessel is used, the addition of formamide and the heating of the titration vessel are not necessary. In our investigations, titration under these conditions took 3 minutes; we obtained similar results with both methods.

Application: Fondant (sucrose and glucose syrup) (L322)

20 mL HYDRANAL®-Methanol dry or HYDRANAL-Methanol Rapid and 20 mL HYDRANAL-Formamide dry are added to the titration vessel, warmed to 50 °C, and titrated to dryness with HYDRANAL-Composite 5.

Sample size: approx. 0.3 g. The sample can be easily handled using a syringe without a needle.

Procedure with dispersing device

40 mL HYDRANAL-Methanol dry or HYDRANAL-Methanol Rapid are added to the titration vessel. The homogenizer is switched on and the vessel is titrated to dryness under these conditions.

Sample size: approx. 0.3 g. Titration starts immediately. After dissolution of the sample, the disperser can be switched off.

The two-component reagent HYDRANAL-Titrant/Solvent can also be used. When using the disperser, 20 mL HYDRANAL-Solvent and 20 mL HYDRANAL-Methanol dry are used as the working medium.

Cat. No.	Brand	Description	Pack Size
34805	Fluka®	HYDRANAL-Composite 5	500 mL, 1 L, 2.5 L
34741	Fluka	HYDRANAL-Methanol dry	1 L, 2.5 L
37817	Fluka	HYDRANAL-Methanol Rapid	1 L, 2.5 L
34734	Fluka	HYDRANAL-CompoSolver E	1 L, 2.5 L
34724	Fluka	HYDRANAL-Formamide dry	1 L
34801	Fluka	HYDRANAL-Titrant 5	500 mL, 1 L, 2.5 L
34732	Fluka	HYDRANAL-Titrant 5 E	500 mL, 1 L, 2.5 L
34800	Fluka	HYDRANAL-Solvent	1 L, 2.5 L
34730	Fluka	HYDRANAL-Solvent E	500 mL, 1 L, 2.5 L

Table 1 Selected HYDRANAL Karl Fischer reagents

Available application reports:

- L413 Caramel mass
- L402 Jelly sweets
- L228 Fruit gum with sugar crust
- L322 Fondant (sucrose and glucose syrup)

Sigma-Aldrich offers over 600 application reports. A full list can be found on our website sigma-aldrich.com/hydranal. To obtain an application report, or to request assistance with your KF application, please contact one of our HYDRANAL laboratories (see below).

References

- [1] Food Chemistry. 4th revised and extended Edition. H.-D. Belitz, W. Grosch, P. Schieberle. Springer-Verlag Berlin Heidelberg, 2009.
- [2] Bundesverband der Deutschen Süßwarenindustrie e.V. www.bdsi.de



Europe and Global Market

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



USA and Canada

Mr. Doug Clark
 HYDRANAL Technical Center
 545 S. Ewing Ave
 St. Louis MO 63103, USA
 Toll free: +1 800 493-7262
 (USA and Canada)
 Fax: +1 314 286-6699
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Questions regarding KF titration? Contact us at hydranal@sial.com

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



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Sigma-Aldrich achieves accreditation according to ISO/IEC 17025 for Water Standards and Karl Fischer Reagents

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

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

**HYDRANAL® Technical Service
accredited according to
ISO/IEC 17025**

Sigma-Aldrich's HYDRANAL® service laboratory in Seelze, Germany, has proved its expertise for testing and calibration and achieved accreditation as a testing laboratory according to ISO/IEC 17025:2005 "General requirements for the competence of testing and calibration laboratories". It was

audited by the German Accreditation Council (Deutscher Akkreditierungsrat DAR) and received accreditation for titration and gravimetry for water standards and reagents for Karl Fischer titration.

From the beginning of our HYDRANAL product line over 30 years ago, the HYDRANAL technical service in Seelze has applied high standards and continual improvement of testing methods for production and quality control of HYDRANAL water standards and HYDRANAL Karl Fischer reagents. These high-quality testing methods have now been proven to comply with the high standards of the auditors and have led to the accreditation of HYDRANAL Technical Service according to ISO/IEC 17025. This accreditation includes all HYDRANAL products, and the accreditation details, along with the logo of the accreditation body, will now appear on the Certificate of Analysis (see **Figure 1**) and on the label for each produced lot.

Accreditation requirements and procedure

Accreditation means that an authoritative body formally recognizes that an organization or individual is competent to execute a specific service as described in the scope of accreditation. ISO/IEC 17025 is associated with a well-defined analytical technique and a stated measurement range comprising analyte, matrix, and concentration range.

HYDRANAL Technical Service is now accredited to perform titration and gravimetry for water standards and reagents for Karl Fischer titration according to the following methods/norms: ISO 760:1978-12, ASTM E203-08 and HYDRANAL Technical Service's in-house methods 01-04. Following the accreditation, evidence of continued improvement is required, including regular internal audits and the demonstration of efforts towards scientific and technological progress.

Analytical results produced in laboratories fulfilling ISO/IEC 17025 can be labeled with a special quality logo that confirms the reliability of the results. Every country has at least one official body that is responsible for accreditation, and each body signifies its accreditation with its own unique logo. Since HYDRANAL Technical Service was accredited by DAR, the DAR symbol and the individual registration number of the laboratory must appear on the certificate of Karl Fischer reagents and water standards. Beginning in 2010, the authoritative body for accreditations in Germany is the Deutsche Akkreditierungsstelle (DAkkS), which will also administrate accreditations issued by former authorities.




Certificate of Analysis		 Fluka Analytical	sigma-aldrich.com
Analysed for Sigma-Aldrich Laborchemikalien GmbH, Production HYDRANAL [®] , Wunstorfer Str.40, D-30926 Seelze			
HYDRANAL[®] Water Standard 10.0 , reference no. 34849, is intended for the control of the volumetric determination of water. The certified water content of this Lot (SZE92080, Exp.date June 2014) is			
10.02 mg/g (expanded uncertainty = 0.03 mg/g, k =2).			
The water content is certified by volumetric titration on 11 samples according to ISO 760 and ASTM E 203. The coulometric KF titration was used as a second technique to confirm the reliability of the specified water content. HYDRANAL [®] Water Standard 10.0 can be used to control KF titrators according to ISO 9001, chapter "Control of monitoring and measuring devices".			
This standard is traceable to SI-Unit (kg) and tested against NIST SRM 2890 .			
Sigma-Aldrich Laborchemikalien GmbH Helga Hoffmann Manager Technical Service HYDRANAL [®] Wunstorfer Str.40, D-30926 Seelze			QC release date Seelze, 04.03.2010
			page 1 of 1

Figure 1 Example of HYDRANAL Certificate of Analysis with accreditation details

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35328	FLUKA	Hydrochloric acid solution, Reag. Ph.Eur., 1 M
35329	FLUKA	Hydrochloric acid solution, Reag. Ph.Eur., 0,5 M
35335	FLUKA	Hydrochloric acid solution, Reag. Ph.Eur., 0,1 M
35354	FLUKA	Sulphuric acid solution, Reag. Ph.Eur., 0,5 M
35256	FLUKA	Sodium hydroxide solution, Reag. Ph.Eur., 1 M
35257	FLUKA	Sodium hydroxide solution, 0,5 M
35263	FLUKA	Sodium hydroxide solution, Reag. Ph.Eur., 0,1 M
35115	FLUKA	Potassium hydroxide solution, 0,5 M denat. Ethanol with Toluene
35127	FLUKA	Potassium hydroxide solution, 0,1 M denat. Ethanol with Toluene

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New High-Purity Standards for Pesticide Residue Analysis

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



Through our PESTANAL® line we offer a wide portfolio of over 1200 high-purity pesticide and metabolite standards, including isotope labeled compounds for food and environmental analysis. This portfolio is continually expanding.

Please find the most recent additions of new products in the list below.

A complete product listing can be found on our website.

Brand	Cat. No.	Description	Pack Size
FLUKA®	32581	Bixafen	100 mg
FLUKA	32430	Cafenstrole	50 mg
FLUKA	32413	Carbendazim-d3	10 mg
FLUKA	32588	Cloethocarb	50 mg
FLUKA	32403	Cyflufenamid	25 mg
FLUKA	32432	Dimefluthrin	100 mg
FLUKA	32497	Dimethachlor Metabolite SYN 528702 sodium salt	100 mg
FLUKA	32499	Dinotefuran	50 mg
FLUKA	32586	Florasulam	50 mg
FLUKA	32405	Flupyrsulphuron-methyl sodium	25 mg
FLUKA	32431	Furilazole	50 mg
FLUKA	32419	Novaluron	25 mg
FLUKA	32427	Orthosulphamuron	50 mg
FLUKA	32428	Orysastrobin	100 mg
FLUKA	32433	Primisulphuron-methyl	100 mg
FLUKA	32429	Prothioconazole-desthio	20 mg
FLUKA	32435	Saflufenacil	100 mg
FLUKA	32498	Silthiofam	25 mg
FLUKA	32584	Tembotrione metabolite AE 1417268	25 mg
FLUKA	32434	TEPP	50 mg
FLUKA	32418	Tralopyril	100 mg

New Veterinary Drug Standards

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



To prevent infections, parasites, or disease, the use of drugs, such as antibiotics, is widespread in modern agriculture. Therefore, foodstuffs of animal origin need to be tested for residues of medicinal products and their metabolites.

Our VETRANAL® product line includes over 200 high-purity standards of the active ingredients of veterinary drugs, including isotope labeled compounds as well as common metabolites. In the table, the most recent new product additions of the VETRANAL line are listed. Please find a complete product listing on our website.

Brand	Cat. No.	Description	Pack Size
FLUKA	32591	Baquiloprim	10 mg
FLUKA	32592	Baquiloprim-d6	10 mg
FLUKA	32423	Benazepril hydrochloride	100 mg
FLUKA	32593	Cambendazole	10 mg
FLUKA	32594	Cambendazole-d7	10 mg
FLUKA	32426	Cefoperazone	100 mg
FLUKA	32422	Ceftiofur hydrochloride	100 mg
FLUKA	32589	Ciclobendazole	10 mg
FLUKA	32425	Cyclosporin A	100 mg
FLUKA	32407	Cymiazole	100 mg
FLUKA	32404	Flavomycin	25 mg
FLUKA	32595	4-Formylaminoantipyrine	10 mg
FLUKA	32412	4'-Hydroxydiclofenac	10 mg
FLUKA	32424	Ibuprofen	100 mg
FLUKA	32725	Imibenconazole solution	2 ml
FLUKA	32417	Natamycin	50 mg
FLUKA	32409	Nicarbazin	100 mg
FLUKA	32436	Ofloxacin-d3	10 mg
FLUKA	32421	Sulphaclozine sodium	100 mg
FLUKA	32402	Sulphaguanidine	100 mg
FLUKA	32414	Trimethoprim-d9	10 mg

Sigma-Aldrich Worldwide Offices

Argentina

Free Tel: 0810 888 7446
Tel: (+54) 11 4556 1472
Fax: (+54) 11 4552 1698

Australia

Free Tel: 1800 800 097
Free Fax: 1800 800 096
Tel: (+61) 2 9841 0555
Fax: (+61) 2 9841 0500

Austria

Tel: (+43) 1 605 81 10
Fax: (+43) 1 605 81 20

Belgium

Free Tel: 0800 14747
Free Fax: 0800 14745
Tel: (+32) 3 899 13 01
Fax: (+32) 3 899 13 11

Brazil

Free Tel: 0800 701 7425
Tel: (+55) 11 3732 3100
Fax: (+55) 11 5522 9895

Canada

Free Tel: 1800 565 1400
Free Fax: 1800 265 3858
Tel: (+1) 905 829 9500
Fax: (+1) 905 829 9292

Chile

Tel: (+56) 2 495 7395
Fax: (+56) 2 495 7396

China

Free Tel: 800 819 3336
Tel: (+86) 21 6141 5566
Fax: (+86) 21 6141 5567

Czech Republic

Tel: (+420) 246 003 200
Fax: (+420) 246 003 291

Denmark

Tel: (+45) 43 56 59 00
Fax: (+45) 43 56 59 05

Finland

Tel: (+358) 9 350 9250
Fax: (+358) 9 350 92555

France

Free Tel: 0800 211 408
Free Fax: 0800 031 052
Tel: (+33) 474 82 28 88
Fax: (+33) 474 95 68 08

Germany

Free Tel: 0800 51 55 000
Free Fax: 0800 64 90 000
Tel: (+49) 89 6513 0
Fax: (+49) 89 6513 1160

Hungary

Ingyenes telefonszám: 06 80 355 355
Ingyenes fax szám: 06 80 344 344
Tel: (+36) 1 235 9063
Fax: (+36) 1 269 6470

India

Telephone

Bangalore: (+91) 80 6621 9400
New Delhi: (+91) 11 4358 8000
Mumbai: (+91) 22 2570 2364
Hyderabad: (+91) 40 4015 5488
Kolkata: (+91) 33 4013 8003

Fax

Bangalore: (+91) 80 6621 9650
New Delhi: (+91) 11 4358 8001
Mumbai: (+91) 22 4087 2364
Hyderabad: (+91) 40 4015 5488
Kolkata: (+91) 33 4013 8000

Ireland

Free Tel: 1800 200 888
Free Fax: 1800 600 222
Tel: (+353) 402 20370
Fax: (+353) 402 20375

Israel

Free Tel: 1 800 70 2222
Tel: (+972) 8 948 4100
Fax: (+972) 8 948 4200

Italy

Tel: (+39) 02 3341 7310
Fax: (+39) 02 3801 0737

Japan

Tel: (+81) 3 5796 7300
Fax: (+81) 3 5796 7315

Korea

Free Tel: (+82) 80 023 7111
Free Fax: (+82) 80 023 8111
Tel: (+82) 31 329 9000
Fax: (+82) 31 329 9090

Malaysia

Tel: (+60) 3 5635 3321
Fax: (+60) 3 5635 4116

Mexico

Free Tel: 01 800 007 5300
Free Fax: 01 800 712 9920
Tel: (+52) 722 276 1600
Fax: (+52) 722 276 1601

The Netherlands

Free Tel: 0800 022 9088
Free Fax: 0800 022 9089
Tel: (+31) 78 620 5411
Fax: (+31) 78 620 5421

New Zealand

Free Tel: 0800 936 666
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Tel: (+61) 2 9841 0555
Fax: (+61) 2 9841 0500

Norway

Tel: (+47) 23 17 60 00
Fax: (+47) 23 17 60 10

Poland

Tel: (+48) 61 829 01 00
Fax: (+48) 61 829 01 20

Portugal

Free Tel: 800 202 180
Free Fax: 800 202 178
Tel: (+351) 21 924 2555
Fax: (+351) 21 924 2610

Russia

Tel: (+7) 495 621 5828
Fax: (+7) 495 621 5923

Singapore

Tel: (+65) 6779 1200
Fax: (+65) 6779 1822

Slovakia

Tel: (+421) 255 571 562
Fax: (+421) 255 571 564

South Africa

Free Tel: 0800 1100 75
Free Fax: 0800 1100 79
Tel: (+27) 11 979 1188
Fax: (+27) 11 979 1119

Spain

Free Tel: 900 101 376
Free Fax: 900 102 028
Tel: (+34) 91 661 99 77
Fax: (+34) 91 661 96 42

Sweden

Tel: (+46) 8 742 4200
Fax: (+46) 8 742 4243

Switzerland

Free Tel: 0800 80 00 80
Free Fax: 0800 80 00 81
Tel: (+41) 81 755 2828
Fax: (+41) 81 755 2815

United Kingdom

Free Tel: 0800 717 181
Free Fax: 0800 378 785
Tel: (+44) 1747 833 000
Fax: (+44) 1747 833 313

United States

Toll-Free: 800 325 3010
Toll-Free Fax: 800 325 5052
Tel: (+1) 314 771 5765
Fax: (+1) 314 771 5757

Vietnam

Tel: (+84) 3516 2810
Fax: (+84) 6258 4238

Internet

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

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