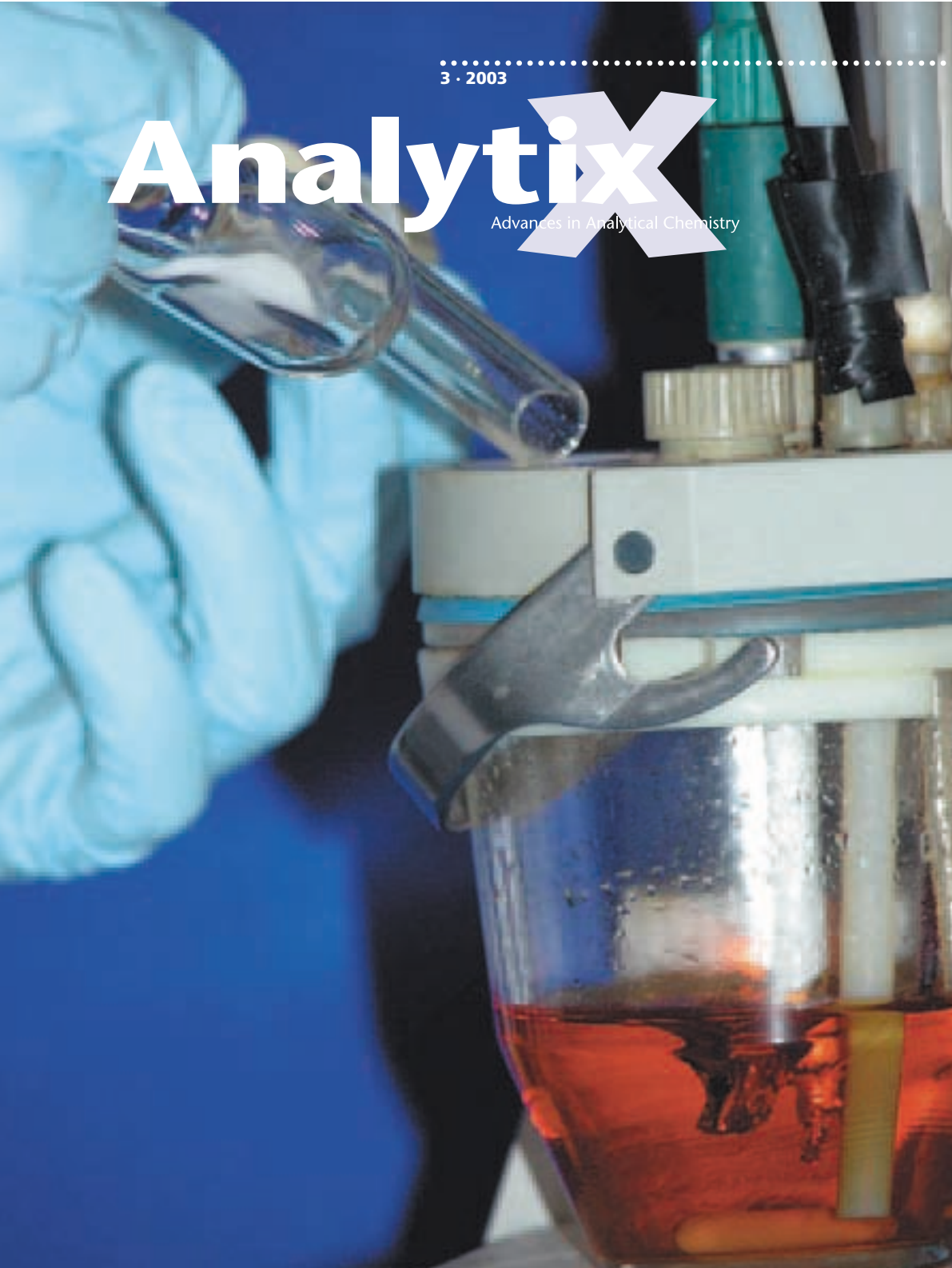


3 · 2003

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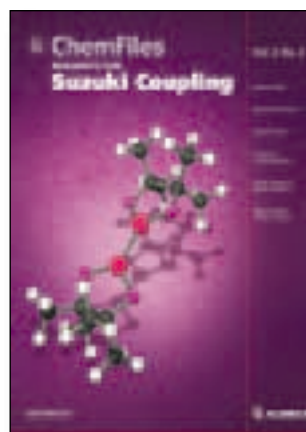
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Analytical News Corner

Dear valued customer,

You are the driver of our innovation! The result of your suggestions has resulted in a broad variety of new standards and reagents of topical interest presented in this AnalytiX. Highlights you will find are for example:

- **GMO Standards** for qualitative determination of non-approved GMO's
- **Phytopharmaca standards** for determination of natural active compounds

Based on many market requests, we have confirmed and documented **NIST traceability** for all our well-known Riedel volumetric solutions, providing you with a higher level of certainty in your daily analytical work.

Due to its reliability and accuracy **Karl Fischer Titration** has the potential to substitute other water determination methods. This is clearly shown in the comparison study written by Prof. Isengard (University of Hohenheim, Germany) one of the leading experts in food analysis. Our label **Hydranal®** has become the synonym for quality products for water determination. Benefit from our enormous application know-how in Karl Fischer titration!

Areas such as **Water Analysis, Polymer Characterization** and **Microbiology** are also covered in the current issue. Please take a few minutes of your time and discover all new offerings presented in this Analytical Newsletter.

We look forward to hearing from you. Please mark your areas of interest on the promotion sheet and fax it back to your local Sigma-Aldrich office.

Dr. Kurt Vorbürger
Head Fluka Riedel-de Haën Product Management
kvorbuerger@sial.com

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Certified Polystyrene Particle Size Standards

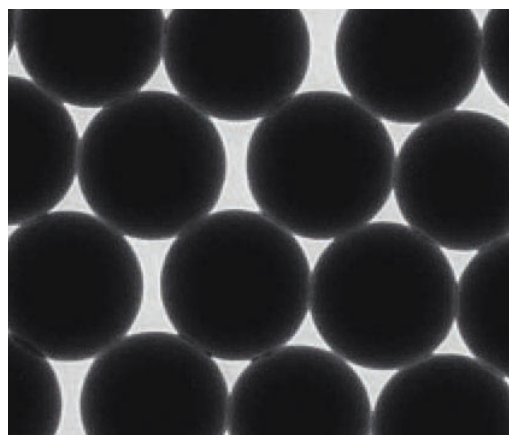
The increasing need for validation and quality assurance requires accurate tools for calibration. Particle size measurement plays a crucial role in applications like:

- Development of new pharmaceutical formulations
- Size determination of biological structures, such as cellular compounds, viruses or bacteria
- Quality assurance of powders in production facilities for food, cosmetics, clay, ceramics, and many others. In these cases, size distribution is an important criterion for checking the consistency of processes and product quality.
- Inspection in automated wafer technology

A variety of methods are used for particle size determination. The most popular among these are sieving (cell sorting, field flow fractionation etc.), sedimentation, light interaction methods (especially light scattering), electronic methods, in particular, the Coulter electrozone principle, or mi-

Figure 1:

Transmission electron microscopy image of PS particles (Photograph by courtesy of Max Planck Institute of Colloids and Interfaces)



croscopy (transmission and scanning electronic microscopy, atomic force microscopy). In **Figure 1** is shown an example of particle size measurement using TEM. Most methods are indirect and measuring the particle size and size distribution by different technologies will not provide the same results. Therefore, calibration standards are required.

Certified Particle Size Standards

Well characterized certified particle size standards are the *polystyrene particles* within the range of 100 nm – 30 µm. The accurate diameter of these particles is determined by methods described by the National Institute of Standards and Technology (NIST, USA), such as

- Transmission electron microscopy
- Scanning electron microscopy
- Optical microscopy
- COULTER COUNTER® particle size analyzer with HDF system

The measuring instruments are calibrated using standards obtained from the Community Bureau

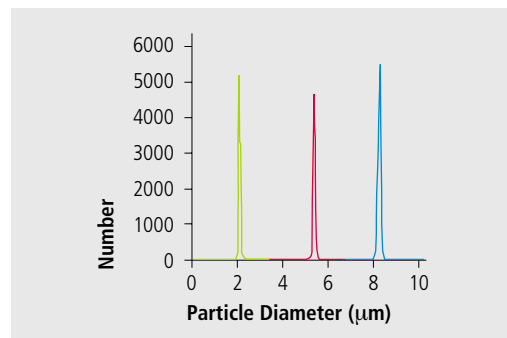


Figure 2:

Size distribution curves of three different calibration standard particles measured using a COULTER COUNTER®

of Reference (BCR, Belgium: Standards RM 165, 166 or 167) and from the NIST (SRM 1690, 1691 or 1692), making our particle size standards traceable to internationally recognized ones.

The excellent quality of the particles supplied by Fluka is demonstrated by the figure above (**Figure 2**). The certificate of analysis for each batch includes the particle diameter and the standard deviation, as well as some physical data (i. e. solids content, particle number per ml, specific weight, refractive index).

Physical and Chemical Properties of Polystyrene Particles

The most important properties of our polystyrene particles are:

- Density: 1.05g/cm³
- Refractive Index: 1.59
- High monodispersity and uniform spherical shape
- Hydrophobic surface
- Non-specific adsorption of proteins
- Low temperature resistance up to 100 °C
- Soluble in organic solvents (dependent on the degree of cross-linking)
- Swellable in organic solvents
- Coefficient of Variation (C.V. value) ≤ 2% for particle size standards

Table 1:

Product Range and Specifications.

Cat. No	Particle size	Concentration aqueous suspension (w/v)	Particle size variation coefficient	Particle size standard deviation
90517	0.1 µm	2%		<0.03 µm
95581	0.2 µm	2%		<0.03 µm
95585	0.5 µm	2%		<0.03 µm
72938	1.0 µm	2%		<0.05 µm
80177	2.0 µm	2%	<3%	<0.05 µm
80304	3.0 µm	2%	<2%	<0.05 µm
81494	4.0 µm	2%	<2%	<0.1 µm
89756	6.0 µm	2%	<2%	<0.1 µm
86996	7.0 µm	2%	<1.5%	<0.1 µm
84192	8.0 µm	2%	<1.5%	<0.1 µm
87466	9.0 µm	2%	<1.5%	<0.1 µm
72822	10.0 µm	2%	<1.5%	<0.15 µm
96020	11.0 µm	2%	<1%	<0.2 µm
88511	12.0 µm	2%	<1%	<0.2 µm
87896	20 µm	2%	<1%	<0.4 µm
95531	30 µm	2%	<1%	<0.5 µm

NEW Qualitative GMO Standards for Maize

NK603, GA21 and CBH-3511

Does Your Sample Contain Non-Approved GMO's?

In many countries there is a wide variety of perceptions of genetically modified organisms (GMO's). Many consumers still avoid GMO's, and most European Union retailers avoid putting food containing GMO's on their shelves. The control and detection of GMO's is dependent on the reference material used. As shown by recent events, it is rather difficult to manufacture acceptable reference materials.

The new Fluka GMO maize standards contain varieties that are not approved in the European Union and many other countries worldwide. For the first time, there will be an easy way to obtain positive controls for three different GMO maize varieties. These new qualitative standards will clearly fill a gap in the requirements of the quality control of GMO analyses.

The New Fluka GMO Standards Give You a Reliable Answer!

The new GMO standards contain GA21-, NK603- or CBH-351 maize DNA that has been extracted from plant material. The percentage of GMO DNA is about 1% of the total DNA. The DNA was extracted using the Wizard® DNA-extraction method, which is the same method described in official protocols (e.g [1]) and tested in several validation studies [2-4]. The extracted DNA was quantified and diluted to a defined concentration. These mixtures were aliquoted and lyophilised to increase the shelf life of the materials.

These new GMO standards present clear advantages. There is no need to extract the DNA before use as a positive control. The fact that the standards are made with DNA extracted from plant material allows their application in many DNA-based assays. Also, their application is not limited to a certain sequence information. Finally, the worldwide availability of GMO standards of this quality will help to compare results obtained in different laboratories.

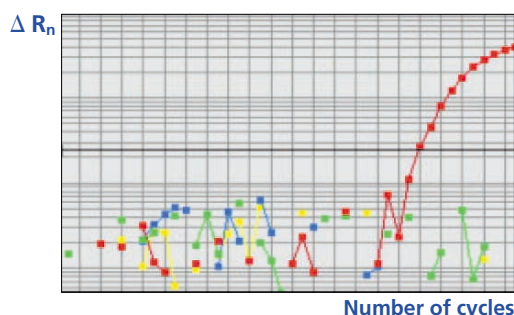


Figure 1: Real-time PCR detection of the CBH-351 standard applying a zein-specific method

Application

The new GMO standards can be used only as qualitative standards. The DNA is lyophilised and can be used as a positive control after being re-suspended. The native maize content of the samples is about 100 times higher than the GMO content (see Figure 1 and 2).

References

- [1] SLMB2001: Swiss Food Manual (SLMB 2001) (2001) Eidgenössische Drucksachen und Materialzentrale CH-3003 Bern, Switzerland or
- [2] U. Pauli, B. Schouwey, P. Hübner, P. Brodmann, A. Eugster. Quantitative Detection of Genetically Modified Soybean and Maize: Method Evaluation in a Swiss Ring Trial. Mitt. Gebiete Lebensm. Hyg. 92, 145-158 (2001)
- [3] M. Lipp, A. Bluth, F. Eyquem, L. Kruse, H. Schimmel, G. Van den Eede, E. Anklam. Validation of a method based on polymerase chain reaction for the detection of genetically modified organisms in various processed foodstuffs. Eur Food Res Technol 212, 497-504 (2001)
- [4] H. Hird, J. Powell, M.-L. Johnson, S. Oehlschläge.: Determination of Percentage of RoundUp Ready Soya in Soya Flour Using Real-Time Polymerase Chain Reaction: Inter-laboratory Study. Journal of AOAC International 86, 66-71 (2003)

69407 GMO Genomic DNA Standard Set for Maize NK603, GA21 and CBH-3511 «Starlink» 1 set

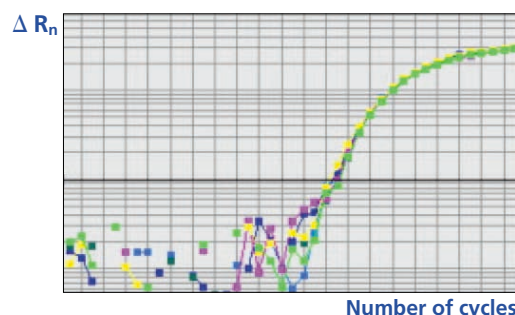
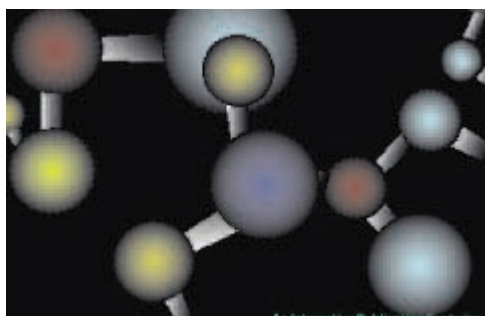


Figure 2: Real-time PCR detection of the CBH-351 standard applying a CBH-351 specific method



ReadyCal Standards for GPC



Today, GPC is the most commonly used method to define molecular masses and distribution of polymers. Their determination, however, is not done in absolute terms: it strictly relies on the use of appropriate standards for calibration. Therefore, precision depends on the quality of the calibration curve.

Designing and recording a calibration curve calls for great care and well-defined conditions. The type of standards that are used, as well as the selected molecular masses and their concentrations are of utmost importance.

GPC analysis of polymers has to be handled in an effective and cost-efficient manner, as working time is important. Additional and yet avoidable work has to be reduced to a minimum. The recording of a calibration curve is a perfect example (see **Table 1** for cost and time estimation). Polymer standards need to be collected, weighed and then dissolved. Furthermore, it has to be ensured that the polymers are present in an appropriate concentration, depending on their molecular weight.

To record a calibration curve, a standard can be weighed either separately or as part of a mixture. The latter method requires an appropriate mix of molecular weights. In particular, it has to be carefully observed that the molecular weights of the polymers only slightly overlap or don't overlap at all. The advantage of measuring three to four polymers simultaneously in a single injection sample lies in the fact that information is gathered in a short time.

All the above considerations have helped us to find a way to help you with your work. Our ReadyCal Standards allow you to record and verify your calibration curve in an efficient, fast and reproducible manner. Given amounts of three or four polymers of the same kind are already weighed in ready-to-use auto-sampler vials.

Add the appropriate amount of solvent to obtain the recommended or intended concentrations, wait until the polymer has dissolved. Inject samples from the three different vials with preset mixes of molecular weights and known concentrations to record a complete calibration curve for nine to twelve standards.

ReadyCal: Calibration made easy. Fast, exact, and reproducible calibration with minimal effort.

ReadyCal Standards Advantages:

- Fast
- Reliable
- Easy and safe to handle
- Free of contamination
- Complete reproducibility in peak position and peak height

The benefits of ReadyCal Standards:

- Pre-weighed
- Supplied in ready-to-use auto-sampler vials
- Fewer injections
- Time-saving analyses

Applicability of ReadyCal Standards:

- Conventional calibration
- Universal calibration
- Instrument validation
- Calibration of viscometer and light scattering detectors

ReadyCal standards come in a handy box containing 10 x 3 vials (1.5 ml or 4 ml). The included certificate contains information about molecular masses, net weight, concentration as well as the chromatogram of the respective cocktail of molecular masses.



Table 1:
Cost and Time estimation for recording a calibration curve

Example – required time	Conventional Standards	ReadyCal Standards
Weighing/ single standard	5–7 min	0 min
Weighing of 12 standards	60–84 min	0 min
Filling of 12 samples into autosampler vials	15 min	0 min
Preparation of weighing	10 min	0 min
Total time to weigh 12 standards	85–109 min	0 min

Conclusion: Considering similar costs for material, the investment of the ReadyCals have paid off after four to five calibration curves.

Remark: Salary for lab assistant: 25 €/hr, costs to weigh 12 standards: 35–45 €

Cap Color	M _p	M _w	M _n	Mass [mg]	Conc. (w/w% in 1.5 ml solvent)	Chromatogram Polystyrene
Blue	2 180 000	2 000 000	1 800 000	0.75	0.05	Polystyrene
	246 000	226 000	214 000	1.50	0.10	ReadyCals
	32 500	32 000	31 000	1.50	0.10	
	3 420	3 470	3 280	1.50	0.10	Blue
Green	1 000 000	960 000	930 000	0.75	0.05	Polystyrene
	128 000	125 000	123 000	1.50	0.10	ReadyCals
	18 000	17 400	16 600	1.50	0.10	
	1 620	1 560	1 500	1.50	0.10	
Red	659 000	644 000	623 000	1.50	0.10	Polystyrene
	67 500	65 000	64 000	1.50	0.10	ReadyCals
	9 130	8 620	8 260	1.50	0.10	
	374	410	360	1.50	0.10	Red

Table 3: Datasheet included with Polystyrene ReadyCal (Cat. No 81434)

ReadyCals are available for organic and aqueous applications: Polystyrene, Poly(methyl methacrylate), Poly(ethylene glycol), Poly(ethylene oxide) and poly(tetrahydrofuran), see **Table 2**. An example of a datasheet is shown in **Table 3** (Cat. No. 81434, Polystyrene standard ReadyCal set M_p 400–2'000'000)

Besides the ReadyCal Standards, Fluka offers a broad range of single Polymer standards, tested and certified for various applications. Examples of these standards are:

- Organic soluble standards (e.g. Polystyrene, PMMA, Polycarbonate, Polypropylene, Polyethylene)
- Water soluble standards (e.g. Poly(ethylene glycol), Pullulanes, Hydroxypropylcellulose)
- Certified Reference Materials (CRMs) (e.g. Polylactide, Poly(ethylene oxide), Poly(styrene) (narrow and broad): Certified by German Federal Institute for Materials Research and Testing (BAM)
- DIN certified standards (e.g. Dextrane, Poly(ethylene glycol), Poly(methylmethacrylate), Poly(styrene): DIN certified standards are characterized by GPC analysis and an absolute molar mass determination.
- Validation kits for MALDI, viscometry and light scattering (e.g. Dextrane, Poly(methyl methacrylate), Poly(styrene). Light scattering / viscometry validation kits are characterized by conventional GPC, together with light scattering detection and viscometry. MALDI validation kits are characterized by conventional GPC, an absolute method and MALDI-TOF mass spectrometry.

Should you need detailed information about these products for GPC analysis or a price list in your local currency, please contact:

Rainer Walz, Ph.D.
Product Manager Fluka Riedel-de Haen
Tel: 0041/81/755-2839
Fax: 0041/81/755-2824
Email: rwalz@sial.com

For further information on polymers please take a look at www.sigma-aldrich.com/aldrich/brochure/polymer2000.pdf

And for further information on GPC Standards, please take a look at: www.sigmaaldrich.com/Brands/Fluka__Riedel_Home/Analytical/Chromatography/LC/GPC_GFC_Standards.html

Cat. No:	Product
87976	Polyethylene glycol Standard ReadyCal Set M _p 100–40'000
02393	Polyethylene glycol/Polyethylene oxide standard ReadyCal set M _p 200–1'700'000
81506	Poly(methyl methacrylate) standard ReadyCal set M _p 500–4'000'000
81434	Polystyrene standard ReadyCal set M _p 400-2'500'000
76551	Polystyrene (high molecular) Standard ReadyCal Set M _p 1'500–7'500'000
76552	Polystyrene (low) Standard ReadyCal Set M _p 250–70'000

Table 2
ReadyCal Kits



Aconitum napellus

Nature plays an important role as a source for pharmaceuticals. The naturally occurring products obtained from infusions, extractions and preparations of medicinal plants are called phytopharmaca. So far, more than 500 000 plants and fungi have been identified but only 10% of the natural products are being actively investigated for their health-promoting potential. By using medicinal plants to meet health care needs, phytopharmaca provides a readily available, low-cost alternative to the more expensive conventional products that are usually marketed in developed countries. As a result, modern medicinal formulations based in phytotherapy have become a main topic in clinical science and phytopharmaca are now widely used in the production of medicaments.

Screening by HTS in Combination with HPTLC and HPLC

Investigating an enormous number of potential bioactive substances with defined pharmacologi-

cal impacts requires highly efficient methods. Today, common methods use high-throughput-screening systems (HTS). Some advantages of HTS are automated procedures, fast detection, high sensitivity and fast data processing. The detection method of choice is fluorescence correlation spectroscopy (FCS). The screening using FCS requires a selective separation of plant extracts by basic chromatographic methods like high performance thin layer chromatography (HPTLC) in combination with high performance liquid chromatography (HPLC) and high performance liquid chromatography-mass spectroscopy (HPLC-MS). For performing a reliable quality control, standards of phytopharmaca are needed. According to international guidelines such as EN 45001, ISO 17025 and ISO 900, analytical standards of phytopharmaceuticals require the following additional data:

- manufacturing date
- storage conditions
- stability
- assay methods
- quality/purity
- data (chromatograms, spectra)

Fluka has been developing this product range and is now proud to offer you a selection of the most important analytical standards (see **Table 1**): For example, Aucubin, Cat. No. 55561 belongs to the family of Iridoids-glucosides. It naturally occurs in Eyebright (*Euphrasia rostkoviana*, see **Figure 1**) and in many other plants. It is an optically active compound, and works as an antioxidant and inhibitor. It also presents bactericide, antiviral and laxative properties [1]. In **Figure 2** you may find a typical HPLC chromatogram for Fluka's analytical standard of Aucubin.



Figure 1: *Euphrasia rostkoviana*

Asiaticosid (Cat. No 43191) is derived from Hydrocotyle (*Centella asiatica*, see **Figure 3**) and belongs to the family of saponine-triterpenes. This natural product has antimicrobial and healing effects: Asiaticosid mixture accelerated the healing process of wounds in repeated skin injury tests.

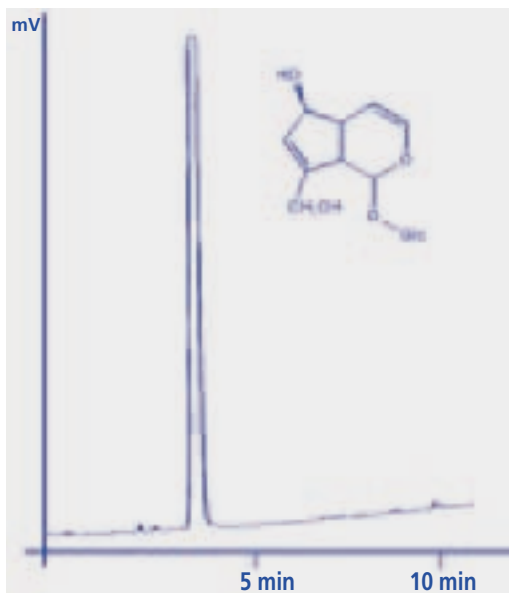


Figure 4: Chromatogram of Aucubine
 Column: Nucleosil 100-7 μ . C18 125 x 4.0 mm
 Eluent: Acetonitrile / 0.01M Phosphoric acid gradient
 Flow Rate: 2 ml/min
 Detection: UV, 208 nm
 Sample: 20 mg in 10 ml
 Injection Volume: 20 μ l

Moreover, in a study of 94 patients with chronic insufficiency of the veins, the application of Asiaticoside led to a significant improvement in both subjective (heaviness in the legs, pain in standing up, oedema) and objective (plethysmographic measurements of vein tone) parameters. The influence of Asiaticosid on the formation of collagen is still under discussion. Asiaticosid probably promotes the metabolic pathways in fibroblasts and inhibits the formation of scars [2]. **Figure 4** shows a typical HPLC chromatogram of Fluka's analytical standard of Asiaticosid.

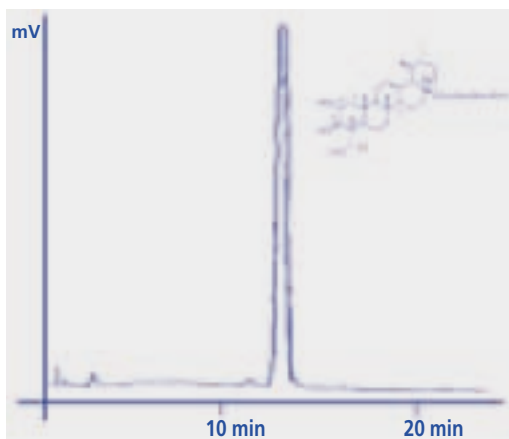


Figure 2: Chromatogram of Asiaticosid
 Column: Nucleosil 100-3 μ . C18 120 x 4.0 mm
 Eluent: Acetonitrile / Water 25:75
 Flow Rate: 2 ml/min
 Detection: UV, 200 nm
 Sample: 1 mg/100 μ l (Eluent + MeOH)
 Injection volume: 20 μ l



Figure 3: *Centella asiatica*

Chromatograms and NMR spectra are available on request. Should you need more information about our complete program of HPLC Standards please contact:

Rainer Walz, Ph.D.
 Product Manager Fluka/Riedel-de Haen
 Tel: 0041/81/755-2839
 Fax: 0041/81/755-2824
 Email: rwalz@sial.com

Literature:

- [1] I.M. Chang. Antiviral activity of aucubin against hepatitis B virus replication, *Phytotherap. Research*, 11,189–192 (1997)
- [2] F. Bonte et al. Influence of asiatic acid, madecassic acid, and asiaticoside on human collagen I synthesis. *Planta medica*, 60, 133–135 (1994)

Cat. No.	Brand	Product	Pack Size
39393	Fluka	Aloe-emodine	25 mg
53017	Fluka	α -Amyrin	10 mg
09236	Fluka	β -Amyrin	10 mg
44692	Fluka	Apigenin-7-glucosid	5, 25 mg
43191	Fluka	Asiaticosid	1,5 mg
55561	Fluka	Aucubin	5, 25 mg
01338	Fluka	Bergamottin	5 mg
74581	Fluka	Boldin	25, 100 mg
88527	Fluka	Citropten	5, 25 mg
28078	Fluka	9,11-Didehydroestriol	1, 5 mg
09258	Fluka	Erythrodiol	10, 50 mg
59780	Fluka	DL-Kavain	500 mg, 5 g
78564	Fluka	Ginkgolid A	10,50 mg
05108	Fluka	Ginkgolid B	10,50 mg
44349	Fluka	Ginkgolid C	5,25 mg
97151	Fluka	Hederacosid C	10, 50 mg
36483	Fluka	Loganin	10, 50 mg
09237	Fluka	Myristicin	10, 50 mg
69249	Fluka	Quercetin dihydrate	1, 5 mg
17799	Fluka	Rhamnetin	1, 5 mg
44699	Fluka	Rosmarinic acid	50, 250 mg
78095	Fluka	Rutin trihydrate	25, 100 mg
75412	Fluka	Sennosid A	25 mg
68909	Fluka	Sennosid B	5,25 mg
78666	Fluka	Taxifolin	25, 100 mg
72477	Fluka	Thymol	500 mg
18143	Fluka	trans-Nerolidol	100 mg
55608	Fluka	Vitexin-2"-O-rhamnoside	5, 25 mg

Table 1:
 Novel Phytopharmaca
 Standards

Volumetric Solutions traceable to NIST

Introduction

We offer a wide range of reagents for titration (Figure 1):

- Acidimetric titration /alkalimetric titration
- Redox titration
- Argentometric titration
- Complexometric titration (IDRANAL®- solutions)

Figure 1:

Fixanal ready to use solutions and concentrates



Please note, all of them are traceable to **EMPA/BAM and/or NIST**.

Most used materials are offered as Ready-to-Use Solutions as well as FIXANAL® concentrates.

The increasing automation and the escalating requirements of quality monitoring systems now available on the market has created a need for ready-to-use solutions for many applications. In comparison with FIXANAL® concentrates, the ready-to-use solutions yield, higher titer precision and can be used immediately. The advantage of the more economical FIXANAL® concentrates is the space-saving storage and, most important, its great flexibility. If you prepare the content of a sodium thiosulfate ampoule containing 0.1 mol (Cat. No 38200: 24.318 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5 \text{H}_2\text{O}$) using a volumetric flask of 1-liter, you'll get a solution of 0.1 mol/l. For a volumetric flask of 0.5-liter the result will be 0.2 mol/l, and for a volumetric flask of 5-liter the final concentration will be 0.02 mol/l.

Titer precision at 20°C:

FIXANAL®-Concentrates: $T=1.000\pm 0.2\%$

Ready-to-Use Solutions: $T=1.000\pm 0.1\%$

Apart from the customary package size up to 5-liter, the Riedel-de Haën range also offers a special 10-liter VOLPAC® package, when larger quantities of ready-to-use solutions are needed. These containers consist of a rugged cardboard cube containing a flexible inner bag made of polyethylene with an outlet tap (Figure 2). As the solution is withdrawn, the polyethylene bag col-

lapses, therefore preventing any contamination by the air from the laboratory. Also, there is no growing room for evaporation and condensing of water, and the consequent increase in the error of the titer. Other advantages of this VOLPAC® package are the small storage space requirement, simple handling, low volume of packaging material and easy disposal.

Certified Volumetric Solutions

Today there are several different suppliers offering volumetric reagents. Unfortunately, the concentrations stated on the labels are not always correct. You can often find a concentration without accuracy or wrong information regarding the traceability. Nevertheless, the values on the labels are uncritically integrated in the measurement and evaluation processes.

It gets more and more important to have reliable, correct and comparable values.

For an analytical laboratory to assure the traceability of their reagents is an elaborate and difficult process. In order to help the Science Workers with this laborious task, we are proud to present reliable and favourable volumetric solutions traceable to EMPA/BAM.

What does traceable to NIST mean?

NIST is responsible for developing, maintaining and disseminating national standards and also the realizations of the SI for the basic measurement quantities and derived measurement quantities. NIST is also responsible for assessing the measurement uncertainties associated with the values assigned to these measurement standards. For more information, please check NIST Web page on www.nist.gov/traceability/

What are EMPA/BAM Certified Reference Materials?

EMPA (Swiss Federal Laboratories for Materials Testing and Research) is a the research institute within the Swiss Federal Institute of Technology. For more information, please check EMPA Web page on www.empa.ch

BAM (Federal Institute for Material Research and Testing). For more information, please check BAM Web page on www.bam.de

After having agreed to the criteria of EMPA for the value assignment, FLUKA produces the reference material according to ISO Guide 34 and any further requirements defined by EMPA. EMPA controls the batch for its homogeneity and approximate content before the material is released for bottling and packaging. The certification analysis is performed by EMPA and BAM using randomly chosen bottles. Two different methods of measurement are applied, and the results are combined to obtain the certified value.

Long term stability tests are carried out by EMPA in parallel. A certificate is only issued when all criteria, including stability, are met. Then the titrimetric standards and the twelve element standard solutions are certified.

EMPA and BAM are similar institutions to NIST, and are officially accredited to certify standards. The resulting certification is the one stated by ISO Guidelines 30–35.

The EMPA/BAM certified reference standards have two important advantages:

- There is a strict separation between manufacturers and institutes for certifications.
- Two independent methods are used from two independent institutes.

These certified standards are also used for analysing our volumetric solutions.

Example of a Certified Volumetric Solution

All solutions are produced using quality grade *puriss. p.a.* raw materials. The production and the quality control of the volumetric solutions are always handled with extreme care and accuracy. The reliability of the equipment and the quality of the products are continuously monitored by means of a system of in-process controls and final checks. Therefore the FIXANAL® concentrates are packaged with the aid of automated filling lines.

In order to produce, for example, a 0.1 mol/l HCl solution, a 37% hydrochloric acid is diluted using monitored demineralized water to a titer (factor) of 1.000 ± 0.001 . For the analysis, we always use different titrators or titroprocessors to adjust the titer against a certified reference material. Whenever possible we use an EMPA/BAM certified reference material, which is often compared with NIST standard reference material during the certification process. This guarantees that the reference material is tested and certified with the know-how of several different institutions with established worldwide reputations.

In the case of hydrochloric acid, we use either TRIS (tris(hydroxymethyl)aminoethane) or sodium carbonate to determine the titer. The material that was actually used for the determination of the titer is displayed on the certificate of analysis for each lot. In **Figure 3** you may find an example of the Certificate of Analysis that is sent with each product.

The permission for filling and packaging a lot is given only if two analysts have found matching results using different analytical equipment for the measurement of at least 2 triple determinations. Of course we check the balances, electrodes or thermometers and calibrate our volumetric equipment in a regular basis, but the well-established double analysis guarantees that any small or slowly increasing faults of measurement are detected.

In case of the ready-to-use solutions, the measured titer (factor) is displayed in the certificate of



Figure 2:
VOLPAC® package

analysis, but for the FIXANAL®-concentrates we take the opportunity to adjust the titer to 1.000 during the filling process. The production is similar to the ready-to-use solutions, only the concentration is considerably higher (e.g. about 1.4 mol/l for a FIXANAL® of 0.1 mol), which has the advantage of a smaller volume. The titer is also determined with a precision of $\pm 0.1\%$, but then the weight of the quantity of liquid corresponding to a content of 0.1 mol is calculated. This quantity of liquid is filled in every ampoule – as precisely as possible. The filling is done with the aid of automated filling lines under weight control, in order to exclude faults resulting from the change of temperature or the formation of bubbles. The variation of the resulting weights is smaller than 0.1%, the typical standard deviation is lower than 0.04%.

The concentration of the liquid inside a FIXANAL® ampoule may vary a little bit from lot to lot, but the content of the declared material is exactly the same: e.g. 0.1 mol HCl (not mol/l), and so you are free to produce the volumetric solution you need by preparing the contents of an FIXANAL® ampoule in different volumes – having the certainty that it is traceable.

For product list, specifications and more detailed information, please check
www.sigmaaldrich.com/fixanal

If you need detailed information about these products, please feel free to contact us at:

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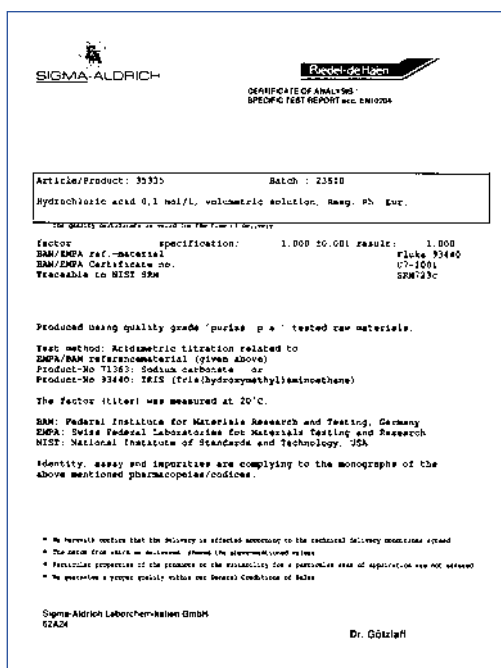


Figure 3:
Sample of Certificate of Analysis that is sent with a certified volumetric solution

Returnable Shuttle Drums for Solvents

The best Protection of the Environment, increased Safety at Work

There are many advantages when using stainless steel returnable shuttle drums for solvents in laboratories and in production:

- there is no packaging that needs to be disposed of
- there is no need to wash out the empty packaging materials and remove the label before disposal,
- there is also no need to dispose of the rinsing fluids.

Not having to dispose of waste packaging materials already makes a significant difference in reducing costs and working time taken.

Furthermore, stainless steel packaging maintains the quality of the solvent better than glass, plastic or aluminium. This detail is of extreme importance for solvents having high levels of purity, such as those used for spectroscopy, HPLC or sample preparation for gas chromatography. The stainless steel returnable shuttle drums have another major advantage in terms of safety at work: they are unbreakable and non-flammable.

7-litres, 18-litres, 45-litres and 200-litres. The 7-litre container is widely used as a supply drum in laboratories, since it is very handy. It is also possible to use these drums for alternative grades and these can be customised to suit requirements.

Dispensing the Solvent

The solvent can be dispensed with all standard pump systems and dispenser sets. However, only our specially developed dispensing systems guarantee an optimum working safety as well as the maintenance of quality. The dispensing system developed and marketed by Riedel-de Haën is made out of stainless steel. The sealing materials are made exclusively with PTFE and perfluorinated elastomers. A built-in dipped tube reaches down to the very bottom of the container. A small degree of overpressure forces the solvent upwards through the tube to the dispensing tap. This overpressure can be produced either by a rubber pump ball or by connecting the dispensing system to a pressured bottle of inert gas.

The dispensing tap is a lever in the case of the classical dispensing system, and is simply moved for opening and closing. The new dispensing system from Riedel-de Haën for the 7-litre stainless steel returnable drum is equipped with a self-closing valve. This prevents uncontrolled release of solvent. The non-return valve guarantees optimum working safety.

Thanks to our many years of experience, we can also provide help at any time with individual queries. We will, of course, provide support in the integration of the returnable systems into existing units or in the design at a fixed installation for reusable components.

The goal at Sigma-Aldrich is to work with you as our partner on the use and development of reusable solvent supply systems that are better ecologically and safer overall.



The Containers

Sigma-Aldrich's brand Riedel-de Haën was the first to offer returnable shuttle drums for the laboratory chemicals market. These containers and the dispensing systems have been constantly developed since their introduction in 1987 and the range of solvents offered has been expanded. Today most of our high-purity solvents are packed in a fully welded packaging made of stainless steel. They are available in sizes of



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For his works in the determination of water in food, he received the International Hydranal-Prize from Riedel-de Haën.

How to Determine Water Content in Foods?



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Water Determination in Food

Water is present in every food, in a range that may vary from extremely low values in dried products to extremely high ones in beverages. Its content is of great significance in many respects, affecting properties like conductivity for heat and electrical current, density and rheological behavior, or even corrosiveness. All these factors must be taken into account for the design of technological processes. The amount of water in food often determines its nutritive value and taste. However, in some cases it can be considered as an impurity. In reference materials, the water content is important as far as specifications are given on the basis of either dry matter or initial mass. The stability and the shelf life of foods are highly dependent on water content, since it is crucial for microbiological life and most enzymatic activities. Storage volume and mass depends also on water content, which can be reflected in transport costs. As water is relatively cheap, its presence in foods in general, and in expensive products in particular, is of commercial interest. For this and other reasons, rules and regulations concerning water content are imposed. As a consequence of all the above, the determination of water content is certainly the most frequent analysis performed on foods. Water occurs in different bonding situations, which have a direct effect on its separability from the matrix and therefore, on its detection. For instance, bulk water is easy to determine. This «free water» is the one that can be found in gaseous and most liquid mixtures, and also at the





outer surface in big cavities or large pores of solid products. Its separation and detection presents no problem. However, the detection of water existing in capillary interstices and adsorbed at the surface in the form of oligomolecular layers is far more complicated. The chemical detection of this fraction is still feasible, but the heat required to separate it from the matrix increases. This makes it less accessible to physical separation methods. For the «last» and very tightly bound monolayer water, the separation energy becomes very high. Such high values of energy will usually lead to decomposition reactions.

Classification of Methods for Determining Water Content

There are several methods to determine the water content of foods [1, 2], which may be classified in different groups. Direct or primary methods are those leading to a quantitative determination of water as such. The physical techniques measure the amount of water obtained after extraction, or the mass loss after separation of water from other components. Apart from physical methods, there are chemical methods, based on a selective reaction of water with specific reagents. Both techniques may be combined by chemically determining the water obtained by previous separation. Indirect or secondary methods may either determine a given macroscopic property that depends on water content (e.g. density, optical activity, refractive index or electrical properties), or measure the effect caused by physical parameters, as happens in low-resolution magnetic resonance measurements, microwave spectroscopy or near infrared spectroscopy. Indirect methods rely on a direct method since the measured entity does not depend only on the water content, but also on other components. In addition, the response may depend on the «degree of liberty» of the water molecules and on the occurrence and frequency of the various bonding states. Therefore, the indirect methods require a calibration against a direct method.

Based on our own experience, in this article we shall focus on direct methods: Karl Fischer titration as a chemical technique and drying techniques as a physical approach.

Karl Fischer Titration

By far, the most important method for chemical determination of water is the Karl Fischer titration [3]. The original interpretation of this selective reaction proved to be incorrect. Methanol, which had been used purely by chance by Karl Fischer, is not only a solvent but also a participant in the reaction [4]. Pyridine is not necessary for the reaction [5, 6] and can even be a disadvantageous base to use due to its low basicity (see reaction scheme below). Pyridine has been replaced by other bases, in particular by imidazole [7]. Methanol can be replaced by other alcohols

either for general [9, 10, 11, 12] or special applications [8].

In protic solvents, such as alcohols, the Karl Fischer reaction follows a two-step scheme [6, 13, 14, 15]. In aprotic solvents, the reaction takes another pathway [15, 16]. In protic surroundings, the first step is the esterification of an alcohol (ROH , normally methanol) with sulphur dioxide; to obtain a quantitative reaction, the ester is neutralized by a base Z to yield alkyl sulphite (**Eq. 1a**). In modern reagents, the «classical» (and relatively weak) pyridine has been replaced by more appropriate bases, such as imidazole. In the second step, alkyl sulphite is oxidized by iodine to give alkyl sulphate in a reaction that requires water. The base, again, provides a quantitative reaction (**Eq. 1b**):



Overall Reaction:



The consumption of iodine is measured. In the coulometric variation of the Karl Fischer titration, iodine is formed from iodide in the titration cell by anodic oxidation. In the volumetric variation, which is more relevant in food analysis, a solution of iodine is added. The sample is placed in the titration cell containing working medium previously titrated to dryness. In the so-called one-component technique, this working medium consists of methanol and the titration solution contains all the other chemical components: iodine, sulphur dioxide and the base in an appropriate solvent. In the two-component technique, the working medium contains sulphur dioxide and the base dissolved in methanol, and the titration agent is a methanolic solution of iodine. The water equivalent of the titrating solution is determined by titrating a known amount of water. For instance a certain volume of a standard with a known water content can be used for this purpose.

The end-point indication, both for the coulometric and volumetric methods, is based on an electrochemical effect. Two platinum electrodes, submerged in the working medium contained in the titration cell, are polarized either by a constant current (voltametric technique) or a constant voltage (amperimetric technique). The voltage or the current, respectively, to maintain this situation is monitored. Once the water is depleted, iodine can no longer react and the redox couple iodine/iodide is then present, making its oxidation and reduction possible. This causes abrupt changes in the voltage necessary to maintain the constant current (voltametric technique) or the current resulting from the constant voltage (amperimetric technique). This drop (voltametric technique) or rise (amperimetric technique) is used to indicate the end point. When the voltage

or the current remains respectively below or above a chosen value during a certain time, the determination is completed. This so-called stop delay time allows the detection of water that may not be immediately available. This is the case for samples which are not completely soluble, or even insoluble, in the working medium. In these cases water reaches the working medium by diffusion and extraction processes only with a certain delay. Another way of determining the end point is the so-called drift. Traces of water intrude into the titration cell through joints and tubes, at a rate that can be registered before starting to titrate. The determination stops when the addition of reagent has reached the rate of the drift measured before.

Karl Fischer titration requires water to be in direct contact with the reagents, which may lead to some difficulties with insoluble samples. There are several ways of circumventing this limitation [17, 18, 19]. They comprise a long stop delay time, the external extraction of the water and titration of an aliquot of this solution, the internal extraction in the titration vessel prior to the start of the titration, the reducing of the particle size by external sample preparation or by using a homogenizer in the titration cell [20], working at elevated temperatures [7 (9), 21], even at the boiling point of the working medium [22, 23], the addition of solvents to the working medium or the replacement of methanol by other alcohols in order to change the polarity [9, 10, 11, 12]. In this case, the electric parameters (polarizing current or voltage, and end-point voltage or current) should be adapted [10, 11].

In other cases, it may not be desirable to detect the total water content but only the adherent or surface water. For example, this characteristic is important for the flowability of a product like sugar. The complete dissolution of the sample may be avoided by adding an «unproprate» solvent to the working medium, working at lower temperatures and applying adequate stop criteria [24].

Chemical interferences with the Karl Fischer reaction have been described. These may be caused by a cross reaction of the components contained in the sample, or from their reaction with components of the Karl Fischer reagents to form or to consume water. An example are esterifications of acids with methanol or the formation of acetals or ketals of carbonyl compounds with methanol. Another source of interference occurs when iodine reacts with compounds in the sample and is not available for the Karl Fischer reaction. When the exact amount of the interfering substance and the stoichiometry of the reaction are known, the error can be calculated [25]. In many cases the interference can be avoided [17, 18] by replacing methanol by less reactive alcohols [8], working at low temperatures or by a pre-titration with an iodine solution in the absence of sulphur dioxide [25].

Drying Methods

Oven Drying

The most frequently applied method for water or moisture determination is the measurement of the mass loss that the product undergoes by a heating process. Drying techniques with convective heating are ordinary oven drying and vacuum oven drying.

It must be pointed out that drying techniques do not measure the water content as such. The result is the mass loss under the conditions applied. These conditions can be freely chosen and the results can be variable. Even in the official methods the parameter set is only a convention, and the results do not necessarily reflect the true water content. Drying to a nearly constant mass is often required. But only in rare cases is a real constancy achieved. Tightly bound water may escape detection, but a distinction between «free» and «bound» water is not possible since a clear definition of these fractions can hardly be given [26]. The higher the temperature applied, the more water will be detected and be considered as «free». The mass loss is not only caused by loss of water but by the loss of all volatile substances under the drying conditions, comprising those already contained in the original sample and those produced by the heating process. The application of low pressure in vacuum ovens reduces the danger of producing volatile decomposition compounds but does not allow a distinction to be made between water and other volatile substances already present in the product. Based on this, the results of drying methods should not be called water content. The term «moisture» is often used, although it is commonly used as a synonym of water. The correct term for the result of a drying method is «mass loss», and, since it depends on the conditions applied, these should be included with the results.

In spite of their inherent problems, determination of water using drying techniques is not only widely used as a conventional method but also compulsory for certain products. The advantage of this technique is that it can be carried out in most analytical laboratories and that, when the set parameters are respected, it usually gives reproducible results. Another advantage is that several samples can be analyzed in parallel. However, it should be mentioned that these samples are not true replicates in a statistical sense. This is due to the fact they are treated in parallel, at the same time under the same conditions within one experiment and therefore, are not repeated measurements.

The discrepancy between water determination by Karl Fischer titration and mass loss determination by drying techniques is illustrated by the following example [27].

The water content of milk powder is determined by drying according to a standard method des-





cribed by the International Dairy Federation [28]. 1–3 g of the test sample are dried at 102 ± 2 °C under atmospheric pressure for 2 h. Drying is continued in steps of 1 h until the difference in mass does not exceed 0.5 mg. This method is referred to as **DM1** in the following.

In order to find a better method, other drying methods and the Karl Fischer titration were proposed and tested. In drying method 2, **DM2**, a controlled stream of pre-dried air is pumped through the sample containers to dry 5 ± 0.3 g of the sample for 5 h. Mass constancy is not controlled [29, 30]. In drying method 3, **DM3**, a controlled stream of nitrogen at 102 ± 2 °C is pumped through glass tubes at a residual pressure of 100 mbar for 2 h to dry 2–3 g portions of sample in sample containers. Samples and sample containers are then cooled in the closed glass tubes. Mass constancy is not checked [30]. A comparison with the Karl Fischer titration, **KFT**, was also made. In a pilot study, beside milk powder samples, α -lactose monohydrate was also tested because it occurs in milk powder in relatively high amounts. It contains 5 g water of crystallization per 100 g. The drying methods were carried out by five laboratories, the **KFT** only by four. **Table 1** (taken from [30]) gives the results.

The Karl Fischer result seems to be the total water content, comprising both the crystallized water and the additional surface water. The drying methods obviously do not detect the overall water content nor the surface water only. The mass loss depends very much on the conditions applied.

In a following ring test with eight participating laboratories, three skimmed milk powders and three whole milk powders were analyzed for water content. **Table 2** (taken from [30]) gives the results.

As could be expected from the results for lactose monohydrate, the mass loss by drying is less than the water content determined by Karl Fischer titration. The drying methods do not reflect the true water content or present the content of free water.

To shorten the long determination times of several hours in drying ovens with convective heating principle, more efficient heating sources have been introduced. The samples are exposed, on the pan of a built-in balance, to infrared (or «halogen») or microwave radiation and the loss in mass is registered. Such measurements take only a few minutes. Handling is very simple and in many cases no sample preparation is necessary.

Infrared and «Halogen» Drying

The apparatus used for infrared drying have different modes of operation. In automatic ones, the analysis is stopped when – at a chosen temperature or heating level – the mass does not change by more than a certain amount within a given time interval. In the ideal case, a practically constant mass is reached. In other modes, the drying time at a chosen temperature or power level – sometimes in different steps – can be specified.

Also, various infrared sources, also called halogen dryers, are in use. The differences between them are their emission spectra, power and temperature. The maximum of the emission spectrum moves to shorter wavelengths with higher temperatures. This is in agreement to the reverse proportionality established by Wien's Law, and it must be taken into account to obtain a maximum efficiency of infrared heating processes. Highest efficiency of heating is attained when the maximum of the emission spectrum corresponds to the maximum of the absorbance spectrum of the sample; in the case of drying processes this corresponds to the best absorbance of water in the range of 3–3.5 μm [31]. Thus, it is not surprising that dryers with different infrared sources need to operate at different temperatures to give the same results [32].

The more intensive heating in infrared dryers as compared to usual drying ovens makes samples even more susceptible to decomposition reactions. Therefore, more volatile matter is produced and a higher water content of the sample, which may not correspond to reality, is obtained.

The drying parameters must therefore be adapted to the type of sample. Depending on the chosen parameters, the results can vary within a wide range. To select suitable drying conditions, a calibration procedure should be established for every product or product group. In some cases, it is possible to find a temperature that allows the automatic mode to be used. In other situations, for instance, for heat-sensitive products that undergo continuous decomposition, it may be better to impose a definite time at a particular temperature. The aim of calibration is to match the mass loss measured by infrared drying with the results of any reference procedure by determining appropriate operating parameters, such as program mode, drying time and temperature or, if available, the temperature program. Good results are obtained [32, 33, 34, 35] even if the true water content is not always measured as such. For instance, this is the case of when two main (and conflicting) sources of error (unde-

Table 1 : Determination of water content or mass loss of α -lactose monohydrate (n replicates) by different methods. Repeatability standard variation: S_r ; reproducibility standard deviation: s_r .

Method	Mass loss/water content [g/100 g]	n	S_r [%]	s_r [%]
DM1	0.76	49	0.25	0.54
DM2	0.51	50	0.15	0.40
DM3	2.71	50	0.27	0.53
KFT	5.09	40	0.023	0.054

Table 2: Determination of water content (WC) or mass loss (ML) of milk powder samples (n replicates) by different methods. Repeatability standard variation: S_r , reproducibility standard deviation: s_r .

Skimmed milk powders					Whole milk powders						
	Method	n	WC or ML [g/100 g]	S_r [%]	s_r [%]		Method	n	WC or ML [g/100 g]	S_r [%]	s_r [%]
Sample 1	DM1	80	3,72	0,081	0,18	Sample 4	DM1	80	3,21	0,057	0,16
	DM2	80	3,62	0,052	0,058		DM2	80	3,16	0,035	0,060
	DM3	80	3,99	0,084	0,10		DM3	80	3,43	0,059	0,081
	KFT	80	4,16	0,063	0,083		KFT	80	3,55	0,050	0,072
Sample 2	DM1	80	3,74	0,092	0,18	Sample 5	DM1	80	2,57	0,069	0,16
	DM2	80	3,57	0,085	0,097		DM2	80	2,52	0,045	0,055
	DM3	80	3,99	0,11	0,12		DM3	80	2,80	0,069	0,094
	KFT	80	4,21	0,081	0,13		KFT	80	2,97	0,049	0,072
Sample 3	DM1	80	4,02	0,082	0,17	Sample 6	DM1	80	2,44	0,080	0,15
	DM2	80	3,93	0,053	0,074		DM2	80	2,38	0,049	0,098
	DM3	80	4,28	0,087	0,20		DM3	80	2,66	0,080	0,11
	KFT	80	4,46	0,066	0,11		KFT	80	2,80	0,057	0,093



tected water remaining in the sample, and the determination of other volatile substances) compensate each other. In other cases, it is not possible to obtain the exact reference value with satisfying reproducibility, but only a value close to it, which needs to be adjusted with a correction factor. Calibration must not be neglected, since the values obtained with a given set of parameters may be very reproducible, giving the misleading impression that a correct result has been obtained. An example is given in **Figure 1**. The drying curves reach practically constant values at several temperatures.

Another difference between infrared sources is their response time. The pause between measurements using sources that cannot change temperature quickly has a decisive effect on the results. Once a method has been established using a certain temperature for a set time and with a certain interval between consecutive measurements, the results may be different if other time intervals between measurements are used. The reason is that the dryer does not reach the same temperature during the programmed analysis time. This easily overlooked effect increases with higher temperatures, shorter determination times and increasing heat sensitivity of the sample [36].

Microwave Drying

The basic principle of microwave drying is similar to the infrared drying technique, where the sample is exposed to a drying process on a built-in balance. Usually, a frequency of 2450 MHz is used to excite the water molecules, to induce them to rotate and, eventually, to evaporate. The mass is continuously monitored and the stop criteria are variable.

The excitation frequency is not specific to water and other substances may also be heated. This can lead to their evaporation, decomposition to volatile matter, and to an increase in the temperature of the whole product, in a similar way to the food heated in household microwave ovens. All this put together can affect other substances that were not originally influenced by the microwaves. A calibration is therefore needed to establish a correlation with a reference method [35, 37, 38].

When the water content is lower than approximately 10 g/100 g there might be a risk of burning the sample at the end of the determination due to high temperatures. If such samples are to be analyzed, the apparatus should have the feature of limiting the microwave power in the final phase. This is possible by controlling the temperature and setting a maximum, or by checking whether the microwave energy is still absorbed. Such limitations should also be taken into account for very heat-sensitive samples [39].

Summary

The Karl Fischer titration is a method for determining the water content, based on a selective chemical reaction. Difficulties may arise for samples that are insoluble or not completely soluble in the working medium. In such cases measures have to be taken to release the water from the matrix. They comprise sample preparation, titration conditions and operating parameters.

Drying techniques are very often used for determining water content. They lead to mass loss of the sample under the conditions applied. All the components that are volatile under these conditions, including those that might be produced by the heating process itself, contribute to the mass loss. Therefore, the result corresponds to water content only when there are not any other volatile compounds, the water is completely released, and when the product does not undergo decomposition reactions during the drying process. There is a high risk of decomposition when the drying is carried out using more efficient heating principles than convective heat

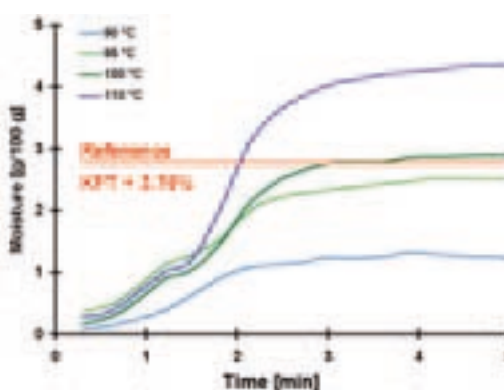


Figure 1: Infrared drying curves of candies at different temperatures (KFT: Karl Fischer Titration)



transfer in classical drying ovens. Such methods are infrared, «halogen» or microwave drying. They can, however, yield the water content if the parameters are chosen in an appropriate way. Since products will behave differently from each other when they are exposed to the respective energy input, calibration against a reference method must be established for each product or product group.

To ensure that the real water content is determined, the calibration of drying methods should preferably be done using the Karl Fischer titration as a reference. Along with its use as a direct analytical method, this is an important use of the Karl Fischer titration. Its use as a reference method is not only relevant for the drying techniques discussed here, but also for indirect methods like near infrared spectroscopy, low-resolution nuclear magnetic resonance measurements or microwave spectroscopy.

In conclusion, the Karl Fischer titration is not only the most important and reliable direct method to determine the water content of foods in a selective manner, but also, through its role as a reference method, the basis for other techniques that require calibration.

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Rapid Water Analysis with AQUANAL®



Under our **AQUANAL®** trademark, we are proud to offer you an extensive product range of ready to use test kits (colorimetric or titrimetric), as well as compact labs and photometers for the rapid analysis of drinking, feed, process and slightly polluted waste water. The tests were developed according to the well-established principles of the German Standard Methods. This product line is constantly being expanded and modified to meet new requirements.

AQUANAL®-plus

The product line **AQUANAL®-plus** consists of approximately 40 test kits with the corresponding refill packs and check solutions for on-the-spot analysis of water samples. The analysis can be done by using the color chart included in the test kit, or the Spectro 2 Photometer for **AQUANAL®-plus** reagents.

The tests are:

- **fast:** results within a few minutes
- **practical:** measuring range can be increased by use of dilution water
- **refillable:** reagents, check solutions, dilution water and accessories can be ordered separately
- **economical:** cost effective

AQUANAL® -plus Spectro 2 Photometer

This microprocessor-controlled LED portable photometer for rapid water analysis was specially designed to be used with **AQUANAL®-plus** Test Kits. The battery-powered photometer has four light sources (480 μm , 565 μm , 585 μm , and 635 μm) and is suitable for use in the field. Please take a look at our product list (Cat. Nos 37781 to 37790).

Our detailed product list is available on www.sigma-aldrich.com/aquanal or per request on aquanal@sial.com

AQUANAL®-plus Test Kits, refill packs, standard solutions

AQUANAL®-plus Tube Tests

The **AQUANAL®-plus** Tube Tests were developed for on-site analyses, for instance, in a sewage treatment plant. They provide fast results under safe and clean operating conditions. Each Tube Test Unit is suitable for 25 tests.

A pipette is used to dispense the sample into the tube containing the test reagent. For some tests, an additional reagent has to be added. Once the reaction is over, the results may be obtained using one of our photometers. You may choose between Spectro 2, Spectro CSB/COD photometer (only for COD tube tests) or **AQUANAL®-professional Spectro 1000**.

The **AQUANAL®-plus** Tube Tests cover the following substances in waste water:

Ammonia (Cat. No 37739) occurs in surface water, and is also found in communal or industrial waste water. The test determines the ammonia concentration, and a correction factor is used to calculate the concentration of ammonium nitrogen. The result is obtained using either the Spectro 2 photometer or the **AQUANAL®-professional Spectro 1000**.

The **COD** (Cat. No 37736, 37737 and 37738) value is a measurement of the quantity of organic pollutants present in water. The COD is determined by oxidizing the substances with calcium dichromate. We offer three different ranges for this test. Results can be obtained by using the Spectro 2, the Spectro CSB/COD photometer or the Spectro 1000.

The heating unit required for this test is also available (Cat. No 37571).

Nitrite (Cat. No 37741) is a product of the nitrification of ammonia, particularly in low-oxygen water. Nitrite can be toxic for organisms even in low concentrations. Once the nitrite concentration has been determined, then N-nitrite can be calculated using a correction factor. The results can be obtained using the Spectro 2 photometer.

Total-P and Orthophosphate (Cat. No 37743, 37744 and 37745): Phosphorus can be found in waste water in various forms. One of these is orthophosphate that can be determined as a phosphate, without decomposition of the sample. Total phosphates include other organic and inorganic phosphate compounds. This determination requires the use of a decomposition reaction. All of these forms should be included in the study of process water. The tests yield the concentrations of phosphate, then a factor is used to calculate the concentration of phosphorus. The result is obtained using the Spectro 2 photometer.

Nitrate: (Cat. No 70107) Due to the high load of nitrates in the environment (NO_x from the air, fertilizers), relatively high nitrate concentrations can be found in potable water. Nitrate is micro-biologically transformed to nitrite, which is an

indicator of the pollution of a body of water. The result of this tube test can only be obtained by using the Spectro 1000.

Total Nitrogen (Cat. No 70108): Nitrogen is mainly found as nitrite, nitrate and ammonia. These compounds are important in terms of water treatment and waste water technology. Depending on the prevailing conditions (i.e., low or high oxygen values), their concentrations fluctuate. Total Nitrogen is also determined to check the influence of the pH-value or the oxygen concentration. The result of this test can only be obtained by using the Spectro 1000.

AQUANAL®-plus Vario Basic Case (Cat. No 37553)

Vario Case consists of

- five test sets or
- four test sets of your choice, plus one Spectro 2 photometer

The tests can be chosen from the **AQUANAL®-plus** product range.

The full price depends on the chosen tests, and the case. The total hardness reagent, as well as the pH-indicator sticks, are sent free of charge.



Figure 1 AQUANAL®-plus Compact Lab

AQUANAL®-plus Compact Lab (Cat. No 37562)

The **AQUANAL®-plus** Compact Lab was developed to provide rapid analysis of water directly on-the-spot at which samples are taken (Figure 1). The case contains all the reagents needed to carry out the trace analysis for each contaminating substance listed in Table 1 as well as the determination of total hardness and pH. Laboratory equipment needed for these analyses (syringes, funnels, filters, collecting bottles...) is also included. There is also room for the Spectro 2 photometer required for these tests. The **AQUANAL®-plus** Compact Lab is ideally suited to meet the requirements of fire brigades, the police and environmental protection officers of local authorities, and is also suitable for use in schools.

We offer a wide range of special assorted compact labs:

AQUANAL®-Ökotest Water Laboratory (Cat. No 37557)

AQUANAL®-EduCase (Cat. No 37518)

AQUANAL®-Fishwater Lab (Cat. No 37566)

AQUANAL®-Analytical case for building restoration (Cat. No 37563)

AQUANAL®-Pool tester chlorine/pH (Cat. No 37546)

Table 1:
AQUANAL®-plus
Compact Lab
(Cat. No 37562):
available tests and
measuring range

Substances	Measuring range (mg/l)	Number of tests
Ammonium	0.2 – 8.0	125
Chloride	5 – 300	150
Chlorine	0.01 – 0.3	300
Chromium	0.005 – 0,1	100
Copper	0.05 – 4.5	75
Cyanide (total)	0.03 – 0.7	200
Iron	0.2 – 15	500
Nickel	0.02 – 0.5	60
Nitrate	1 – 50	200
Nitrite	0.005 – 0.1	200
Phenol	0.1 – 3.0	300
Phosphate	0.02 – 0.4	100
Total hardness	1 drop = 1°dH = 0.178 mmol/L Ca	75
Oxygen	1 – 12	30
Sulfate	50 – 330	100
Sulfide	0.03 – 0.6	100
pH-value	4,5 – 9	100



Figure 2:
Use of Vario H Powder Packs

Table 2: Product Range – only for use with Photometers of the HACH company

Cat. No	Product Name	Determination Range	HACH Cat. No
70235	Vario H Acid HR Silica A	Silica 0–100 mg/l	21074-69
70241	Vario H Acid HR Silica B	Silica 0–200 mg/l	1042-99
70222	Vario H Ammonia Cyanurate	Nitrogen 0–0,5 mg/l	23954-66
70223	Vario H Ammonia Salicylate	Nitrogen 0–0,5 mg/l	23952-66
70236	Vario H Citric Acid A	Silica 0–100 mg/l	21062-69
70242	Vario H Citric Acid B	Silica 0–200 mg/l	14548-99
70203	Vario H Copper 1	Copper 0–5 mg/l	21058-69
70200	Vario H DPD Total Chlorine 10	Chlorine 0–2 mg/l	21056-69
70112	Vario H DPD Free Chlorine 10	Chlorine 0–2 mg/l	21055-69
70202	Vario H DPD Total Chlorine 25	Chlorine 0–5 mg/l	14064-99
70113	Vario H DPD Free Chlorine 25	Chlorine 0–5 mg/l	14070-99
70205	Vario H Iron	Iron 0–3 mg/l	21057-69
70208	Vario H Manganese Citrate Buffer	Manganese 0–20 mg/l	21076-69
70212	Vario H Moly 1	Molybdate 0–35 mg/l	26042-99
70213	Vario H Moly 2	Molybdate 0–35 mg/l	26043-99
70217	Vario H Moly 3	Molybdate 0–35 mg/l	26044-99
70237	Vario H Molybdate HR Silica A	Silica 0–100 mg/l	21073-69
70243	Vario H Molybdate HR Silica B	Silica 0–200 mg/l	1041-99
70218	Vario H Nitrite 10	Nitrite 0–0.2 mg/l	21071-69
70219	Vario H Nitrite 25	Nitrite 0–0.2 mg/l	14065-99
70229	Vario H Phosphate	Phosphate 0–2.5 mg/l	21060-69
70207	Vario H Sodiumpersulfate	Manganese 0–20 mg/l	21077-69
70244	Vario H Sulfate	Sulfate 0–70 mg/l	21067-69
70204	Vario H TPTZ Iron	Iron 0–1.8 mg/l	26087-99

AQUANAL®-professional

NEW! AQUANAL®-professional. These products offer you many choices for the determination of impurities in water. Most are tablet tests that guarantee easy handling. Optimal results can be achieved with the Spectro 1000 photometer. The **AQUANAL®-professional** Tube Tests may be used in most laboratories, such as those found in sewage water treatment plants. They provide fast results under safe and clean conditions. Results are obtained with the Spectro 1000, as the Spectro COD is suitable only for the evaluation of COD tube tests.

AQUANAL®-professional Vario H reagents are provided in powder packs that make handling as convenient and exact as possible.

AQUANAL®-professional Vario H Powder Packs

Vario H Powder Packs were specially designed to be a replacement for HACH powder pillows. The use of dry powdered reagents minimizes the problems associated with leakage and deterioration. The powdered product is packed in individual, pre-measured foil packs (**Figure 2**). Optimal results can be achieved using HACH photometers.

Take a close look: Free and Total Chlorine Test

Free Chlorine

Free chlorine (hypochlorous acid and hypochlorite ion) oxidizes DPD, a reaction that is pH dependent. Between pH 6.3–6.5, the reaction between DPD and free chlorine forms a magenta-colored compound. Both Vario H Powder by Sigma-Aldrich and HACH Powder are able to handle high levels of hardness without precipitation.

Total Chlorine

Potassium Iodide is used to determine combined chlorine forms. Chloramines oxidize the iodide to iodine; the liberated iodine reacts with DPD. Both, AQUANAL® Vario H Powder and HACH Powder contains DPD, potassium iodide and a buffer system.

Ready-to-use Solutions for Waste Water Analysis

Ready to use solutions for waste water analysis according to German Standard Methods are listed by chemical name in the alphabetical part of our catalogue (e.g., mercury(II) sulfate solution, Cat. No 31014).

If there are any questions concerning our **AQUANAL®** products, please don't hesitate to contact us: we will be glad to help you! We are looking forward to receiving your inquiries at Aquanal@sial.com.

70012 – AQUANAL®-professional Spectro 1000

AQUANAL®-professional SPECTRO 1000 is a modern single-beam spectrophotometer with an excellent price/performance ratio that was specially designed for water testing. It is ideal for use in water and waste water analysis and can also be used with a wide range of other applications. The unit is equipped with a wide spectrum of pre-programmed methods based on the proven range of tablet reagents and liquid reagents. Besides all **AQUANAL®-professional Tests**, routine analyses made in rectangular boxes (from 1 cm to 5 cm) or in tubes (16 mm or 22 mm diameter) can be performed using the Spectro 1000. All results can be exported to your PC.

The Benefits

- Wavelength scan from 330 to 900 μm
- High accuracy and reproducibility
- Large graphic-display
- Multiple measurement capabilities
- Easy maintenance
- Easy to use
- For the determination of more than 50 water-analysis Parameters

70034- AQUANAL®-plus Spectro Turbidity (Cat. No 70034)

The **AQUANAL®-plus Spectro Turbidity** was designed as a compact, easy to use instrument for the fast and accurate determination of turbidity. A light emitting diode (LED) is used as a light source. The photodetector is positioned to detect light scattered by the sample at 90° to the incident beam. The LED is characterized by maximum long-term stability and monochromatic light emission with minimum power input. The sample chamber, which is the most critical part of any photometer, is fully sealed, so no water can penetrate into the electronic components. The size of the sample chamber ensures easy cleaning of the light entry surfaces. Battery operation makes the unit suitable for both mobile use and laboratory applications. It is supplied in a handy case as a fully functional unit, complete with accessories and calibration standards. Three different turbidity standard sets are available, Cat. No 70036 (1 and 10 NTU), Cat. No 70037 (100 and 1000 NTU), and Cat. No 70110 (1,10,100 and 1000 NTU).

Kit Content

AQUANAL®-plus Spectro Turbidity is supplied in a plastic case with round vials, calibration solutions (1,10,100 and 1000 NTU), accessories, instruction manual, warranty sheet and 9 V battery.

70109- AQUANAL®- plus Spectro HAZEN

One of the standard methods recommended by the ASTM to determine the color of lightly-colored liquids is the platinum-cobalt scale. This test method is also referred to as APHA color tests. The preparation of these platinum-cobalt standards was first described by A. Hazen. That is why the term *Hazen Color* is often associated with this test. The **AQUANAL®-plus Spectro Hazen** is specially designed for the determination of a large number of APHA color tests.

Technical Support

We will be glad to provide you any support in the analysis of your sample. Please feel free to contact our **AQUANAL®** application laboratory. Please don't hesitate to contact us at:

Ms. Petra Haubold

Technical Support **AQUANAL®**

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Email: Aquanal@sial.com

Table 3:
Product Range and
Specifications

Cat Number	Product Name	Description
70012	Spectro 1000	single-beam spectrometer (330–900 μm) suitable for all AQUANAL®-PROFESSIONAL tests 16 and 24 mm cyclic tubes 10 to 50 mm rectangular tubes
70016	Light source replacement for Spectro 1000	Pre-adjusted light source
70024	Cable connection (printer)	RS232 C, D9F-D25M for Spectro 1000
70026	Cable connection (PC)	RS232 C, D9F-D25F for Spectro 1000
70027	Cylindric cells	12 cylindrical tubes diameter 24 mm
70028	Cylindric cells	5 cylindrical tubes diameter 16 mm
70029	Rectangular cell	10 mm optical glass for Spectro 1000
70032	Rectangular cell	50 mm optical glass for Spectro 1000
70033	Rectangular cell	10 mm QUARTZ-UV glass for Spectro 1000
AQUANAL®-PROFESSIONAL REAGENTS		
100 tablets/unit for Spectro 1000		
70054	Alka-m-Photometer	range: 5–200 mg/l
70056	Alka-p-Photometer	range: 5–300 mg/l
70057	Ammonia no. 1	use with: Ammonium no. 2 range: 0.02–1 mg/l
70059	Ammonia no. 2	use with: Ammonium no. 1 range: 0.02–1 mg/l
70085	Copper no. 1	use with: Copper no. 2 range: 0.05–5 mg/l
70086	Copper no. 2	use with: Copper no. 1 range: 0.05–5 mg/l
70073	DEHA	use with: DEHA solution range: 0.02–0.5 mg/l
70075	DEHA solution	use with: DEHA tablets range: 0.02–0.5 mg/l
70061	DPD no. 1	determination of bromine, chlorine, chlorinedioxide and ozone
70062	DPD no. 1 High Calcium	determination of bromine, chlorine and ozone
70064	DPD no. 3	determination of chlorine and ozone
70084	Hardcheck P / total hardness	range: 2–50 mg/l
70076	Iron no. 1 LR	use with: iron no. 2 LR range: 0.01–1 mg/l
70077	Iron no. 2 LR	use with: iron no. 1 LR range: 0.01–1 mg/l
70087	Manganese LR 1	use with: manganese LR 2 range: 0.05–4 mg/l
70091	Molybdate no. 2	use with: molybdate no. 1
70245	Phosphate no. 1 LR	use with: phosphate no. 2 range: 0.05–4 mg/l
70095	Phosphate no. 2 LR	use with: phosphate no. 1 range: 0.05–4 mg/l
70102	Hydrogen peroxide LR	range: 0.01–1.5 mg/l
70070	Acidifying GP	determination of chlorine
70083	Calibration standard fluoride	use with: Spadns reagent range: 0.02–1.5 mg/l
70071	Chloride	range: 5–50 mg/l; liquid reagents
70068	Chlorine HR	determination of chlorine
70103	Copper/Zinc LR	use with: EDTA and Dechlorine range: 0.02–1 mg/l
70104	Dechlor	use with: EDTA and copper/zinc range: 0.02–1 mg/l
70063	DPD 1 Buffer solution	determination of bromine, chlorine and chlorine dioxide
70066	DPD 1 Reagent solution	determination of bromine, chlorine and chlorine dioxide
70067	DPD 3 Reagent	determination of chlorine
70105	EDTA	use with: copper/zinc and dechlorine range: 0.02–1 mg/l
70093	Glycine	use with: DPD no. 1, DPD n. 3 and DPD High Calcium range: 0.02–1 mg/l
70106	Nickel	range: 0.2–7 mg/l; liquid reagents
70094	Phenol Red	range: 6.5–8.4 mg/l
70096	Silica no. 1	use with: Silica no. 2
70097	Silica no. 2	use with: Silica no. 1
70081	SPADNS reagent	use with: calibration fluoride range: 0.02–1.5 mg/l
70098	Sulfide no. 1	use with: Sulfide no.2 range: 0.04–0.5 mg/l
70101	Sulfide no. 2	use with: Sulfide no.1 range: 0.04–0.5 mg/l



Rapid Water Analysis with AQUANAL®

Under our **AQUANAL®** trademark we are proud to offer you an extensive product range of ready-to-use test kits (colorimetric or titrimetric), as well as compact labs and photometers for the rapid analysis of drinking, feed, process and slightly-polluted waste water

NEW! AQUANAL®-professional.

Now available: more choices for the determination of impurities in water, including powder packs and tablet tests that guarantee easy handling. Optimal results can be achieved with the Spectro 1000 photometer.

Test a sample – free of charge!

Please indicate your choice on the form below and you will receive it in a few days.

If you need more information or application support, please feel free to contact our **AQUANAL®** application laboratory: Aquanal@sial.com

Ordering Form

I want to order one of the following products free of charge

- 37507-1EA-R AQUANAL®-OEKOTEST-WATER LABORATORY
- 37508-1EA-R AQUANAL® PLUS NITRIT
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