

Brighter water Determination

Aquastar™ reagents for
brilliant Karl Fischer titration results.

Aquastar™ Karl Fischer Reagents

Using Karl Fischer titration together with our Aquastar™ reagents, the water content of gases, liquids and solids can be determined easily, and with a high degree of accuracy. It is not without reason that no other method is as widespread as this one: Determination of water content according to Karl Fischer is rapid, accurate and reliable. It has become the method of choice in quality and in-process control, production, and research and development.

Advantages

- High accuracy and excellent precision
- Rapid and reproducible titration results
- Large water capacity
- No crystallization
- Innovative formulations for special applications
- Comprehensive product line
- Wide range of water standards

Brilliant results
with Aquastar® reagents
for brighter accuracy.

What's Inside ?

Precise water determination every time	4
Aquastar™ products: quality standard for water determination	6
The right method for any application	8
Volumetric titration	10
One component reagents	12
Two component reagents	13
Solvents for oils and fats	14
Reagents for aldehydes and ketones	15
Coulometric titration	16
CombiCoulomat frit CombiCoulomat fritless	18
Auxillaries for Karl Fischer Titration	19
Standards	20
Water standards in ampoules	22
Water standard oven 1%	22
Water standard oil 15 – 30 ppm	22
Lactose standard 5%	22
Sodium tartrate dihydrate 15.66%	22
Water standard 5 mg/mL	22

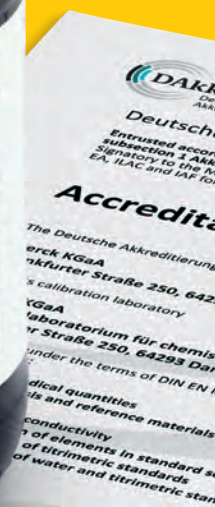
Precise water determination every time, with 40 years of experience

A proven method, modified according to the latest research

Since we apply the highest standards to production processes and stringent testing requirements, Aquastar™ reagents and standards are distinguished by excellent quality. The measured results are therefore always reliable and accurate. From the selection of raw materials and packaging, to quality

control of the finished reagents in our DIN EN ISO /IEC 17025 accredited laboratory, we apply the strictest standards. This ensures a sophisticated consistency, quality and thus comparable and transparent results. The certificates of analysis for reagents and standards contain all the necessary information for quality management documentation.

Aquastar™ products, the standard for reproducible results



Brand quality

We only use high-quality and approved raw materials. This ensures batch-to-batch consistency and comparable analytical results.

Experience

You benefit from our high scientific standards and our long international experience.

Safety

We place great importance on safety. For this reason, the Aquastar™ Karl Fischer reagents contain no toxic components or, if this is not possible, they are limited to a minimum.

Service

Our international sales network and local offices ensure safe delivery and service – worldwide.

Support

Whenever you need help, we can supply it via our application and development laboratories. We will advise you on applications, offer technical support, and assist you in validation projects.

Transparency

Our quality control is carried out in our DIN EN ISO/IEC 17025 accredited calibration and testing laboratory for Karl Fischer Titration, according to the most stringent standards. This renders the results achieved completely transparent.



The quality standard for water determination

The significance of water determination with Karl Fischer titration is emphasized by the fact that it has been included in the most important Pharmacopoeias, American Society for Testing and Materials (ASTM), industrial norms and other guidelines. Together with our Aquastar™ standards our Karl Fischer product line is always reliable, rapid, accurate and globally available to facilitate international product transfer.

Specification / Traceability

- American Society for Testing and Materials (ASTM)
- International Organization for Standardization (ISO)
- National Institute of Standards and Technology, USA (NIST)
- EP European Pharmacopeia
- USP United States Pharmacopeia



Aquastar™ Reagents

Karl Fischer reagents and standards gives you sophisticated, precise and consistent water determination. Excellent quality distinguishes our Aquastar™ range.

Karl Fischer titration

Since their development in the 1930s, Karl Fischer titrations have been used worldwide. With this method the water contents of a large variety of materials can be determined over a wide concentration range from 1 ppm to 100%. In contrast to other methods, Karl Fischer titration is based on a chemical reaction with water, so that water is exclusively determined. Karl Fischer titration has a wide spectrum of applications and is used in a broad range of fields, for example water determination in foodstuffs, chemicals, pharmaceuticals, cosmetics and mineral oils.

Two methods for water determination

Two methods are available for the determination of water using the Karl Fischer method: a volumetric and a coulometric method. The method selected depends primarily on the amount of water to be determined.

- **Volumetry:** In the case of higher levels of water (0.1 – 100%), the volumetric method should be used. For volumetric titration, we supply both one component and two component reagents.
- **Coulometry:** For very low levels of water (<1%) or for water determination of very expensive substances with small sample amount, the coulometric method should be used. In this case, a difference is made between cells with and without a diaphragm. For both cell types, the appropriate Aquastar™ reagents are available.

Aquastar™ Standards

The Aquastar™ range is rounded off with water standards for monitoring and qualification of Karl Fischer instruments, for checking measuring results and titer determination.

**Aquastar™ Reagents
and standards
for your individual needs**

The right method for any application

To meet the requirements of modern laboratories we supply a broad range of different Karl Fischer reagents in our Aquastar™ product line. It covers the whole range of volumetric and coulometric Karl Fischer reagents together with water standards.

Choose the Karl Fischer reagents most suitable for your purposes, depending on your requirements and the available Karl Fischer instrument. The choice of

the »right« reagent is a decisive factor in obtaining correct and reproducible results. It depends on the type of sample and the matrix in which the water is encapsulated. The key is to have the sample either completely dissolved or dispersed in the Karl Fischer solvent or to make sure that all the water is extracted. The overview of the table offers advice regarding the correct choice and combination of reagents.

Titrants	Solvents						
	Samples dissolving in Ethanol	Samples dissolving in Methanol	Samples dissolving in Methanol	Samples containing aldehydes + ketones	Mineral oils	Oils + fats in food	Samples of long-chain hydrocarbons
	CombiSolvent [188008]	CombiMethanol [188009]	Solvent [188015]	CombiSolvent Keto [188007]	CombiSolvent oils [188020]	CombiSolvent fats [188021]	Solvent oils & fats [188016]
CombiTitrant 5 [188005]	•	•	•		•	•	•
Titrant 5 [188010]			•				•
CombiTitrant 5 Keto [188006]				•			
CombiTitrant 2 [188002]	•	•	•		•	•	•
Titrant 2 [188011]			•				•
CombiTitrant 1 [188001]	•	•	•		•	•	•
CombiCoulomat frit [109255]	Can be used for anode and cathode compartment in cells with diaphragm						
CombiCoulomat fritless [109257]	Can be used for cells with and without diaphragm						
Standards	Water standard: 0.01% / 0.1% / 1% / oven 1% / oil 15 – 30 ppm / 5 mg/mL, Lactose standard 5% Sodium tartrate dihydrate 15.66%						

Benefit from our experience



Volumetric titration

This method is preferably used for water contents in samples between 0.1 and 100%. The unknown water content of the sample is determined by measuring the required volume of Karl Fischer Titrant to reach the titration endpoint. The sample is completely dissolved or dispersed in a suitable solvent with strict exclusion of atmospheric moisture and titrated with the Karl Fischer Titrant. The titration end point is indicated by an excess of iodine and is determined potentiometrically.

Advantages

- High titration rate
- For water content from 0.1% – 100%
- Accurate and reliable results



water
determination
according to
Karl Fischer



Volumetric titration

1. One component reagents
2. Two component reagents
3. Solvents for oils and fats
4. Reagents for aldehydes and ketones

1. One component reagents

CombiTitrant | CombiSolvent | CombiMethanol

One component reagents advantages

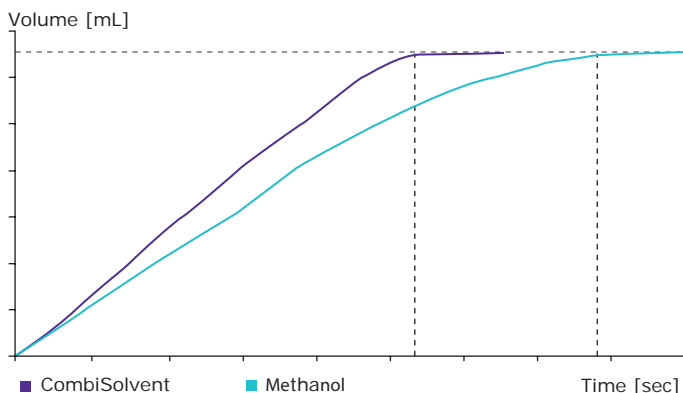
- High titration rate for fast analyses
- Distinct endpoint to ensure confidence in results
- Ensured constant and consistent high quality for comparable results
- No toxic alcohols in CombiTitrant and CombiSolvent for more safety
- More flexibility – the solvent can be suited to the sample matrix
- Unlimited water capacity – more titrations are possible than with two component reagents

CombiTitrant

In one component volumetric Karl Fischer analysis, the titrant contains all ingredients required by the Karl Fischer reaction: iodine, base, sulfur dioxide, and an alcohol. Aquastar™ CombiTitrants and CombiSolvent are free of toxic alcohols and available in packaging compatible with all major brands of titrators. The range includes titrants with nominal titer values of 1, 2, 5 mg H₂O/mL to cover a wide range of practical applications.

CombiSolvent

The Aquastar™ CombiSolvent is an innovative methanol-free solvent for one component volumetric titration. This ethanol-based solvent does not contain toxic alcohols and when used in conjunction with the Aquastar™ CombiTitrants allows a non toxic titration. This means more safety for the laboratory as well as easy storage of the reagents.



Titration curve: The Aquastar™ CombiSolvent is a methanol-free solvent for the one component system. This solvent brings about a higher titration rate and a more distinct endpoint.



CombiMethanol

Methanol – with a low water content – is the solvent typically used in one component titration for standard applications. The quality of methanol used for the Karl Fischer Titration is critical to minimize both pretitration, and interfering side reactions. Aquastar™ CombiMethanol is optimized specifically for use in Karl Fischer titrations by strictly limited key impurities and water content.

Ordering information

Product	Content	Packaging	Ord. No.
CombiTitrant 5, approx. 5 mg H ₂ O/mL	500 mL	Glass bottle	1.88005.0500
	1 L	Glass bottle	1.88005.1000
	2.5 L	Glass bottle	1.88005.2500
CombiTitrant 2, approx. 2 mg H ₂ O/mL	1 L	Glass bottle	1.88002.1000
CombiTitrant 1, approx. 1 mg H ₂ O/mL	1 L	Glass bottle	1.88001.1000
CombiSolvent, methanol-free solvent	1 L	Glass bottle	1.88008.1000
	2.5 L	Glass bottle	1.88008.2500
CombiMethanol, dried methanol for Karl Fischer titration max. 0.01% water	1 L	Glass bottle	1.88009.1000
	2.5 L	Glass bottle	1.88009.2500

For water determinations in oils and fats we recommend CombiSolvent oils (188020) and CombiSolvent fats (188021).

2. Two component reagents

Titrant | Solvent

Unlike one component Karl Fischer reagents, in two component reagent systems, the titrant contains only iodine and methanol, while the solvent contains the other Karl Fischer reaction components – sulfur dioxide and a suitable base dissolved in methanol. It is used as the working medium in the Karl Fischer cell. Aquastar™ Titrants are available with concentrations of 2 and 5 mg H₂O/mL together with Aquastar™ Solvent.



Two component reagents advantages

- Slightly faster titration in comparison to one component titration
- High accuracy for reliable results
- Better buffer capacity
- Higher titer stability than one component reagents
- Ensured consistent high quality for comparable results

Two component reagents are distinguished by greater long term stability and a slightly faster titration time in comparison to one component reagents. This is due to the fact that the Karl Fischer reaction components are divided in between the two reagents making each reagent less susceptible to slow side reactions over time.

Faster titration rates result from the initial availability of sulfur dioxide and base in the solvent to which the sample to be analyzed is added.

Ordering information

Product	Content	Packaging	Ord. No.
Titrant 5, approx. 5 mg H ₂ O/mL	500 mL	Glass bottle	1.88010.0500
	1 L	Glass bottle	1.88010.1000
	2.5 L	Glass bottle	1.88010.2500
Titrant 2, approx. 2 mg H ₂ O/mL	1 L	Glass bottle	1.88011.1000
Solvent, for the two component titration	1 L	Glass bottle	1.88015.1000
	2.5 L	Glass bottle	1.88015.2500

For water determinations in oils and fats we recommend Solvent oils & fats (188016).

3. Solvents for oils and fats

CombiSolvent oils | CombiSolvent fats | Solvent oils & fats

When determining the exact water content in oils and fats it is important that the samples are completely dissolved or dispersed. If the sample is not fully dissolved or dispersed, the water is not completely extracted during determination. Depending on the type of oil or fat to be determined, different solvents are suited for this purpose.



CombiSolvent fats is based on decanol, butyl acetate, and methanol, and is recommended for volumetric Karl Fischer analysis of water in fatty food, such as butter, margarine, vegetable fats, chocolate, mayonnaise, etc.

CombiSolvent oils | CombiSolvent fats

Mineral oils are primarily a mixture of long-chain hydrocarbons with aromatic compounds. Fats consist primarily of glycerol esters of higher fatty acids. The two classes of substance have different dissolution properties requiring different dissolution aids. We have therefore developed appropriate solvents for one component titration in both applications: CombiSolvent oils for mineral oils and CombiSolvent fats for fats in foodstuffs. Both are used in combination with CombiTitrants.

Solvent oils & fats

Solvent oils & fats is a universal solvent for long-chain, nonpolar substances as well as light fats and oils. It can be used with two component titrants, as well as with CombiTitrants.

Solvents for oils and fats advantages

- Good solubility of oils and fats in the corresponding solvent
- No addition of dissolution aids necessary – immediately ready for use
- Constant and consistently high quality
- Without chloroform

Ordering information

Product	Content	Packaging	Ord. No.
Solvent oils & fats, for long-chain substances	1 L	Glass bottle	1.88016.1000
CombiSolvent oils, for mineral oils	1 L	Glass bottle	1.88020.1000
CombiSolvent fats, for fats in foodstuffs	1 L	Glass bottle	1.88021.1000

Solvent oils & fats can be used in combination with two component Titrant 5 (188010), but also in combination with CombiTitrant 5 (188005). For titration of oils and fats with a very low water content we recommend our titrants with the factor 2 and 1, respectively: CombiTitrant 1 (188001), CombiTitrant 2 (188002) and Titrant 2 (188011).

4. Reagents for aldehydes and ketones

CombiTitrant 5 Keto | CombiSolvent Keto

CombiTitrant 5 Keto and CombiSolvent Keto are two products of the Aquastar™ line. When performing water determinations with the Karl Fischer method in samples containing aldehydes and ketones it must be taken into account that these determinations are influenced by side reactions if methanol is used as a solvent.

CombiTitrant 5 Keto

CombiTitrant 5 Keto is a one component titrant – all reactive components required for the Karl Fischer reaction: SO₂, I₂ and the base dissolved in a long-chain alcohol, are combined in a single reagent.



CombiTitrant 5 Keto together with CombiSolvent Keto are designed for samples containing aldehydes and ketones.

CombiSolvent Keto

CombiSolvent Keto is a solvent based upon a mixture of alcohol suppressing to a large extent the impact of side reactions with aldehydes and ketones. At the same time, its composition ensures optimum conditions for titration according to the Karl Fischer method with respect to stoichiometry, reaction rate and indication of the endpoint.

Reagents for aldehydes and ketones advantages

- Fast titration rate
- No toxic alcohol contained
- Improved accuracy and reproducibility of the results
- Suppression of side reactions
- Consistently high quality

Ordering information

Product	Content	Packaging	Ord. No.
CombiTitrant 5 Keto, approx. 5 mg H ₂ O/mL for aldehydes and ketones	1 L	Glass bottle	1.88006.1000
CombiSolvent Keto, methanol-free solvent for aldehydes and ketones	1 L	Glass bottle	1.88007.1000

Coulometric titration

With diaphragm | Without diaphragm

Coulometric Karl Fischer titration is preferred for water contents below 1%. In coulometric Karl Fischer titration the iodine required for reaction is produced in the titration vessel itself by anodic oxidation at the generator electrode. The water content is accurately determined by calculation from the amount of current used over a specific time period. The measuring cell for coulometric determination consists of anode and cathode compartments which can be separated by a diaphragm.

Advantages

- For low water contents below 1%
- Highly accurate and reliable reproducible results
- Same reagents for anode and cathode cell

water
determination
according to
Karl Fischer





Coulometric reagents

CombiCoulomat frit | CombiCoulomat fritless

Our combined coulometric reagents are excellently suited for water determinations according to the coulometric Karl Fischer method.

CombiCoulomat frit | CombiCoulomat fritless

The CombiCoulomat fritless can be used universally for cells with or without diaphragm. Both reagents, CombiCoulomat frit and CombiCoulomat fritless are, of course, free from chlorinated hydrocarbons. The coulometric reagents Aquastar™ CombiCoulomat frit and CombiCoulomat fritless are comparable to the volumetric one component reagents. All reaction components are contained in one solution. This makes handling easier for the user.

Compared with volumetric reagents, coulometric reagents do not contain iodine, but iodide. The iodine required for the Karl Fischer reaction to take place develops through electrochemical oxidation. The coulometric method is primarily used for the determination of low water contents (<1%).

Coulometric reagents advantages

- No confusion between anode and cathode cell solutions, since only one solution is required
- Very accurate and reproducible results
- Cell very rapidly ready for use after the first filling
- For samples with low water contents – below 1%
- Consistent quality for comparable results



Ordering information

Product	Content	Packaging	Ord. No.
CombiCoulomat frit, for cells with diaphragm	500 mL	Glass bottle	1.09255.0500
CombiCoulomat fritless, for cells with and without diaphragm	500 mL	Glass bottle	1.09257.0500
CombiCoulomat fritless, for cells with and without diaphragm	2.5 L	Glass bottle	1.09257.2500

We recommend CombiCoulomat frit for coulometric devices with a diaphragm cell. For users working without a diaphragm or who frequently switch from cells with diaphragm to cells without diaphragm we recommend CombiCoulomat fritless. It can be used for both cell types.

Auxillaries for Karl Fischer Titration

Buffer solutions

Of decisive importance for water determination according to Karl Fischer is firstly, the complete dissolution of the sample, and secondly, maintaining the optimum pH of between pH 5 – 7. When strong acids or alkalis are used as samples, the pH value can be out of equilibrium. By the addition of special buffer solutions, the Karl Fischer reaction can proceed in the correct pH range. The two ready-to-use buffer solutions are used in the Karl Fischer titration method, particularly in connection with samples that may disturb the pH optimum for the Karl Fischer reaction. Normally the buffering of the reaction medium by the Karl Fischer reagent is sufficient. With some samples, however, such as strong bases or strong acids, the pH is shifted into the acid or alkaline range. In such cases additional buffer solution must be added to maintain the pH in the optimum range.

Ordering information

Product	Content	Packaging	Ord. No.
Buffer solution for strong acids, additive to the Karl Fischer solvent for titration of strong acids	500 mL	Glass bottle	1.88035.0500
Buffer solution for strong bases, additive to the Karl Fischer solvent for titration of strong bases	500 mL	Glass bottle	1.88036.0500
Karl Fischer Test Kit for Karl Fischer Titration without instrument			
Test Kit for water determination, according to Karl Fischer, Set*	1 set*	Fibre carton	1.88025.0001
Test Kit Titrant for water determination, according to Karl Fischer, Refill pack	100 mL	Glass bottle	1.88026.0100
Test Kit Solvent for water determination, according to Karl Fischer, Refill pack	500 mL	Glass bottle	1.88027.0500

*1 set consisting of: 100 mL Titrant (Glass bottle), 500 mL Solvent (Glass bottle), 1 Syringe, 1 Measuring flask.

Standards

Increasing regulations demand that analytical results become more transparent and comparable. This is particularly true for water determinations according to Karl Fischer. In order to ensure this, reliable reference materials are necessary. Our Aquastar™ product line offers a series of excellent standards. In addition to their use in monitoring Karl Fischer equipment and performing titer determination of volumetric Karl Fischer reagents, they can also be used for checking measuring results in order to evaluate their accuracy. To ensure the highest quality, our Aquastar™ standards are manufactured under the strictest control and accurately measured using validated procedures in our DIN EN ISO/IEC 17025 accredited calibration and testing laboratory for Karl Fischer Titration.

Advantages

- Batch-specific Certificate of Analysis in the package for QM documentation
- Reliable and correct results



A series of
**excellent
standards**



Water standards in ampoules

Our Aquastar™ product range offers water standards in ampoules with different water contents. The standards consist of solvent mixtures with a defined water content. They are tested against standard reference material from NIST – National Institute of Standards and Technology, Gaithersburg, USA. Each package contains a batch-specific certificate with the exact measured water content of the batch, uncertainty data, measuring method, the NIST SRM, and minimum shelf-life.

Water standard 1% is recommended for the use of titer determination using the volumetric method with one component and two component reagents. The two water standards 0.01% and 0.1% are used for the coulometric method.

Water standard oven 1%

This standard is an entirely solid standard for the Karl Fischer oven method. In contrast to other solid standards like lactose, citrate or tartrate, this standard has a considerably lower water content of only 1% and can be used over a wide temperature range from 150 – 400°C. The composition of the solid standard is based on an inorganic substance, which is stable even at high temperatures. In contrast, with organic substances decomposition reactions with formation of water may already set in at temperatures above 150°C, leading to erroneous results. The low water content of 1% is particularly suited when using the Karl Fischer oven with a coulometer. Each package contains a batch-specific certificate with the exact measured water content of the batch, uncertainty data, measuring methods – Karl Fischer method and thermogravimetry – and minimum shelf-life.

Water standard oil 15 – 30 ppm

This standard is especially designed for users who need to measure the water content in oil samples. The water standard exactly corresponds to the low water content and the matrix of oil samples and the exact value for each batch is stated in the certificate. Practical ampoules ensure ease of handling.

Lactose standard 5%

The Aquastar™ lactose standard is a further solid standard with a water content of about 5%. The batch-specific Certificate of Analysis states the exact value measured in the respective batch. The lactose standard can be applied universally. Due to its ease of solubility in methanol and its water content, this standard is suitable as a solid standard for coulometry, and also for volumetric Karl Fischer titrations. It is especially suitable if the working medium used consists of solvent mixtures in which sodium tartrate dihydrate is only slightly soluble. But it can also be used as an oven standard in a temperature range preferably from 140 – 160°C.

Sodium tartrate dihydrate 15.66%

Sodium tartrate dihydrate is the volumetric standard for Karl Fischer titration. Under normal conditions it is stable and non-hygroscopic. Sodium tartrate dihydrate has a stoichiometric water content of 15.66% and is primarily used for titer determination in volumetry.

Water standard 5 mg/mL

This liquid water standard is based on a long-chain alcohol. Under normal conditions the adjusted water content is maintained after opening the bottle. This water standard 5 mg/mL can be used for daily titer control, but is not recommended for exact titer determinations.

Ordering information

Product	Content	Packaging	Ord. No.
Water standard 0.01%, 1 g contains 0.10 mg H ₂ O	10 x 8 mL	Glass ampoule	1.88050.0010
Water standard 0.1%, 1 g contains 1.0 mg H ₂ O	10 x 8 mL	Glass ampoule	1.88051.0010
Water standard 1%, 1 g contains 10 mg H ₂ O	10 x 8 mL	Glass ampoule	1.88052.0010
Water standard oven 1%, solid standard for KF oven method	5 g	Glass bottle	1.88054.0005
Water standard oil, standard for oil samples for coulometric Karl Fischer titration (15 – 30 ppm)	10 x 8 mL	Glass ampoule	1.88055.0010
Lactose standard 5%, for volumetry and KF oven method	10 g	PE bottle	1.12939.0010
Sodium tartrate dihydrate, volumetric standard for water determination according to Karl Fischer, contains 15.66% H ₂ O	100 g	PE bottle	1.06664.0100
Water standard 5 mg/mL, 1 mL contains 5 mg water	250 mL	Glass bottle	1.09259.0250



Deutsche Akkreditierungsstelle GmbH

Entrusted according to Section 8 subsection 1 AkkStelleGBV
Signatory to the Multilateral Agreements of
EA, ILAC and IAF for Mutual Recognition

Accreditation



The Deutsche Akkreditierungsstelle GmbH attests that the

Merck KGaA
Frankfurter Straße 250, 64293 Darmstadt

with its calibration laboratory

Merck KGaA
Kalibrierlaboratorium für chemische Messgrößen
Frankfurter Straße 250, 64293 Darmstadt

is competent under the terms of DIN EN ISO/IEC 17025:2005 to carry out calibrations in the following fields:

- Chemical and medical quantities**
- Chemical analysis and reference materials**
 - pH value
 - Electrolytic conductivity
 - Mass fraction of elements in standard solutions
 - Mass fraction of titrimetric standards
 - Mass fraction of water and titrimetric standards

Braunschweig,
24.02.2016

This document is a translation. The definitive version is the original German accreditation certificate.
See notes overleaf.

Dr. Michael Wolf
Head of Division

Translation issued:
2016-02-24

Head of Division

