

Accurate Water Determination

Aquastar[®] reagents and standards for precise Karl Fischer titration results



1 88005,1000 Aquastar

CombiTitrant 5 ore-component reagent for volumetric kan Fischer trattoion toxians lodine, Imidazole, 2-kethylimidazole, 2-Methylimidazoli toxinen Yodo, Imidazole, 2-Methylimidazolo toxinen Yodo, Imidazole, Methyl-2-imidazole toxinen Yodo, Imidazolo, 2-Methilimidazolo Ibarai Jood, Imidazolo, 2-Methilimidazolo Ibarai Jood, Imidazol, 2-Methylimidazolo Imili e.a. S ma IH-O 1 ml ± ca. 5 mg H₂O

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Aquastar[®] Karl Fischer Reagents

Karl Fischer titration using our Aquastar[®] reagents and standards allows for easy and highly accurate water content determination in gases, liquids and solids. 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 is therefore the method of choice for quality and in-process control, production, and research and development.

Advantages

- High accuracy and excellent precision
- Rapid and reproducible results
- Large water capacity
- No crystallization
- Innovative formulations for special applications
- Reagents for all types of applications
- Wide range of water standards
- Fast conditioning and stable drift

For more information fishersci.com/supelco

Accurate results with Aquastar® Reagents

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Precise water determination every time with 40 years of experience

A proven method, modified according to the latest research

We apply the highest standards to our production processes along with stringent testing requirements, so that our Aquastar® reagents and standards are distinguished by their excellent quality. The measuring results are therefore always reliable and accurate. From the selection of materials, to packaging and quality control of the finished reagents and

standards we apply the strictest standards. This ensures consistency, quality and thus comparable and transparent results. The certificates of analysis for our reagents and standards contain all the information important for quality management documentation.

Aquastar[®] products, the standard for reproducible results



Aquastar® Anolyte for the coulometric Karl Fischer Titration without a diaphragm

Anolyte

Anolyte

1.88079.0500

Aquastar®

for the coulometric Karl Fischer Titration without a diaphragm Anolyte

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Brand quality	We use only high-quality and approved raw materials. This ensures batch-to-batch consistency and comparable analytical results.
Competence	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 and standards 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. Here we can advise you on applications, offer technical support, and assist you in validation projects. Contact your local account manager.
Transparency	Our quality control is carried out according to the most stringent standards. This renders the results achieved completely transparent. The Aquastar® water standards are Certified Reference Materials acc. to ISO 17034 and measured in our DIN EN ISO 17025 accredited calibration lab.

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 Pharmacopoeias, American Standard Methods (ASTM), ISO regulations (International Organization for Standardization), and other industrial norms and guidelines. Together with our Aquastar[®] water standards our Karl Fischer product line is always reliable, fast, accurate and globally available to facilitate international product transfer.

Regulations / Traceability

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



Aquastar® reagents

Karl Fischer reagents for sophisticated, precise and consistent water determination. Excellent quality distinguishes our Aquastar[®] range.

Karl Fischer titration

Since its development in the 1930s, the Karl Fischer titration method has found use worldwide. With this method the water content of a large variety of materials can be determined over a wide concentration range from 10 ppm up to 100%. In contrast to other methods, the 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 and the properties of the sample.

- Volumetry: In the case of higher levels of water (0.01% to 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, Aquastar[®] reagents are available for cells with and without a diaphragm.

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 different requirements of all laboratories a broad range of different Karl Fischer reagents and standards are available in the Aquastar[®] portfolio. It covers water standards, volumetric and coulometric reagents, specific titrants and solvents for samples which cannot determined with the standard reagents.

Choose the best Karl Fischer reagents to fit your needs and 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. To ensure accurate results, it is important that the sample is completely dissolved or dispersed in the Karl Fischer solvent, or that all the water is extracted. Consult the table below for advice on how to choose the most suitable reagents combination for your sample.

	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
Titrants	CombiSolvent [M188008]	CombiMethanol [M188009]	Solvent [M188015]	CombiSolvent Keto [M188007]	CombiSolvent oils [M188020]	CombiSolvent fats [M188021]	Solvent oils & fats [M188016]
CombiTitrant 5 [M188005]	•	•	•		•	•	•
Titrant 5 [M188010]			•				•
CombiTitrant 5 Keto [M188006]				•			
CombiTitrant 2 [M188002]	•	•	•		•	•	•
Titrant 2 [M188011]			•				•
CombiTitrant 1 [M188001]	•	•	•		•	•	•
CombiCoulomat frit [M109255]	Can be used for	anode and cathod	e compartmen	t in cells with diaphragm			
CombiCoulomat fritless [M109257]	Can be used for	cells with and with	nout diaphragn	1			
Standards		: 0.01% / 0.1% / 1 e dihydrate 15.66%	,	/ oil 15 – 30 ppm / 5 mg	/mL, Lactose stan	dard 5%	

Benefit from our experience











Volumetric titration

This method is preferably used when the water content of your sample is between 0.01 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 the water is completely extracted with strict exclusion of atmospheric humidity 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 0.01% 100%
- Accurate and reliable results
- Stable endpoint and fast drift stability
- No crystallization

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

Advantages of one component reagents

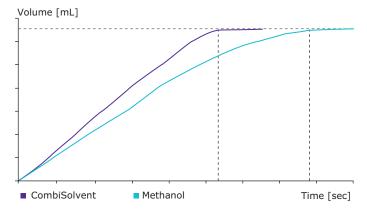
- Fast titration
- Distinct endpoint to ensure confidence in results
- Ensured constant and consistent high quality for comparable results
- No toxic alcohols in CombiTitrant and CombiSolvent for better safety for lab personnel
- More flexibility the solvent can be suited to the sample matrix
- Without chloroform

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 from toxic alcohols and available in a packaging compatible with all major brands of titrators. The range includes titrants with nominal titer values of 1, 2, or 5 mg H₂O/mL to cover a wide range of applications.

CombiSolvent

The Aquastar[®] CombiSolvent is an innovative methanol-free solvent for one component volumetric titration. It is ethanol-base and used in conjunction with the Aquastar[®] ComibTitrants and allows for titration without toxic alcohols.



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 purity of methanol used for the Karl Fischer titration is critical to avoid interfering side reactions. Our Aquastar[®] CombiMethanol is optimized specifically for use in Karl Fischer titrations thanks to strictly limited key impurities and water content.

Ordering information

Product	Content	Packaging	Ord. No.
CombiTitrant 5,	500 mL	Glass bottle	M1.88005.0500
approx. 5 mg H_2O/mL	1 L	Glass bottle	M1.88005.1000
	2.5 L	Glass bottle	M1.88005.2500
CombiTitrant 2, approx. 2 mg H ₂ O/mL	1 L	Glass bottle	M1.88002.1045
CombiTitrant 1, approx. 1 mg H ₂ O/mL	1 L	Glass bottle	M1.88001.1045
CombiSolvent,	1 L	Glass bottle	M1.88008.1000
methanol-free solvent	2.5 L	Glass bottle	M1.88008.2500
CombiMethanol, dried methanol for Karl Fischer titration max. 0.01% water	1 L	Glass bottle	M1.88009.1000
	2.5 L	Glass bottle	M1.88009.2500

For water determinations in oils and fats we recommend CombiSolvent oils, (M188020) and CombiSolvent fats (M188021).



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 and used together with Aquastar[®] solvent.

Advantages of two component reagents

- Slightly faster titration in comparison to one component titration
- High accuracy for reliable results
- Better buffer capacity
- Higher titer stability than one component in titrants
- without chloroform 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 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	M1.88010.0545
	1 L	Glass bottle	M1.88010.1045
	2.5 L	Glass bottle	M1.88010.2545
Titrant 2, approx. 2 mg H ₂ O/mL	1 L	Glass bottle	M1.88011.1045
Solvent, solvent for the two component titration	1 L	Glass bottle	M1.88015.1000
	2.5 L	Glass bottle	M1.88015.2500



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 dissolved or dispersed completely, 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 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.

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.

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.

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	M1.88016.1000
CombiSolvent oils, for mineral oils	1 L	Glass bottle	M1.88020.1002
CombiSolvent fats, for fats in foodstuffs	1 L	Glass bottle	M1.88021.1000

Solvent oils & fats can be used in combination with two component Titrant (M188010), but also in combination with CombiTitrant (M188005). 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 (M188001), CombiTitrant 2 (M188002) and Titrant 2 (M188011).



4. Reagents for aldehydes and ketones

CombiTitrant 5 Keto | CombiSolvent Keto

CombiTitrant 5 Keto and CombiSolvent Keto for the determination of water content in samples containing aldehydes or ketones. 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. Methanol reacts with aldehydes or ketones to acetals or ketals and water. Additonally we have a second side reaction called the bisulfite addition. This reaction consumes water and therefore we need a very fast titration.



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

CombiTitrant 5 Keto

CombiTitrant 5 Keto is a one component titrant — all reactive components required for the Karl Fischer reaction: SO_2 , I_2 and the base dissolved in a long-chain alcohol, are contained in a single reagent system.

CombiSolvent Keto

CombiSolvent Keto is a methanol free solvent based upon a mixture of alcohol designed to suppress 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.

Advantages

- · Fast titration to surpress bisulfite addition
- Methanol free to avoid acetal & ketal formation
- Improved accurate and reproducable results
- Without chloroform

Ordering information

Product	Content	Packaging	Ord. No.
CombiTitrant 5 Keto , approx. 5 mg H_2O/mL for aldehydes and ketones	1 L	Glass bottle	M1.88006.1000
CombiSolvent Keto, methanol-free solvent for aldehydes and ketones	1 L	Glass bottle	M1.88007.1002

Coulometric titration

With diaphragm | Without diaphragm

The coulometric Karl Fischer titration method 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 precise determination of low water contents below 1 %
- Highly accurate and reliable reproducible results
- Fast conditioning an drift stability
- Same reagents used for anode and cathode cell

water determination according to Karl Fischer





Coulometric reagents

CombiCoulomat frit | CombiCoulomat fritless Anolyte | Anolyte K | Catholyte K

Our combined coulometric reagents have long since proved to be 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 are free from chlorinated hydrocarbons. Our Aquastar[®] coulometric reagents CombiCoulomat frit and CombiCoulomat fritless are comparable to the volumetric one component reagents. All reaction components are contained in one solution, making handling easy.

Anolyte K | Catholyte K for aldehydes and ketones

Anolyte K and Catholyte K are special reagents to determine the water content in aldehyde and ketone samples in coulometric titration cells with a diaphragm. Both reagents need to be use in tandem, filled in the anode and cathode compartment. The benefit of these reagents, compared to standard reagents, is the use of a methanol free solvent. The absence of methanol averts side reactions with aldehydes and ketones and stops the formation of ketals or acetals and water. A fast titration avoids the second side reaction, the bisulfite addition, which is consuming water. It is more dominant for aldehyde samples who are more reactive as ketones. Further-more, the reagents enable sharp endpoint detections for precise results.

Compared with volumetric reagents, coulometric reagents do not contain iodine, but iodide and 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%).

Anolyte for coulometry without diaphragm

This Anolyte can be us for all kind of liquid samples for coulometric titration in cells without a diaphragm. Oils, fats, strong acids or bases can be determined with the addition of solubilizer or additional buffer and solid samples, crude oils, engine oils with the Karl Fischer oven method or an external extraction of the water.

Coulometric reagents advantages

- One reagent for anode and cathode cell
- Very accurate and reproducible results
- Fast conditioning and stable drift
- For samples with low water contents below 1%
- Special reagents for aldehydes & ketones 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.

Auxiliaries for Karl Fischer Titration

Both the complete dissolution of the sample plus the maintenance of a pH between 5 and 7 are extremely important in order to achieve accurate water determination results. When strong acids or alkalis are used as samples, the pH value can be pushed out of equilibrium. The addition of special buffer solutions ensures that the Karl Fischer reaction can proceed in the correct pH range. When working with samples that may disturb the pH of the Karl Fischer reaction such as strong bases or strong acids, we offer two ready-to-use buffer solutions. These can be added to maintain the optimum pH range for accurate Karl Fischer results.

Ordering information

Product	Content	Packaging	Cat. 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 solventfor titration of strong bases	500 mL	Glass bottle	1.88036.0500

Water Standards

Within the European Union and worldwide, there is an increasing demand for more transparent and comparable analytical results. This is particularly true for water determinations according to Karl Fischer, it is therefore necessary to use reliable reference materials to ensure accurate results. 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 verifying measuring results in order to evaluate their accuracy and the performance of the titration process. To ensure the highest quality, our Aquastar® water standards are manufactured under the strictest control and are accurately measured using validated procedures in our DIN EN ISO/IEC 17025 accredited calibration laboratory for Karl Fischer titration. All Aquastar® Karl Fischer water standards are Certified Reference Materials according to ISO 17034.

Advantages

- Batch-specific Certificate of Analysis for QM documentation
- Reliable and correct results
- CRM according to ISO 17034
- Analyzed by a ISO/IEC 17025 accredited calibration lab
- Highly precise and accurate

A series of EXCELLENT Standards



Water standards in ampoules

The 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 batch, and minimum shelf-life.

Water standard 1% is recommended for the use of titer determination with the volumetric method with one component and two component reagents. The two water standards 0.01% and 0.1% are preferred for the coulometric method, but are also usable to verify low water content results in volumetry.

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 100-300 °C. The composition of the solid standard is based on inorganic substances, which are stable even at high temperatures. In contrast, with organic substances decomposition reactions with formation of water may already set in at temperatures above 200 °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, uncertainty data, measuring method 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. 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 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 can be used as a solid standard for both coulometric and 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 140–190 °C.

Sodium tartrate dihydrate 15.66%

Sodium tartrate dihydrate is a solid water standard for the volumetric Karl Fischer titration. It has a stoichiometric water content of 15.66% and is usable for titer determination or to verify results.

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 5 mg/mL water standard 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_20	10 x 8 mL	Glass ampoule	M1.88050.0010
Water standard 0.1%, 1 g contains 1.0 mg H_20	10 x 8 mL	Glass ampoule	M1.88051.0313
Water standard 1%, 1 g contains 10 mg H ₂ 0	10 x 8 mL	Glass ampoule	M1.88052.0313
Water standard oven 1%, solid standard for KF oven method	5 g	Glass bottle	M1.88054.0005
Water standard oil, standard for oil samples for coulometric Karl Fischer titration (15 – 30 ppm)	10 x 8 mL	Glass ampoule	M1.88055.0010
Lactose standard 5%, for volumetry and KF oven method	10 g	PE bottle	M1.12939.0010
Sodium tartrate dihydrate, volumetric standard for water determination according to Karl Fischer, contains 15.66% $\rm H_2O$	100 g	PE bottle	M1.06664.0100
Water standard 5 mg/mL, 1 mL contains 5 mg water	250 mL	Glass bottle	M1.09259.0250



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Merck KGaA Kalibrierlaboratorium für chemische Messgrößen Frankfurter Straße 250, 64293 Darmstadt die Kompetenz nach DIN EN ISO/IEC 17025:2005 besitzt, Kalibrierungen in folgenden Bereichen durchzuführen:

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Die Akkreditierungsurkunde silt nur in Verbindung mit dem Bescheid vom 24.02.2016 mit der Akkreditierungsnummer D.-K-15185-01 und ist gültig bis 18.11.2019. Sie besteht aus diesem Deckblatt, der Rückseite des Deckblatts und der folgenden Anlage mit insgesamt 4 Seiten. Registrierungsnummer der Urkunde: D-K-15185-01-00

Braunschweiß, 24.02.2016

weise auf der Rückse

Auftrage of Im Auftrag Dr. Michael Wolf Abteilungsleiter



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