Scharlau

The wise choice

Aquagent®

The new range of pyridine-free reagents for reliable Karl Fischer titration

More stable and robust factor
Faster initial stabilisation
Greater reaction speed
More stable end point

On many occasions it is essential to know the amount of water present in a sample. Water can influence the reactivity, stability, and shelf life, etc, of products. Karl Fischer titration has been the globally accepted method for the determination of water since the beginning of the 20th century. It is based on the Bunsen reaction, a fast, two-phase reaction, with a stoichiometric relationship between the I_2 consumed and the amount of water in the sample.

$$ROH + SO_2 + R'N \longrightarrow [R'NH]SO_3R$$

$$H_2O + I_2 + [R'NH]SO_3R + 2R'N \longrightarrow [RNH]SO_4R + 2[R'NH]I$$

pH 5-7

ROH = Alcohol, normally methanol
R'N = Nitrogen based

The first KF reagents developed contained pyridine in their formulation, supposedly essential for the reaction, but later experiments showed that pyridine only acts as a buffer substance, and could be replaced by other basic compounds capable of carrying out the same function. For this reason, the new Karl Fischer Aquagent® reagents contain imidazoles instead of pyridine, alternative bases with good buffering capacity that allow stable titration end points to be quickly obtained.
The newly developed manufacturing and control method allows us to launch a new Aquagent® with multiple advantages for the user.

New Aquagent® advantages

- **More stable and robust factor**
- **Faster initial stabilisation**
- **Greater reaction speed**
- **More stable endpoint**
- **Greater homogeneity between batches and within the same batch**
- **Suitable for a wide variety of matrices**
- **Greater variety of formats**
- **Less environmental impact**
- **Longer shelf life**

Aquagent®
Reliable results in Karl Fischer volumetric and coulometric titration

Aquagent®: new range of reagents

Aquagent® is the Scharlau name given to a wide range of Karl Fischer titration reagents. We offer a wide, improved range of reagents for sample titration with reliable results, which satisfy the needs of modern-day laboratories in the determination of water using Karl Fischer.

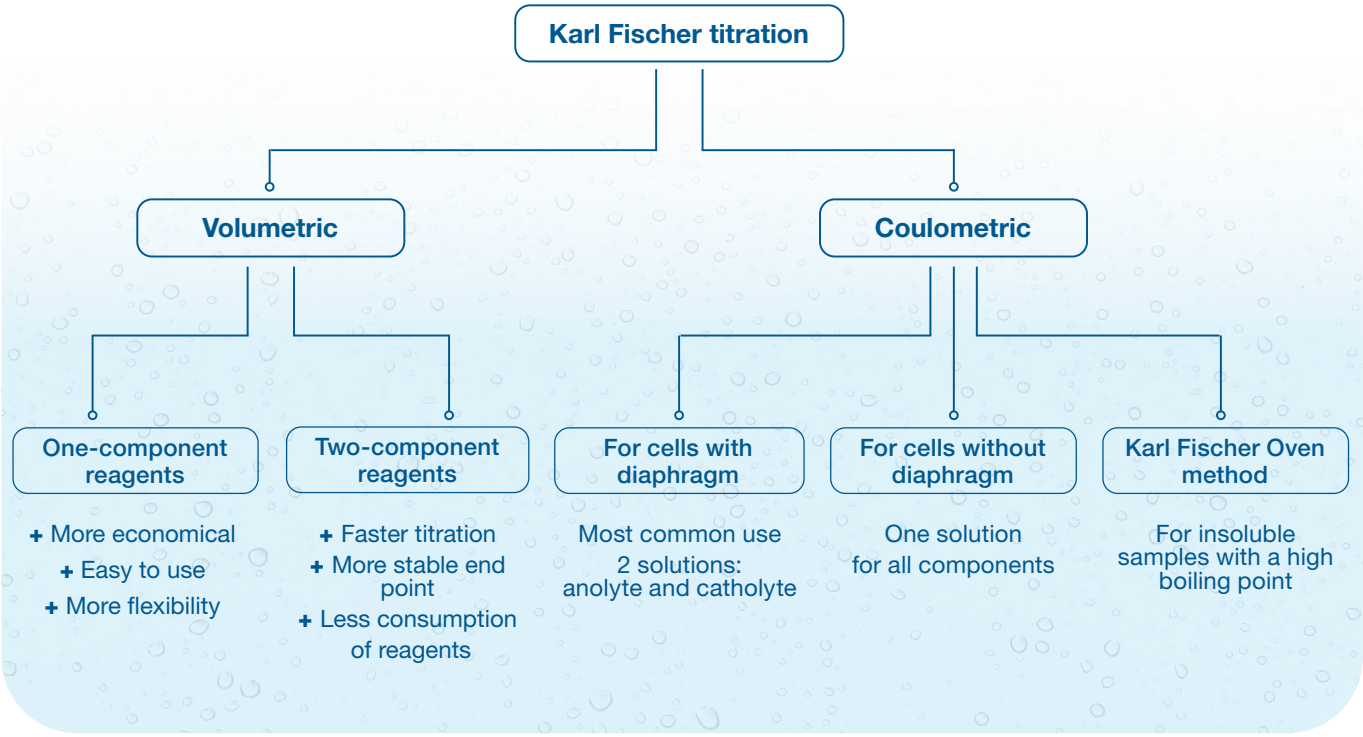
Aquagent® comprises:

- One or two-component reagents for volumetric titration
- Reagents for coulometric titration in cells with or without diaphragm
- Reagents for the Karl Fischer oven method
- Standards

Suitable for specific applications:

- Ketones and aldehydes
- Carbohydrates, inorganic salts and proteins
- Oils and fats
- Crude and related products
- Strong acids
- Bases
- Insoluble solid samples with a high boiling point

There are two methods based on Karl Fischer titration for the determination of water in a sample: volumetric and coulometric. The choice of one over the other depends upon the quantity of water expected in the sample. For samples with a water content higher than 0.1%, the volumetric method is generally used, whereas samples with a water content lower than 0.1% are normally analysed using the Karl Fischer coulometric titration. Choosing the correct method is essential to obtain accurate, reliable results.



Equipment manufacturers recommend us

TITRATION APPLICATION NOTE K-073

Volumetric Karl Fischer titration with Scharlau Aquagent® reagents

Test measurements using Aquagent® Complet 5 and Methanol Fast

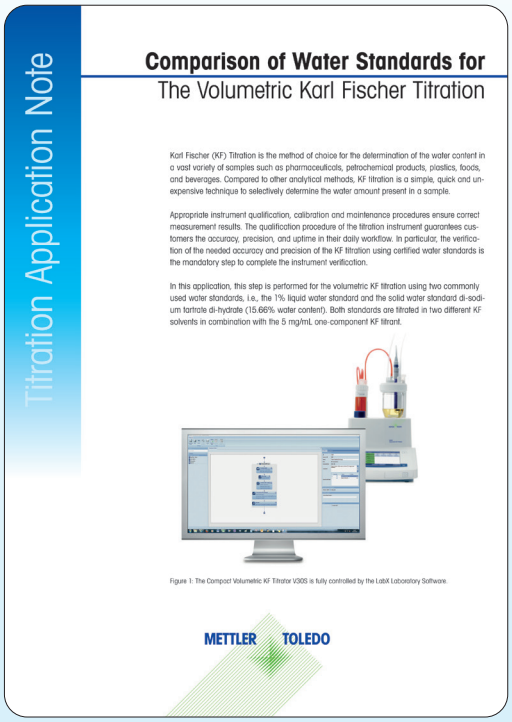
This Application Note summarizes a series of test measurements performed with an OMNIS KF Titrator and Karl Fischer reagents Aquagent® Complet 5 and Methanol Fast from Scharlau.

Three series of titer determinations using various water standards were carried out. The results obtained using different water standards were found to lie in a similar range. The reproducibility of the results was determined to be very good.

Using an OMNIS titration system from Metrohm and the Scharlau Karl Fischer reagents, titer determinations can be carried out quickly without any decline in the reproducibility of results.

www.metrohm.com

Metrohm



Titration Application Note

Comparison of Water Standards for

The Volumetric Karl Fischer Titration

Karl Fischer (KF) Titration is the method of choice for the determination of the water content in a vast variety of samples such as pharmaceuticals, petrochemical products, plastics, foods, and beverages. Compared to other analytical methods, KF titration is a simple, quick and unexpensive technique to selectively determine the water amount present in a sample.

Appropriate instrument qualification, calibration and maintenance procedures ensure correct measurement results. The qualification procedure of the titration instrument guarantees customers the accuracy, precision, and uptime in their daily workflow. In particular, the verification of the needed accuracy and precision of the KF titration using certified water standards is the mandatory step to complete the instrument verification.

In this application, this step is performed for the volumetric KF titration using two commonly used water standards, i.e., the 1% liquid water standard and the solid water standard di-sodium tartrate di-hydrate (15.66% water content). Both standards are titrated in two different KF solvents in combination with the 5 mg/mL one-component KF titrant.

Figure 1: The Compact Volumetric KF Titrator V30S is fully controlled by the LabX Laboratory Software.

METTLER TOLEDO

HANNA instruments

DETERMINACIÓN DE CONTENIDO DE AGUA EN ALQUITRÁN

Caso real con valorador HI933 y reactivos Aquagent Complet 5 y Aquagent Metanol fast.

Una empresa especializada en la destilación de alquitrán de hulla procedente de las baterías de cok de grupos siderúrgicos nos solicitó un valorador KF para la determinación la concentración de agua de sus distintos productos, principalmente alquitrán aunque también disolventes.

Para ello junto con los especialistas de SCHARLAB se seleccionaron los reactivos Aquagent Complet 5 (como valorante) y Aquagent Metanol fast junto con Xileno (como disolventes) para la determinación del contenido de agua utilizando un valorador Karl Fischer volumétrico HI933 de HANNA instruments.

La estandarización del valorante y verificación del equipo se realizó utilizando un patrón Aquagent solución patrón 10.0 obteniéndose valores repetitivos dentro de los valores de aceptación.

Tras esto se realizaron determinaciones de distintos productos como alquitrán utilizando la mezcla de disolvente Metanol:Xileno (1:1) obteniéndose gráficas claras y resultados repetitivos y comparables a los resultados del cliente mediante otras técnicas utilizadas hasta el momento.

Gracias a esta nueva técnica implantada la empresa determinará el contenido de agua en sus productos de forma rápida, repetitiva y precisa, agilizando la labor del laboratorio.

www.hanna.es

Titration Application Note

Coulometric Karl Fischer Titration

Comparison of Water Standards

Karl Fischer (KF) Titration is the method of choice for the determining of the water content in a vast variety of samples such as pharmaceuticals, petrochemical products, plastics, foods, and beverages. Compared to other analytical methods, KF titration is a simple, quick and unexpensive technique to selectively determine the water amount present in a sample.

Appropriate instrument qualification, calibration and maintenance procedures ensure correct measurement results. The qualification procedure of the titration instrument guarantees customers the accuracy, precision, and uptime in their daily workflow. In particular, the verification of the needed accuracy and precision of the KF titration using certified water standards is the a mandatory step to complete the instrument verification.

In this application, this step is performed for the coulometric KF titration using two commonly used water standards, i.e., the 1.0 mg/g and 0.1 mg/g liquid water standard. The water standards are titrated using different coulometric electrolytes for both generator cells, i.e., with and without diaphragm.

Figure 1: METTLER TOLEDO C30SD Compact KF Coulometer with diaphragmless generator cell.

METTLER TOLEDO

Aquagent® volumetric solutions: One-component reagents

In Karl Fischer one-component volumetric titration, all the substances needed for the reaction are included in a single reagent: the titrant. One-component reagents are easy to use and allow for greater flexibility in the choice of the most suitable solvent for each type of sample. On the other hand, due to the reactivity of their components, the factor of one-component reagents must be checked regularly.

Scharlau offers a range of one-component reagents suitable for both general as well as specific applications.

GENERAL PURPOSE

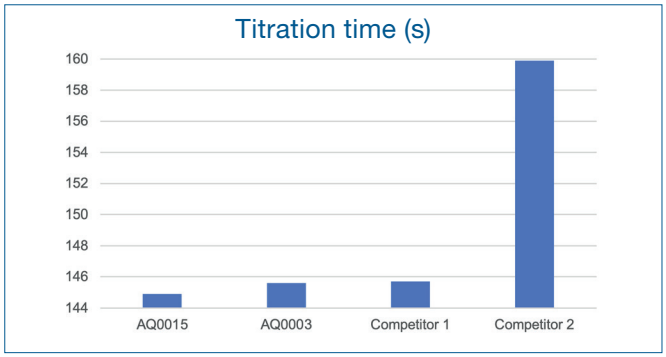
REAGENTS:

Aquagent® Complet 5

A general-purpose reagent for samples with medium or high water content. 1 ml titrates approximately 5 mg of water. It is generally used in combination with methanol as a solvent.

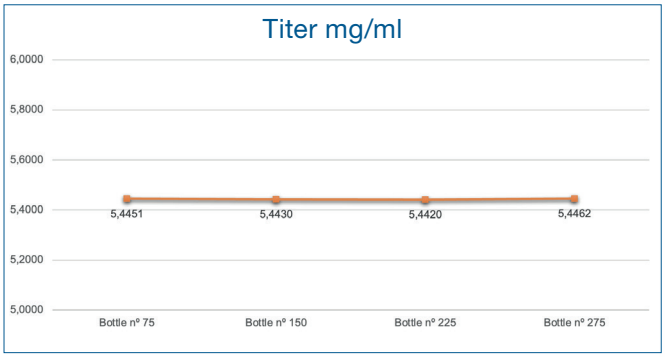
DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Complet 5	500 ml	AQ00150500
	1 l	AQ00151000
	2.5 l	AQ00152500

One of the important points to consider in the choice of one-component reagents is titration speed. In the following graph, we can see the titration speed of the new Aquagent® Complet 5, it is one of the fastest on the market, improving on our previous formulation.



Comparison of the titration times of the new Complet 5 (AQ0015) reagent versus the old formulation (AQ0003) and two of the main market competitors.

The new, more robust manufacturing process allows us to guarantee greater consistency between batches and within the same batch.
Within the same batch, the factor of all the bottles is kept constant, as seen in the graph below.



KF factor of different bottles in the same batch.

Advantages of the new one-component Aquagent® reagents

More stable and robust factor
Faster initial stabilisation
Greater reaction speed
More stable endpoint

Suitable for a wide variety of matrices
Longer shelf life
Minimized precipitation risk

Aquagent® Complet 2

A general-purpose reagent for samples with low or medium water content. 1 ml titrates approximately 2 mg of water. It is generally used in combination with methanol as a solvent.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Complet 2	500 ml	AQ00330500
	1 l	AQ00331000
	2.5 l	AQ00332500

Aquagent® Complet 1

A general-purpose reagent for samples with low water content. 1 ml titrates approximately 1 mg of water. It is generally used in combination with methanol as a solvent.

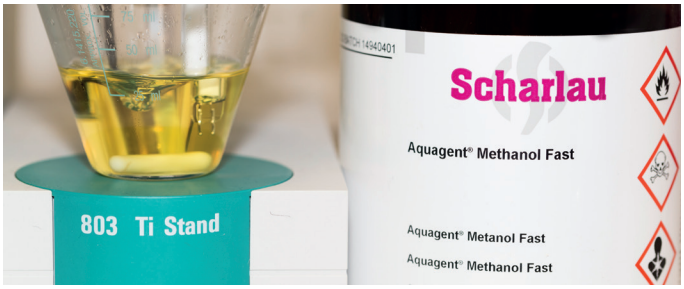
DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Complet 1	1 l	AQ00361000
	2.5 l	AQ00362500

SOLVENTS:

Dry methanol

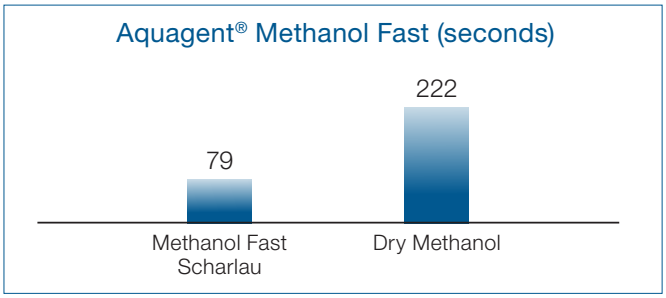
To correctly determine the water content of a sample, it must be previously dissolved in a dry solvent. The most common is dry methanol. If the sample is not soluble in methanol, it is possible to use other solvents (see the section on Specific Applications).

DESCRIPTION	PACKAGING	REFERENCE
Methanol, dry (max. 0.005% H_2O), for analysis	1 l	ME03041000
	2.5 l	ME03042500



Aquagent® Methanol Fast

Thanks to its improved formula, it allows faster Karl Fischer titration.



Indicates the time required to reach the endpoint in the KF titration reaction using one-component reagents and various types of methanol as the solvent. Sample: 20 mg of H_2O injected by weight.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Methanol Fast	1 l	AQ00111000
	2.5 l	AQ00112500

SPECIFIC APPLICATIONS

Aquagent® Complet 5K for Aldehydes and Ketones

Aldehydes and ketones react with methanol to form water. Therefore, when the sample contains aldehydes and/or ketones, the use of methanol can give inaccurate results. In the case of samples with aldehydes and ketones, a specific reagent is needed: Aquagent® Complet 5K. It is used in combination with Aquagent® Medium K, a specific solvent that does not contain methanol. This reagent can titrate 5 mg of water/ml.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Complet 5K	500 ml	AQ00340500
	1 l	AQ00341000

Aquagent® Medium K

Methanol reacts with aldehydes and ketones producing water as a by-product. Therefore, when the sample contains aldehydes or ketones, methanol must be substituted for another solvent; our Aquagent® Medium K.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Medium K	500 ml	AQ00050500
	1 l	AQ00051000

Aquagent® Buffer Acid (additive)

For an optimum Karl Fischer reaction, pH must be between 5 and 7. For a correct determination of the water content in strong acids, it is recommended to neutralize the working medium with our Aquagent® Buffer Acid.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Buffer, acid	500 ml	AQ00090500
	1 l	AQ00091000

Dry formamide (additive)

Formamide improves the solubility of carbohydrates, proteins and inorganic salts in methanol. This solvent can be added to methanol in no more than 50% in volume.

DESCRIPTION	PACKAGING	REFERENCE
Formamide dry (max. 0.02% H_2O), for analysis	1 l	FO00281000

Aquagent® volumetric solutions:
Two-component reagents

In the two-component system, the solvent not only acts as a medium to dissolve the sample, but it also contains part of the reagents needed for the reaction to occur. This allows for greater reagent shelf life and avoids the need of frequently determining the factor. Compared with one-component reagents, two-component reagents are more costly, but they also have important advantages: faster titration, less consumption of reagents and greater long-term stability.

Scharlau offers a range of two-component reagents suitable for both general as well as specific application use.

GENERAL USE

Aquagent® Titrant 5

A general use reagent which contains iodine and methanol. It titrates approximately 5 mg of water/ml. It must be used with Aquagent® Solvent.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Titrant 5	500 ml	AQ00590500
	1 l	AQ00591000
	2.5 l	AQ00592500

Aquagent® Titrant 2

A general use reagent which contains iodine and methanol. It titrates approximately 2 mg of water/ml. It must be used with Aquagent® Solvent.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Titrant 2	500 ml	AQ00600500
	1 l	AQ00601000

Scharlau offers a general solvent, as well as others for specific applications:

Aquagent® Solvent

A general reagent which contains SO_2, imidazole and methanol. It must be used with Aquagent® Titrant.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Solvent	1 l	AQ00291000
	2.5 l	AQ00292500

SPECIFIC APPLICATIONS

Aquagent® Solvent CM

It acts as a solvent for the titration of fats and oils. It is modified to improve the solubility of long-chain hydrocarbons. Contains chloroform.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Solvent CM	1 l	AQ00081000
	2.5 l	AQ00082500

Aquagent® Solvent Oil

It acts as a solvent for the titration of fats and oils, halogenated hydrocarbon free. Contains 1-hexanol.

Both reagents have different solubilisation capacities, which gives the analyst the option of choosing the one that suits them best.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Solvent Oil	1 l	AQ00101000

Advantages of the new two-component Aquagent® reagents

More stable and robust factor
Faster and more stable titration than one-component reagents
Greater accuracy

Aquagent® coulometric solutions:
For cells with or without diaphragm

Coulometric titration is the method used for samples with a low water content (<0.1%) or for determining the quantity of water in valuable samples. In coulometric titrations, the iodine required is generated in the titrated cell by the iodide oxidation on the anode. The concentration of water is precisely calculated from the current used for a determined time period. The cell measured contains two compartments: anode and cathode, which may be separated by a membrane or diaphragm. Therefore, the titration cells can have a diaphragm or not depending on whether they are separated.

Scharlau offers its Aquagent® reagents suitable for both cell types.

Aquagent® for cells with diaphragm

ANOLYTE:

Aquagent® Coulometric A
Anolyte for KF coulometric titrations

It is suitable for cells with diaphragm. This general-purpose reagent contains the components for the anode compartment of the electrolytic cell. It must be used with Aquagent® Coulometric CG.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Coulometric A, anolyte	500 ml	AQ00180500

Aquagent® Coulometric Oil
Anolyte for KF coulometric titrations

It is suitable for cells with diaphragm. This reagent for the anode compartment is especially formulated for petroleum samples and its derivatives. It must be used with Aquagent® Coulometric CG.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Coulometric Oil, anolyte	100 ml	AQ00250100

CATHOLYTE:

Aquagent® Coulometric CG
Catholyte for KF coulometric titrations

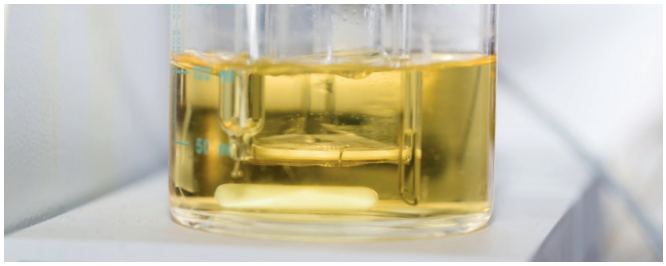
It is suitable for cells with diaphragm. This reagent contains the components for the cathode compartment of the electrolytic cell. It must be used with Aquagent® Coulometric A or Oil.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Coulometric CG, catholyte	100 ml	AQ00140100
	10 x 5 ml	AQ00140050

Aquagent® Coulometric AK
Anolyte for KF coulometric titrations

It is suitable for cells with diaphragm. This reagent contains the component for the anode compartment of the electrolytic cell needed to analyse samples with aldehydes and ketones. It must be used with Aquagent® Coulometric CG-K.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Coulometric AK, anolyte	500 ml	AQ00320500



CATHOLYTE:

Aquagent® Coulometric CG-K
Catholyte for KF coulometric titrations

It is suitable for cells with diaphragm. This reagent contains the component for the cathode compartment of the electrolytic cell needed to analyse samples with aldehydes and ketones. It must be used with Aquagent® Coulometric AK.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Coulometric CG-K, catholyte	10 x 5 ml	AQ00130050

Aquagent® for cells without diaphragm

ANOLYTE:

Aquagent® Coulometric AG
For KF coulometric titrations

It is suitable for cells with or without diaphragm.

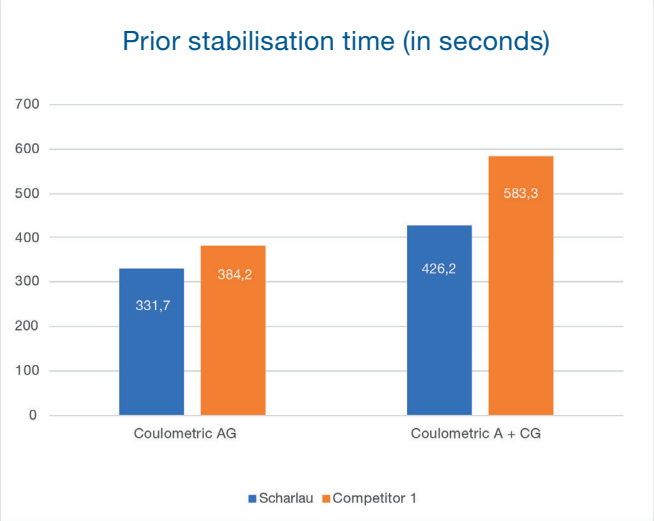
DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Coulometric AG	500 ml	AQ00580500
	1 l	AQ00581000

Aquagent® Coulometric AD
For KF coulometric titration

Optimised for cells without diaphragm.
It contains all the reaction components in a single reagent.

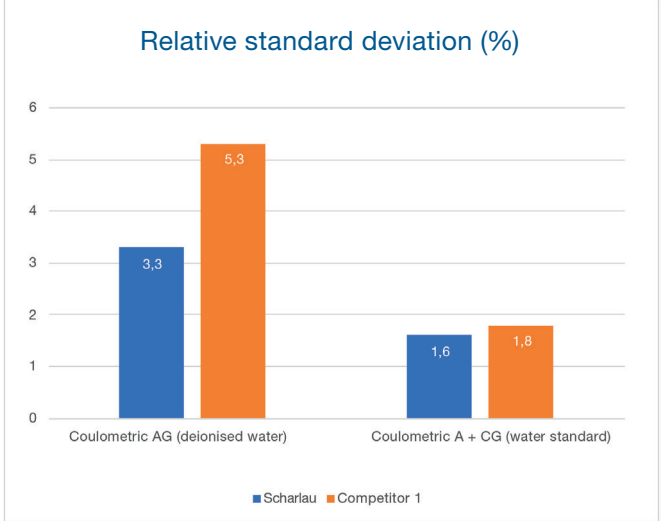
DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Coulometric AD, anolyte	500 ml	AQ00390500

The stabilisation time is one of the key factors when measuring the amount of water. The following graph shows that both the Aquagent® Coulometric AG and the combination of Aquagent® Coulometric A with CG need a shorter stabilisation time than the competition's reagents. This means the operator can start taking measurements sooner.



Comparison of the stabilisation time (s) of the Aquagent® Coulometric AG reagent and the combination of Scharlau's Aquagent® Coulometric A + CG reagent against that of a competitor.

We have achieved a lower relative standard deviation thanks to the recent improvements in our formulations. The graph below shows that there is only a small difference when using water as the standard, but the difference with the competition is considerable when using deionised water.
This measure gives an idea of the reproducibility of results with the different reagents.



Comparison of the relative standard deviation (%) of Scharlau's Aquagent® Coulometric AG reagent and the combination of Scharlau's Aquagent® Coulometric A + CG reagents against that of a competitor.

Aquagent® Oven for insoluble samples with a high boiling point

Aquagent® Coulometric AG Oven
For coulometric KF titrations

Within the area of coulometric titrations, the use of a Karl Fischer oven can broaden the range of samples that can be analysed.

The technique is mostly used for insoluble solids with a high boiling point, i.e., samples that do not dissolve in common Karl Fischer reagents, or which release water at higher temperatures.

The oven is connected to the Karl Fischer equipment and used to heat the test sample to the optimum temperature for the Karl Fischer reaction, so that it releases the water and can be drawn into the cell where the Karl Fischer reaction will occur. This temperature will vary according to the stability of the sample, always avoiding its decomposition.

The process starts by weighing the sample in a vial sealed with a septum, before placing it in the oven to evaporate the water. The water vapour released by the sample is subsequently transported to the Karl Fischer equipment by means of a carrier gas, usually air or molecular sieve-dried nitrogen.
This method ensures accurate, reliable results for the measurement of water content as it avoids side reactions and contamination.

This guarantees that the titration is carried out accurately and produces reliable results for the amount of water in the sample.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® Coulometric AG Oven, anolyte	500 ml	AQ00380500
	1 l	AQ00381000

Advantages of the new Aquagent® coulometric reagents

Excellent precision and accuracy
Good reproducibility of results
Shorter initial neutralisation time
Increased reaction time
Greater water capacity per reagent

Aquagent®: Scharlau standards for Karl Fischer titration

To determine the titre of the reagents, standards with a known quantity of water must be used. Water standards are every time more in demand, to obtain more reliable results which are comparable.

Our family of Aquagent® standards offer:
- **Solid standards:** di-Sodium tartrate dihydrate, stable, non-hygroscopic, with a water content of around 15.66%.
- **Liquid standards:** Aquagent® Standard 0.01% and 0.1% for coulometric titrations and Aquagent® Standard 1% for volumetric titrations. We package our standards in vials in an inert atmosphere, of 0.01%, 0.1% and 1% to keep their conditions optimum until opening. Each vial contains enough standard for one titration. Our Aquagent® Standard 0.5% is suitable for routine factorisation of reagents, as well as equipment validation.

DESCRIPTION	PACKAGING	REFERENCE
Aquagent® di-Sodium tartrate dihydrate*	25 g	AQ00260025
	100 g	AQ00260100
Aquagent® standard solution 0.01%* (0.1 mg/g)	10 x 8 ml	AQ00120080
Aquagent® standard solution 0.1%* (1 mg/g)	10 x 4 ml	AQ00190040
Aquagent® standard solution 1%* (10 mg/g)	10 x 8 ml	AQ00200080
Aquagent® standard solution 0.5% (5 mg/ml)	100 ml	AQ00210100
	500 ml	AQ00210500
Aquagent®, D(+)-Lactose monohydrate, 5% secondary standard for KF-Oven 150 °C*	10 g	AQ00270010

NIST verified

Advantages of the new Aquagent® standards

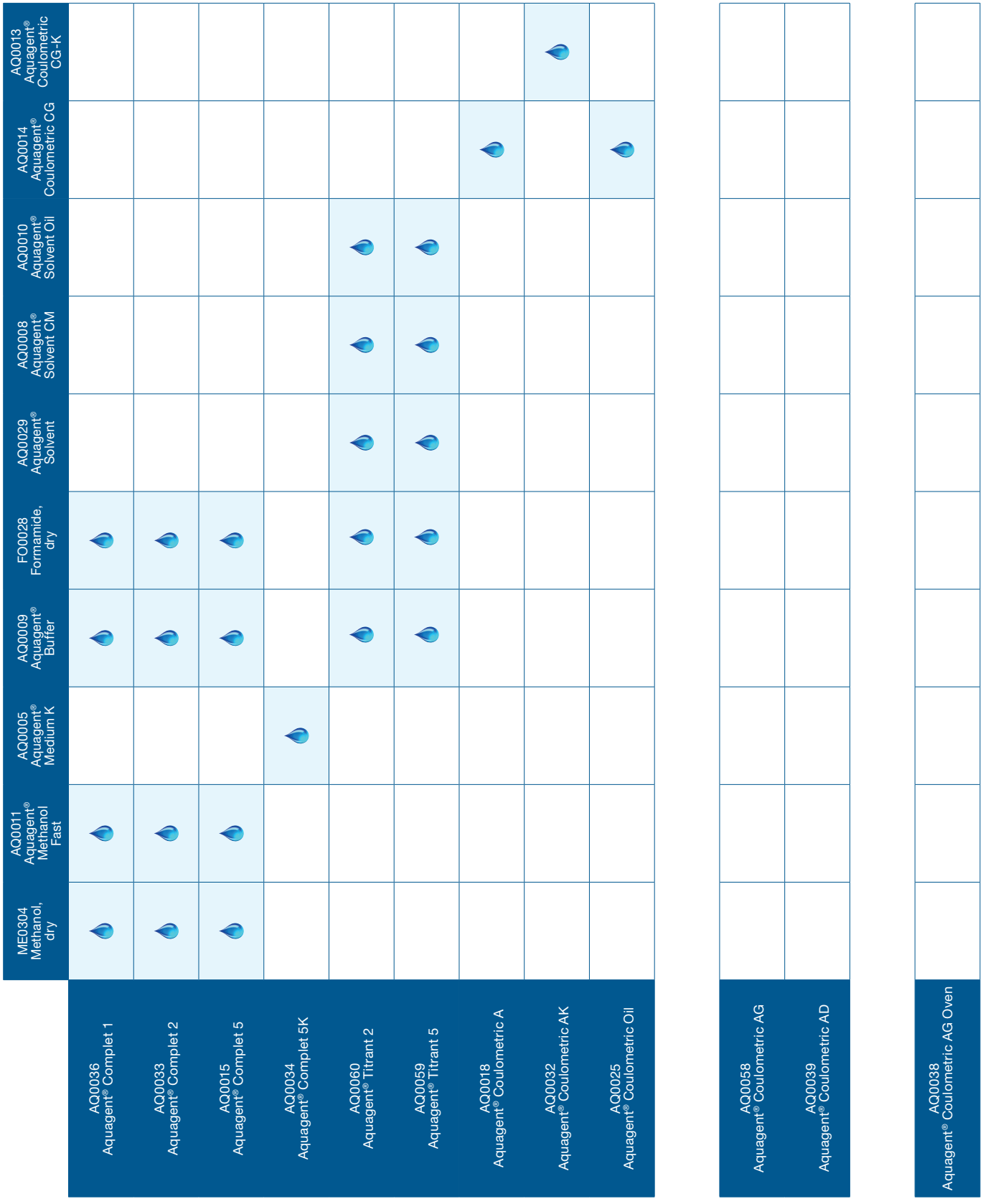
NIST verified
Longer shelf life

Practical packaging
Full Certificate of Analysis

OTHER RELATED PRODUCTS:

DESCRIPTION	PACKAGING	REFERENCE
3 Å molecular sieve, sodium aluminium silicate beads of 2 - 3 mm	250 g	TA01400250
	1 kg	TA01401000
Glass wool, washed	100 g	LA00750100
	250 g	LA00750250
	1 kg	LA00751000

Aquagent®: Quick guide



Aquagent®: Order information

AQUAGENT® PRODUCT FAMILY				PACKAGING	REFERENCE
Volumetric		Reagents	Aquagent® Complet 1	1 l	AQ00361000
				2.5 l	AQ00362500
			Aquagent® Complet 2	500 ml	AQ00330500
				1 l	AQ00331000
				2.5 l	AQ00332500
			Aquagent® Complet 5	500 ml	AQ00150500
				1 l	AQ00151000
				2.5 l	AQ00152500
			Aquagent® Complet 5K	500 ml	AQ00340500
				1 l	AQ00341000
		Solvents	Methanol, dry (max. 0.005% H_2O), for analysis	1 l	ME03041000
				2.5 l	ME03042500
			Aquagent® Metanol Fast	1 l	AQ00111000
				2.5 l	AQ00112500
			Aquagent® Medium K	500 ml	AQ00050500
				1 l	AQ00051000
		Additives	Aquagent® Buffer, acid	500 ml	AQ00090500
				1 l	AQ00091000
			Formamide, dry (max. 0.02% H_2O), for analysis	1 l	FO00281000
		Titrants	Aquagent® Titrant 2	500 ml	AQ00600500
				1 l	AQ00601000
			Aquagent® Titrant 5	500 ml	AQ00590500
				1 l	AQ00591000
				2.5 l	AQ00592500
		Solvents	Aquagent® Solvent	1 l	AQ00291000
				2.5 l	AQ00292500
			Aquagent® Solvent CM	1 l	AQ00081000
				2.5 l	AQ00082500
			Aquagent® Solvent Oil	1 l	AQ00101000
Coulometric	Cells with diaphragm		Aquagent® Coulometric A, anolyte	500 ml	AQ00180500
			Aquagent® Coulometric Oil, anolyte	100 ml	AQ00250100
			Aquagent® Coulometric CG, catholyte	10 x 5 ml	AQ00140050
				100 ml	AQ00140100
			Aquagent® Coulometric AK, anolyte	500 ml	AQ00320500
			Aquagent® Coulometric CG-K, catholyte	10 x 5 ml	AQ00130050
	Cells without diaphragm		Aquagent® Coulometric AG	500 ml	AQ00580500
				1 l	AQ00581000
			Aquagent® Coulometric AD	500 ml	AQ00390500
			Aquagent® Coulometric AG Oven, anolyte	500 ml	AQ00380500
				1 l	AQ00381000
Standards	Liquids		Aquagent® standard solution 0.01%	10 x 8 ml	AQ00120080
			Aquagent® standard solution 0.1%	10 x 4 ml	AQ00190040
			Aquagent® standard solution 1%	10 x 8 ml	AQ00200080
			Aquagent® standard solution 0.5%	100 ml	AQ00210100
				500 ml	AQ00210500
	Solids		Aquagent®, D(+)-Lactose monohydrate, 5% secondary standard for KF-Oven 150 °C	10 g	AQ00270010
			Aquagent® di-Sodium tartrate dihydrate	25 g	AQ00260025
				100 g	AQ00260100

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