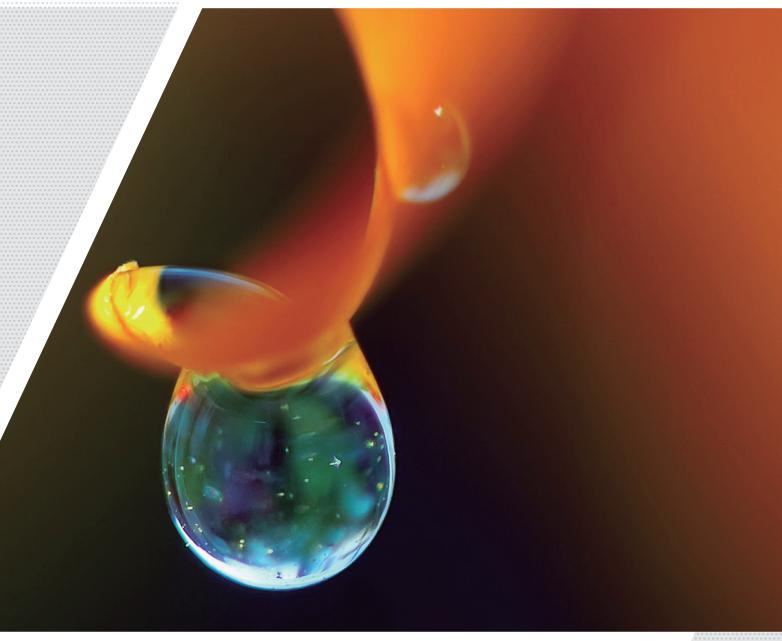
For volumetric and coulometric water determination











The Karl Fischer titration (KFT) is an analytical technique for the water determination in food, pharma, petrochemical and other samples. The water content is measured as an important quality parameter of several goods. While in food and pharma the water content has an important influence on the expiry date/stability of the final product, the water content of crude oil determines the price of this heavily used raw material.

By applying KFT, moisture and crystal water are determined all together. This can be done by carrying out volumetric or coulometric KFT. Volumetric KFT is applicable to solid and liquid samples with water content greater than 1%, while coulometric KFT is used only in liquid samples with water content smaller than 1%.

AQUAMETRIC® is the in-house produced range of pyridine-free Karl Fischer reagents and applicable for volumetric and coulometric KFT. AQUAMETRIC® is a complete solution for all laboratories carrying out water determinations using KFT, and consists of water standards, working media and dried solvents.



ITW Reagents product offering

- Reagents for volumetric Karl Fischer titration (water content between 1 and 100 mg, ideally 10 mg of water)
- Reagents for coulometric Karl Fischer titration (water content between approximately 10 µg and 10 mg, ideally 1 mg of water)
- Specific reagents for volumetric titration of oils, ketones and aldehydes.
- · Auxiliary reagents
- Water standards
- Pack sizes: 10 mL ampoules, 100 g, 500 mL, 1 L and 2.5 L bottles

Who can use AQUAMETRIC® branded Karl Fischer reagents?

Quality control departments in industries such as pharmaceuticals, chemicals, petrochemicals and oil, food and beverage, feed, cosmetics and medicines, paints, lacquers, adhesives, plastics and resins manufacturers and research laboratories can use AQUAMETRIC® branded Karl Fischer reagents for the determination of the water content in raw materials, during production and in finished products.

Main advantages

AQUAMETRIC® branded Karl Fischer reagents are pyridine-free products. Imidazole is used as a replacement of pyridine to minimize health risks to users.

ITW Reagents offers a wide range of **reliable** reagents with **stable and precise titer** (titer is influenced by water from ambient air) and **low drift**.

ITW Reagents products are **manufactured and controlled in Europe** according to ISO 9001 (quality), ISO 14001 (environmental) and ISO 45001 (occupational health and safety).

A constant stock level and a **long shelf life**, up to 5 years depending on the reagent, are guaranteed. ITW Reagents also offers excellent **customer service and technical support** committed to customers' needs.



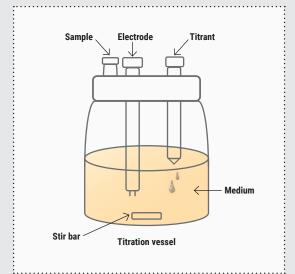
Volumetric Karl Fischer titration

The volumetric Karl Fischer titration is used for samples with a water content greater than 1% in solid and liquid samples. As the name indicates, the amount of water is determined by volumetry, based on a redox reaction

(iodine-iodide) sensitive to very low moisture levels.

In presence of a base and a solvent (i.e. imidazole and methanol)

The amount of iodine consumed in the titration is proportional to the water content of the sample. The end point of this titration is detected by a polarized double platinum pin electrode.

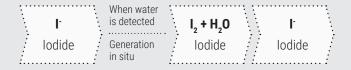


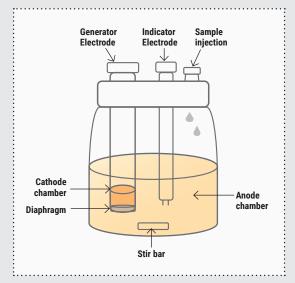
Coulometric Karl Fischer Titration

The coulometric Karl Fischer titration is used for liquid samples with a water content of less than 1%. As the name indicates, the amount of water is determined by coulometry. Coulometric KFT needs a reaction cell with two electrodes to carry out the water determination:

Electrode 1: **Indicator electrode**, detects the moisture.

Electrode 2: **Generator electrode**, transmits a very low electric current causing the iodide oxidation into iodine. The iodine required for the reaction in the sample is generated in situ (in the titration vessel) using a reagent solution containing iodide.





In the coulometric titration the amount of water in the sample is calculated by measuring the current needed for the electrochemical generation of iodine (I_2) from iodide (I^-) .

There are two different types of coulometric cells, with and without diaphragm.





Products for volumetric Karl Fischer titration

The range of products for volumetric Karl Fischer titration consists of two major groups:

- one-component reagents
- two-component reagents

In **one-component reagents** all the necessary reagents are mixed (iodine, sulfur dioxide and imidazole) and are dissolved in diethyleneglycol mono-ethyl ether. The advantage of using one-component reagents is that they give more flexibility in choosing the appropriate solvent to dissolve the sample, and having the necessary reagents in a single solution. The one-component reagents are the most common reagents used for the volumetric Karl Fischer titration.

In **two-component reagents** the chemicals used are in different packaging, which increases stability and shelf-life. The titrant solution contains iodine dissolved in methanol. The rest of necessary compounds (imidazole and sulfur dioxide in methanol) are in the solvent, typically used in combination with the titrant.



AQUAMETRIC® one-component reagents

Below you will find three tables with a list of products showing AQUAMETRIC $^{\circ}$ one-component reagents.

- Reagents for normal or **standard** procedure (table 1). These are the most commonly used reagents for the volumetric one-component water determination covering many sample types for Karl Fischer titration excluding ketones, aldehydes, amines, sugars and oils.
- Reagents suitable for an excellent water determination in **ketones and aldehydes** (table 2). By using these reagents, side reactions that form extra water that is added to the solvent, thus giving misleading results, can be avoided.
- Reagents suitable for water determination in industrial oils (table 3). In these cases, a
 different solvent is used to ensure the solubilization of the oils and permit the Karl Fischer
 reaction to take place optimally.

AQUAMETRIC® Composite 2 is suitable for low and medium water content samples.

AQUAMETRIC® Composite 5 is more advisable for high water content samples, being, this last one, the most used. The appropriate working medium used with these reagents is dry methanol.

The typical procedure consist of filling the burette with AQUAMETRIC® Composite 2 or 5 and dissolve the sample into the titration vessel with dry methanol. The endpoint is determined by titration according to the usual method.

The advantages of using one-component reagents is that they give more flexibility in choosing the appropriate solvent to dissolve the sample, and having the necessary reagents in a single solution.



Table 1: One-component reagents for volumetric Karl Fischer titration

Product code	Product name	Medium	Titrant	Pack size
285813.1611	AQUAMETRIC Composite 2 for Karl Fischer volumetric analysis		•	1 L
285812.1610			•	500 mL
285812.1611	AQUAMETRIC Composite 5 for Karl Fischer volumetric analysis		•	1 L
285812.1612			•	2.5 L
481091.1611	Methanol dry (max. 0.005% water) - Karl Fischer's Reagent (Reag. Ph. Eur.), ACS, ISO	•		1 L
481091.1612		•		2.5 L

Table 2: One-component reagents for the volumetric Karl Fischer titration of ketones and aldehydes

Product code	Product name	Medium	Titrant	Pack size
285814.1611	AQUAMETRIC Composite 5K for Karl Fischer volumetric analysis		•	1 L
285821.1611	AQUAMETRIC Working Medium for Karl Fischer volumetric analysis	•		1 L

Table 3: One-component reagents for the volumetric Karl Fischer titration of industrial oils

Product code	Product name	Medium	Titrant	Pack size
285813.1611	AQUAMETRIC Composite 2 for Karl Fischer volumetric analysis		•	1 L
285812.1610			•	500 mL
285812.1611	AQUAMETRIC Composite 5 for Karl Fischer volumetric analysis		•	1 L
285812.1612			•	2.5 L
286154.1611	AQUAMETRIC Solvent Oil B for Karl Fischer volumetric analysis	•		1 L











AQUAMETRIC® two-component reagents

In the two-component reagents, the ingredients are separated in different packagings, which gives higher stability and better shelf life.

The **AQUAMETRIC® Titrant solution** contains iodine dissolved in methanol. The rest of necessary compounds (imidazole and sulfur dioxide in methanol) are in the working medium **AQUAMETRIC® Solvent**, typically used with AQUAMETRIC® Titrant.

Below two tables are included showing AQUAMETRIC® two-component reagents.

In table 4 are the most common reagents used in a two-components Karl Fischer titration for water determination.

In table 5 the recommended reagents for water determination in food oils and fats are shown. These reagents promote optimal solubilization of the sample and thus facilitate the determination of water.

AQUAMETRIC® Titrant 2 is recommended for low and medium water content samples.

AQUAMETRIC® Titrant 5 is more advisable for samples with higher water contents.

The typical procedure consists of filling the burette with AQUAMETRIC® Titrant 2 or 5 and the vessel with AQUAMETRIC® Solvent. The endpoint is determined by titration according to the usual method.

The advantages of using two-component system are **reduced reaction times** and an increased **accuracy** of the results.

Table 4: Two-component reagents for volumetric Karl Fischer titration

Product code	Product name	Medium	Titrant	Pack size
285816.1611	AQUAMETRIC Titrant 2 for Karl Fischer volumetric analysis		•	1 L
285815.1611			•	1 L
285815.1612	AQUAMETRIC Titrant 5 for Karl Fischer volumetric analysis		•	2.5 L
285817.1611		•		1 L
285817.1612	AQUAMETRIC Solvent for Karl Fischer volumetric analysis	•		2.5 L

Table 5: Two-component reagents for volumetric Karl Fischer titration for food oils and fat

Product code	Product name	Medium	Titrant	Pack size
285816.1611	AQUAMETRIC Titrant 2 for Karl Fischer volumetric analysis		•	1 L
285815.1611			•	1 L
285815.1612	AQUAMETRIC Titrant 5 for Karl Fischer volumetric analysis		•	2.5 L
285819.1611		•		1 L
285819.1612	AQUAMETRIC Solvent CM for Karl Fischer volumetric analysis	•		2.5 L



Products for coulometric Karl Fischer titration

The coulometric KFT is an absolute method determining the amount of water in the sample by a potentiometric end point indication. The iodine required for the reaction is generated in situ (in the titration vessel) using a reagent solution containing iodide.

The coulometric method is used for liquid samples containing less than 1% water. Sample size is an important consideration, as too much sample will prolong the time required for analysis. Normally a sample containing, in absolute quantities, between 0.1 and 5 mg of water is used, ideally 1 mg.

For coulometric determination in instruments with a diaphragm, two reagent solutions, an anolyte and a catholyte, are required. The anolyte is introduced into the anode chamber of the cell and the catholyte into the cathode chamber prior to the determination. In instruments without a diaphragm the Karl Fischer reaction takes place in the same electrolyte and there is no distinction between anolyte and catholyte.

The list of AQUAMETRIC® coulometric reagents is shown in the tables below. The reagents are necessary to perform an optimal Karl Fischer determination.

Table 6: Reagents for coulometric Karl Fischer titration in reaction cells with diaphragm

Product code	Product name	Anolyte	Catholyte	Pack size
286181.1610	AQUAMETRIC Coulomat A for Karl Fischer coulometric analysis	•		500 mL
286180.1610	AQUAMETRIC Coulomat AG for Karl Fischer coulometric analysis	•		500 mL
287192.2504	AQUAMETRIC Coulomat CG for Karl Fischer coulometric analysis		•	10 x 5 mL

Table 7: Reagents for coulometric Karl Fischer titration in reaction cells without diaphragm

Product code	Product name	Anolyte	Catholyte	Pack size
286180.1610	AQUAMETRIC Coulomat AG for Karl Fischer coulometric analysis	•		500 mL







Karl Fischer volumetric titration in brief

One-component reagents

All the necessary components for the Karl Fischer reaction are in the titrant reagent (iodine, sulfur dioxide and imidazole):

AQUAMETRIC® Composite.

The medium generally used is dry methanol.

- Easy to use: All-in-one
- Flexibility to select the appropriate solvent (as medium) to extract the water from the sample

Two-component reagents

The reagents necessary for the Karl Fischer reaction are separated in two components:

AQUAMETRIC® Titrant containing iodine dissolved in methanol.

AQUAMETRIC® Solvent, medium containing the rest of necessary compounds (imidazole and sulfur dioxide in methanol).

- · High stability and shelf life
- · Faster titration and high accuracy

Karl Fischer coulometric titration in brief

Cells with diaphragm

The anode and the cathode chambers are separated with a diaphragm.

Oxidation of I^- to I_2 occurs at the anode and the reduction of protons to H_2 occurs at the cathode.

Two reagents are needed:

AQUAMETRIC® Coulomat A or AG for the anode chamber

AQUAMETRIC® Coulomat CG for the cathode chamber

Highest accuracy

Cells without diaphragm

The anodic and cathodic compartments are not separated, and only one reagent, the anolyte, is needed.

The reactions are performed in the same electrolyte:

AQUAMETRIC® Coulomat AG

More convenient



Water standards

Validation of methods and instruments is of vital importance for quality control in any type of industry, such as pharmaceutical, food or petrochemical. Instruments are often validated by the manufacturers themselves, but the methods and reagents must be validated by the end users. In the case of KFT, such validation can be carried out using water standards.



These standards are used to calculate the titer of AQUAMETRIC® Karl Fischer reagents for the volumetric titration and also to check the reliability of water determination using the coulometric method. AQUAMETRIC® Karl Fischer water standards are traceable to NIST SRMs and make the work of the laboratory staff much more reliable.

Table 8: Karl Fischer water standards

Product code	Product name	Volumetric	Coulometric	Pack size
395459.2522	Karl Fischer Water Standard 1.0 mg/g (Reag. Ph. Eur.)		•	10 x 10 mL
395458.2522	Karl Fischer Water Standard 10 mg/g (Reag. Ph. Eur.)	•		10 x 10 mL
241719.1608	Sodium Tartrate 2-hydrate (Reag. Ph. Eur.) standard for volumetry, ACS		•	100 g

For other type of titrations, please consult our program of volumetric solutions at www.itwreagents.com.

Volumetric solutions

Top-quality products for unparalleled precision. All you need for volumetric analysis:

- · Volumetric solutions ready to use
- Concentrated volumetric solutions
- Indicators
- Standards







Dried solvents

Other solvents for Karl Fischer titration can be found in the table below. They can be used for special applications instead of or mixed with methanol. PanReac AppliChem branded dried solvents are specified for a low water content to enable accurate volumetric Karl Fischer titrations.

Table 9: Dried solvents for volumetric Karl Fischer titration

Product code	Product name	CAS number	Pack size
481007.1611	Acetone dry (max. 0.01% water)	67-64-1	1 L
481881.1612	Acetonitrile dry (max. 0.005% water), ACS	75-05-8	2.5 L
481254.1611	Dichloromethane dry (max. 0.005% water) stabilized with ~ 20 ppm of amylene, ACS, ISO	75-09-2	1L
482770.0311	Diethyl Ether dry (max. 0.0075% water) stabilized with ~ 6 ppm of BHT, ACS, ISO	60-29-7	1L
481785.1611		68-12-2	1L
481785.1612	N,N-Dimethylformamide dry (max. 0.005% water) over molecular sieves		2.5 L
481086.1611	Ethanol absolute dry (max. 0.02% water)	64-17-5	1L
481091.1611	Methanol dry (max. 0.005% water) - Karl Fischer's Reagent (Reag. Ph. Eur.),	(7.5(.1	1L
481091.1612	ACS, ISO	67-56-1	2.5 L
481457.1611	Pyridine dry (max. 0.01% water) (Reag. Ph. Eur.), ACS	110-86-1	1 L
483537.1612	Tetrahydrofuran dry (max. 0.0075% water) stabilized with ~ 300 ppm of BHT, ACS	109-99-9	2.5 L
481745.1611	Toluene dry (max. 0.005% water), ACS, ISO	108-88-3	1 L

You may also find other interesting PanReac AppliChem branded solvents that we can offer for your daily lab work. See our full range of solvents at www.itwreagents.com.

HPLC / UHPLC solvents

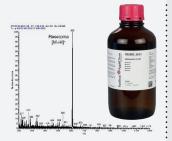
Improved specifications for UHPLC supergradient grade solvents

- · Lower non-volatile matter content
- Improved transparency at low wavelengths
- · New control of base line drift



LC-MS solvents

- Very low metal concentration <100 ppb
- Microfiltered 0.2 µm
- Suitability for LC-MS
- High UV transmittance and an excellent baseline



Solvents for Headspace GC

For the analysis of residual solvents in actives, excipients and medicines according to the ICH (International Council for Harmonization of Technical), USP and Ph. Eur.



For pesticide residues analysis

For food and environmental industry. Controlled by GC: Using ECD detector from Lindane to DDT, no peaks > 5 ng/L of Lindane. Using FID detector in the interval from 2-Octanol to Tetradecanol.





How to order

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For further contact details of our distributors please visit **itwreagents.com**

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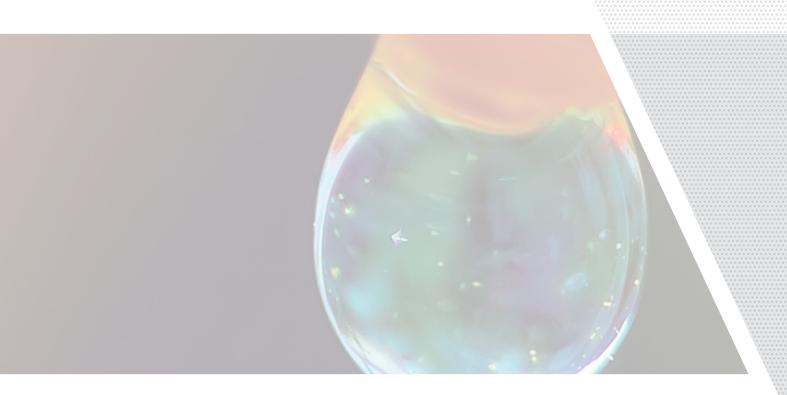
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