

Determination of Water Content in Kerosene Using Karl Fischer Titration

Product Group

Hydrocarbons, Petroleum products

General Information concerning the product group

Hydrocarbons

Saturated hydrocarbons can in most cases be titrated according to standard methods. To overcome solubility problems of unpolar or weakly polar substances, the addition of a solubiliser to the solvent is necessary. In the case of long-chain and cyclic hydrocarbons, long-chain alcohols (e.g. propyl alcohol or decyl alcohol) or chloroform are thus recommended. Toluene, xylene or chloroform improve the solubility of aromatic compounds. Unsaturated hydrocarbons can usually be titrated in the same way. Interferences due to double bonds only occur with some very reactive compounds. In the case of interferences (unstable end point or none at all) a methanol-free, alcoholic solvent (e.g. CombiSolvent or CombiSolvent Keto) should be utilised instead of methanol. Recommended methods are both the volumetric titration with one or two component reagents, as well as the coulometric analysis. The latter is predominantly applied for low water concentrations (< 0.1 %).

Petroleum products

Petroleum products are mixtures of long-chain or aromatic hydrocarbons. They are hardly soluble in methanol. Water determination by Karl Fischer therefore requires the addition of solubilisers. For light oils, long-chain alcohols are suitable. For dissolving of heavier oils toluene, xylene or chloroform are added. For the volumetric titration specific KF solvents for oils are available. Due to the very low water concentration titrants with a low factor (2 mg/ml or 1 mg/ml) are recommended. During coulometric determination without diaphragm 20% solubiliser can be added to the working medium, or 40% solubiliser to the anolyte in the case of coulometry with diaphragm. Note that oils are often heterogeneous compounds with uneven distribution of water and should thus be homogenised (e.g. with Ultra-Turrax) prior to KF determination. Additives in oils can cause side reactions during KF determination. Here, the direct coulometric analysis is not possible, the volumetric titration only conditionally. As an alternative, the KF oven technique can be utilised in combination with coulometry, whereby the release of water is best achieved at temperatures between 120 and 140 °C.

Special Information concerning the sample and the methods

Water determination can be carried out volumetrically or coulometrically. Addition of solubilisers (long-chain alcohols or chloroform) is necessary.

Titration one component system

Reagents:

Titration

188002 Aquastar™ - CombiTitration 2 - One component reagent for volumetric Karl Fischer Titration, 1 mL = approx. 2mg water

Solvent

50 mL 188020 Aquastar™ - CombiSolvent Oil - Solvent for volumetric Karl Fischer Titration with one component reagents for oils

or

25 mL 188009 Aquastar™ - CombiMethanol - Solvent for volumetric Karl Fischer Titration with one component reagents

Titration Parameters:

Stirring time: 60 sec.

Default titration settings, e.g.:

$I(\text{pol}) = 20 - 50 \mu\text{A}$, $U(\text{EP}) = 100 - 250 \text{ mV}$

Stop criterion: drift < 20 $\mu\text{L}/\text{min}$

Sample size:

10 mL

Procedure:

The titration medium is first placed into the cell and titrated dry by means of the titrant. Then the sample is added with a syringe without hollow needle (exact sample weight determination by weighing of syringe before and after injection) and the titration is started. A stirring time of 60 seconds is recommended.

Titration two component system

Reagents:

Titrant

188011 Aquastar™ - Titrant 2 - Titrant for volumetric titration with two component reagents, 1 mL = approx. 2 mg water

Solvent

50 mL 188016 Aquastar™ - Solvent oils and fats - Solvent for volumetric Karl Fischer titration with two component reagents for oils & fats

or

25 mL 188015 Aquastar™ - Solvent - Solvent for volumetric Karl Fischer titration with two component reagents

Titration Parameters:

Stirring time: 60 sec.

Default titration settings, e.g.:

$I(\text{pol}) = 20 - 50 \mu\text{A}$, $U(\text{EP}) = 100 - 250 \text{ mV}$

Stop criterion: drift < 20 $\mu\text{L}/\text{min}$

Sample size:

10 mL

Procedure:

The titration medium is first placed into the cell and titrated dry by means of the titrant. Then the sample is added with a syringe without hollow needle (exact sample weight determination by weighing of syringe before and after injection) and the titration is started. A stirring time of 60 seconds is recommended.

Coulometry with diaphragm

Reagents:

Catholyte

5 mL 109255 Aquastar™ - CombiCoulomat frit - Coulometric Karl Fischer reagent for cells with diaphragm

Anolyte

80 mL 109255 Aquastar™ - CombiCoulomat frit - Coulometric Karl Fischer reagent for cells with diaphragm

and

20 mL 102445 Chloroform - as solubiliser

Titration Parameters:

Stirring time: 60 sec.

Default coulometer settings for cell with diaphragm:

For end point indication, e.g.:

$I(\text{pol}) = 5 - 10 \mu\text{A}$, $U(\text{EP}) = 50 - 100 \text{ mV}$

Stop criterion: drift < 10 $\mu\text{g}/\text{min}$

Sample size:

2 mL

Procedure:

The Karl-Fischer reagent is placed into the cathode and anode compartment of the titration cell with diaphragm. The coulometer is started and the solvent is titrated dry. After preliminary titration and stabilisation of drift the sample is injected into the titration cell with a syringe without hollow needle (exact sample weight determination by weighing of syringe before and after injection) and the water determination is started. A stirring time of 60 seconds is recommended.

Coulometry without diaphragm

Reagents:

Working medium

80 mL 109257 Aquastar™ - CombiCoulomat fritless - Coulometric Karl Fischer reagent for cells with or without diaphragm

and

20 mL 102445 Chloroform - as solubilizer

Titration Parameters:

Stirring time: 60 sec.

Default coulometer settings for cell without diaphragm:

For end point indication, e.g.:

$I(\text{pol}) = 5 - 10 \mu\text{A}$, $U(\text{EP}) = 50 - 100 \text{ mV}$

Stop criterion: drift < 10 $\mu\text{g}/\text{min}$








Sample size:

2 mL

Procedure:

The Karl-Fischer reagent is placed into the titration cell without diaphragm. The coulometer is started and the solvent is titrated dry. After preliminary titration and stabilisation of drift the sample is injected into the titration cell with a syringe without hollow needle (exact sample weight determination by weighing of syringe before and after injection) and the water determination is started. A stirring time of 60 seconds is recommended.

Materials

Product #	Image	Description ▼	Molecular Formula
1.02445		Chloroform for analysis EMSURE® ACS,ISO,Reag. Ph Eur	CHCl ₃
1.09255		CombiCoulomat frit Karl Fischer reagent for the coulometric water determination for cells with diaphragm Aquastar™	
1.09257		CombiCoulomat fritless Karl Fischer reagent for coulometric water determination for cells with and without diaphragm Aquastar™	
1.88002		CombiTitrant 2 one component reagent for volumetric Karl Fischer titration 1 ml ca. 2 mg H ₂ O Aquastar™	
1.88011		Titrant 2 titrant for volumetric Karl Fischer titration with two component reagents 1 ml ca. 2 mg H ₂ O Aquastar™	
1.88015		Solvent AQUASTAR®	
1.88016		Solvent Oils & Fats Solvent for volumetric Karl Fischer titration with two component reagents for oils and fats Aquastar™	
1.88020		CombiSolvent Oil Solvent for volumetric Karl Fischer titration with one component reagents for oils Aquastar™	