

Water in Heavy fuel

Karl Fischer application

Product group

Petroleum products

General Information concerning the product group

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 analyte 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 of heavy fuel can be carried out directly by means of the volumetric method. Toluene and chloroform are suitable solubilisers. Specific solvents for oils are available for one and two component titration. The sample may also be dissolved externally in toluene prior to analysis.

Titration one component system

Reagents

Titrant: Aquastar - CombiTitrant 5 188005

One component reagent for volumetric Karl Fischer titration, 1 mL = approx. 5 mg water

or Aguastar - CombiTitrant 2 188002

One component reagent for volumetric Karl Fischer titration, 1 mL = approx. 2 mg water

Solvent: Aquastar - CombiSolvent oils 188020 50 mL

Solvent for volumetric Karl Fischer titration with one component reagents for oils

Titration parameters

Stirring time: 60 sec.

Default titration settings, e.g.:

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

Stop criterion: drift < 20 µL/min

Sample size

5 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 (exact sample weight determination by weighing of syringe before and after injection) or volumetric

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pipette and the titration is started. For complete dissolution of the sample a stirring time of 60 seconds is recommended.

Titration two component system

Reagents

Titrant: Aquastar - Titrant 5 188010

Titrant for volumetric titration with two component reagents, 1 mL = approx. 5 mg water

or Aquastar - Titrant 2 188011

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

Solvent: Aquastar - Solvent oils & fats 188016 40 mL

Solvent for volumetric Karl Fischer titration with two component reagents for oils & fats

and Toluene 108325 10 mL

as solubiliser

Titration parameters

Stirring time: 60 sec.

Default titration settings, e.g.:

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

Stop criterion: drift < 20 µL/min

Sample size

5 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 (exact sample weight determination by weighing of syringe before and after injection) or volumetric pipette and the titration is started. For complete dissolution of the sample a stirring time of 60 seconds is recommended.

Ordering Information

Product	Catalog No.
Toluene for analysis EMSURE® ACS,ISO,Reag. Ph Eur	108325
CombiTitrant 2 one component reagent for volumetric Karl Fischer titration 1 ml □ ca. 2 mg H2O Aquastar [™]	188002
CombiTitrant 5 one-component reagent for volumetric Karl Fischer titration 1 ml □ ca. 5 mg H2O Aquastar [™]	188005
Titrant 5 titrant for volumetric Karl Fischer titration with two component reagents 1 ml □ ca. 5 mg H2O Aquastar [™]	188010
Titrant 2 titrant for volumetric Karl Fischer titration with two component reagents 1 ml □ ca. 2 mg H2O Aquastar [™]	188011
Solvent Oils & Fats Solvent for volumetric Karl Fischer titration with two component reagents for oils and fats Aquastar™	188016
CombiSolvent Oil Solvent for volumetric Karl Fischer titration with one component reagents for oils Aquastar™	188020