INTERNATIONAL STANDARD

ISO 4317

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Surface-active agents and detergents — Determination of water content — Karl Fischer methods

Agents de surface et détergents — Détermination de la teneur en eau — Méthodes de Karl Fischer

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 4317 was prepared by Technical Committee ISO/TC 91, *Surface active agents*.

This third edition cancels and replaces the second edition (ISO 4317:1991), which has been technically revised to incorporate the following changes:

- the coulometric titration method in 5.3 has been added;
- the former Annex B has been deleted;
- interlaboratory test results have been added in a new Annex B;
- the Bibliography has been added.

Surface-active agents and detergents — Determination of water content — Karl Fischer methods

1 Scope

This International Standard specifies two titration methods (volumetric and coulometric) using Karl Fischer reagent for the determination of the water content of surface active agents and detergents.

These methods are applicable to products in the form of powders, pastes and solutions.

They are applicable only if so indicated in the specific standard for each product.

As alkaline compounds react with Karl Fischer reagent, the methods give values which are too high in the case of samples containing alkali metal silicates, carbonates, hydroxides or borates. Therefore, samples need to be analysed for the presence of such alkali metal salts prior to the determination of the water content.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 607, *Surface active agents and detergents — Methods of sample division*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

water content

mass fraction of free water, water of crystallization, absorbed water or occluded water, calculated from the amount of Karl Fischer reagent used in accordance with this International Standard

NOTE The water content is expressed as a percentage by mass.

4 Principle

The water content of a test portion is determined on the basis of the reaction of water with Karl Fischer reagent.

5 Test methods

5.1 General

Two titration methods using Karl Fischer reagent are described: a volumetric titration method and a coulometric titration method.

The volumetric titration method shall be used for samples with a water content of 2 % or more; the coulometric titration method shall be used for samples with a water content of 0,1 % or less. Both methods may be used for water contents in the range of 0,1 % to 2 %.

5.2 Volumetric titration method

5.2.1 Principle

Any water present in a test portion is reacted with a solution of iodine and sulfur dioxide in an appropriate mixture (Karl Fischer reagent), previously standardized by titration with an exactly known mass of water. The water content is calculated as a percentage by mass from the amount of reagent used.

5.2.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only grade 2 distilled water (5.2.2.1).

5.2.2.1 **Distilled water** or water of at least equivalent purity, complying with the requirements of ISO 3696 for grade 2.

5.2.2.2 **Karl Fischer reagent**, preferably commercially prepared.

A reagent containing pyridine may be prepared as described in Annex A for information purposes. A non-pyridine reagent can be used if it has been shown to be suitable; if so, this shall be indicated in the test report.

NOTE A suitable non-pyridine-containing reagent containing iodine, sulfur dioxide and an amine dissolved in 2-methoxyethanol is commercially available.

5.2.2.3 **Disodium tartrate dihydrate** or **oxalic acid dihydrate**, to be used as the primary standard for Karl Fischer reagent, or **water** (5.2.2.1).

5.2.2.4 **Solvent**, e.g. 2-methoxyethanol, propan-1-ol or methanol.

If it is known that these solvents interfere with the determination (e.g. for the determination of water in aldehydes or ketones), trichloromethane or dichloromethane may be used; if so, this shall be indicated in the test report.

WARNING — Trichloromethane and dichloromethane are harmful if inhaled or swallowed, and are irritating to the skin, with the risk of irreversible effects and the danger of serious damage to health in the event of prolonged exposure.

5.2.3 Apparatus

Use usual laboratory apparatus and, in particular, the following.

5.2.3.1 **Karl Fischer apparatus**, fully automatic or semi-automatic, consisting of

— a titrator with double platinum electrode,

— a 20 ml piston burette,

- drying tubes containing activated silica gel, calcium chloride or magnesium perchlorate,
- a titration vessel,
- a magnetic stirring apparatus.

5.2.3.2 Micrometer syringe, capacity 100 µl.

5.2.3.3 Graduated pipette, capacity 20 ml.

5.2.3.4 Syringe, consisting of a glass tube of nominal capacity about 10 ml, having an internal diameter of 2 mm to 4 mm, with a movable needle attached to the syringe by suitable means.

5.2.3.5 Balance, accurate to 0,1 mg.

5.2.4 Sampling

The laboratory sample of surface-active agent or detergent shall have been prepared and stored in accordance with the instructions given in ISO 607.

5.2.5 Procedure

5.2.5.1 General

Atmospheric moisture is the biggest source of interference in Karl Fischer titration. Take care to thoroughly dry the apparatus used and to handle the test portions and solvents speedily. To use the Karl Fischer apparatus, follow the manufacturer’s instructions.

5.2.5.2 Determination of the water equivalent of Karl Fischer reagent (5.2.2.2)

Determine the water equivalent of the reagent on each new container of reagent and repeat at least weekly.

It is necessary to check the titre of Karl Fischer reagent, as the titre of standard solutions is subject to change. The type of apparatus used and desired analytical accuracy govern the frequency of such titre checks.

Place the magnetic stirring bar into the titration vessel of the Karl Fischer apparatus (5.2.3.1) using pincers, and transfer 10 ml of one of the solvents (5.2.2.4) used for the determination into the titration vessel and titrate with Karl Fischer reagent (5.2.2.2).

Where absolutely necessary, e.g. for the determination of water in aldehydes or ketones, use chloroform or dichloromethane (see 5.2.2.4) as solvent.

Either:

Weigh 200 mg to 250 mg of disodium tartrate dihydrate or oxalic acid dihydrate (5.2.2.3) in a weighing scoop to the nearest 0,1 mg. Transfer the tartrate or oxalic acid to the titration vessel and reweigh the weighing scoop. Dissolve the tartrate or oxalic acid in the solvent by gentle stirring.

Or:

Introduce approximately 40 mg of water (5.2.2.1) from a dropping bottle, weighed before and after introducing the water into the titration vessel.

The amount of solvent used shall be such as to immerse the electrode by 2 mm to 3 mm.

Add Karl Fischer reagent until the electrometric end point has been reached, that is, is when, after adding Karl Fischer reagent drop by drop, the observed value remains constant for 30 s.

The water equivalent, ρ_{H_2O}, expressed in milligrams of water per millilitre of reagent, is given by one of the following equations:

$$\rho_{H_2O} = \frac{m_1 \times w_{H_2O}}{100 V_1}$$

or

$$\rho_{H_2O} = \frac{m_2}{V_1}$$

where

m_1 is the mass, in milligrams, of disodium tartrate dihydrate or oxalic acid dihydrate (5.2.2.3) used;

m_2 is the mass, in milligrams, of water (5.2.2.1) used;

w_{H_2O} is the water content, expressed as a percentage by mass, of the primary standard (5.2.2.3) used, i.e. 15,66 for sodium tartrate dihydrate or 28,57 for oxalic acid dihydrate;

V_1 is the volume, in millilitres, of Karl Fischer reagent (5.2.2.2) used in the titration.

5.2.5.3 Determination of water content

5.2.5.3.1 General

Depending on the characteristics of the laboratory sample, one of the following two methods shall be chosen: the general method (5.2.5.3.2) or the dissolution method (5.2.5.3.3).

The general method is suitable for samples which are homogeneous and easy to dispense into the titration vessel.

The dissolution method shall be used for samples for which homogeneity is questionable and/or for samples that are difficult to dispense into the titration vessel.

5.2.5.3.2 General method

5.2.5.3.2.1 Determination

Place 20 ml of solvent (5.2.2.4) and the magnetic stirring bar (using pincers) in the titration vessel of the Karl Fischer apparatus (5.2.3.1). Whilst stirring, add Karl Fischer reagent (5.2.2.2) until the end point is reached as indicated by the instrument reading. Do not record the amount of reagent used.

Add the test portion. Check the amount to the nearest 0,1 mg by difference of masses, m_0. Choose the amount so as to consume a volume of Karl Fischer reagent corresponding to more than 50 % of the capacity of the piston burette. Stir to dissolve and titrate to the same end point. Record the volume of Karl Fischer reagent used, V_2.

Carry out a second determination by adding another test portion and repeating the titration.

5.2.5.3.2.2 Expression of results

The water content of the sample, w_S, expressed as a percentage by mass, is given by the formula:

$$w_S = \frac{\rho_{H_2O} \times V_2}{m_0} \times 100$$

where

V_2 is the volume, in millilitres, of Karl Fischer reagent (5.2.2.2) used for the determination;

m_0 is the mass, in milligrams, of the test portion;

ρ_{H_2O} is as defined in 5.2.5.2.

5.2.5.3.3 Dissolution method

5.2.5.3.3.1 Determination

Weigh a suitable amount of laboratory sample, m_1, into a dry flask. Add a suitable amount of any organic solvent, m_2, that does not interfere with Karl Fischer reagent (5.2.2.2). The amounts of sample and solvent shall be chosen so as to use the greatest amount of sample and smallest proportion of the solvent possible, in order to obtain a manageable blend and not compromise the representative of the sample uptake. Record the mass to the nearest 0,001 g. Stopper the flask and shake until a homogeneous blend is obtained.

If insoluble salts remain at the bottom of the flask at this stage, the method has to be abandoned because

a) part of the water might have been removed by the insoluble salts as crystallization water, and

b) a rigorous procedure would have to involve the correct determination of the mass of the insoluble salts.

Determine the water content of both the solvent, w_2, and the blend, w_b, following the procedure given in 5.2.5.3.2.

5.2.5.3.3.2 Expression of results

The water content of the product, w_P, expressed as a percentage by mass, is given by the formula:

$$w_P = \frac{w_b (m_1 + m_2) - m_2 \times w_2}{m_1}$$

where

m_1 is the mass, in grams, of sample taken in 5.2.5.3.3.1;

m_2 is the mass, in grams, of solvent used in 5.2.5.3.3.1;

w_b is the water content in the blend according to 5.2.5.3.3.1, as a percentage (mass fraction);

w_2 is the water content in the solvent according to 5.2.5.3.3.1, as a percentage (mass fraction).

5.3 Coulometric titration method

5.3.1 Principle

Place the electrolytic solution and sample in an electrolysis cell, electrolyse it by flowing electrolytic current, carry out the Karl Fischer reaction between the iodine produced and the water in the solution, and find the water content, making use of the quantity of electricity which is required to get an end point at titration. Detect the end point by a voltage-detecting method under current control.

IMPORTANT — The coulometric titration method shall not be applied, for example, if the sample containing aniline or phenol would cause an electrode reaction by itself; or if the sample could cause a blockage of the diaphragm due to an impurity or could influence the current effiiciency. Especially in the case of a solid sample, confirm whether it is completely dissolved or not.

5.3.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only grade 2 distilled water (5.2.2.1). Anolyte, catholyte and water-alcohol solution or water-propylene carbonate solution found in the market may be used.

5.3.2.1 Anode electrolyte solution (anolyte). The anolyte shall be the mixture of, for example, iodine ion, sulfur dioxide, pyridine, or an alternative base, and organic solvent as methanol.

For samples containing compounds such as aldehydes or ketones, which may react with methanol, use chloroform, 2-methoxyethanol, propylene carbonate, or the like instead of methanol.

5.3.2.2 Cathode electrolyte solution (catholyte). The catholyte shall be prepared by mixing inorganic salts and organic salts such as quartenary ammonium salts or amine hydrochloride with an organic solvent such as methanol or the like.

5.3.2.3 Water–alcohol solution or **water–propylene carbonate solution**. Add water to 2-methoxyethanol or to propylene carbonate so that 1 ml of the solution contains about 4 mg of water. This solution may be used to add small amounts of water.

5.3.3 Apparatus

Use usual laboratory apparatus and, in particular, the following.

5.3.3.1 Automatic titration device, composed of a titration part, a controlling part, and an indicating and recording part. An example of the construction is shown in Figure 1.

5.3.3.2 Electrolytic cell, consisting of a glass vessel with a capacity of about 200 ml and composed of an anode room and cathode room, each equipped with platinum-net electrode, and separated by a diaphragm (e.g. ceramic diaphragm, ion-exchange membrane, or similar).

When a microsyringe or injector is used for sampling, the charging aperture for the sample shall be equipped with a stopper which is made of stainless steel or tetrafluoroethylene resin with a packing. The electrolytic cell shall be equipped with a detector and drying tube containing desiccant such as silica gel. An example of the cell is shown in Figure 2. A rotor should be put in the cell when titration is carried out.

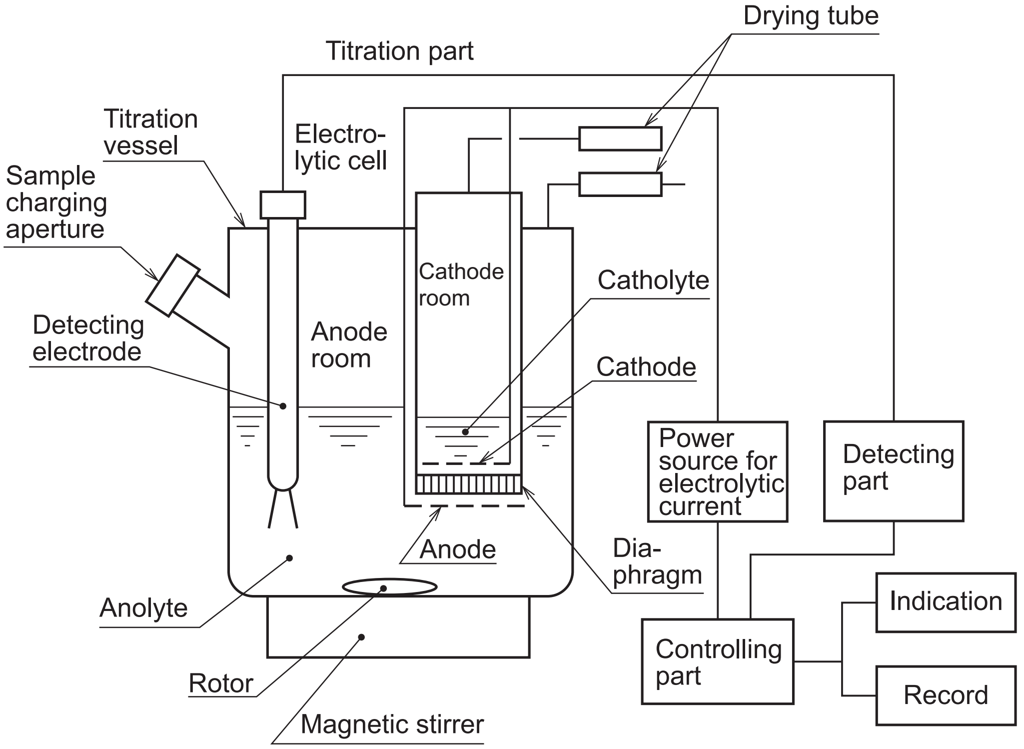
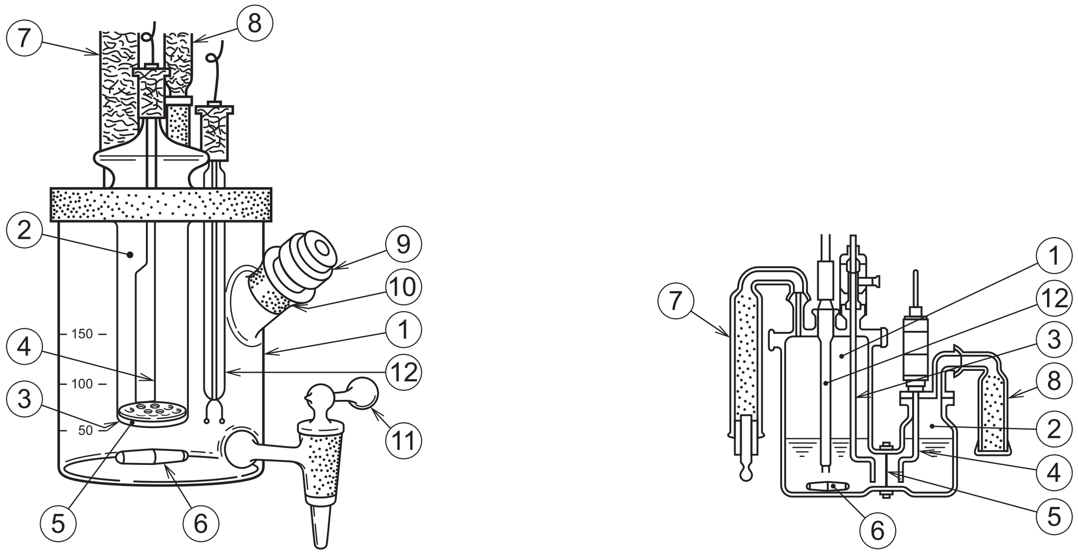


Figure 1 — Example of coulometric titration apparatus



a) Interior cathode-room type

b) Exterior cathode-room type

Key

1 anode room
2 cathode room
3 anode
4 cathode
5 diaphragm
6 rotor
7 drying tube for anode room
8 drying tube for cathode room
9 stopper
10 sample charging aperture
11 discharging cock
12 detector

Figure 2 — Example of electrolytic cell

5.3.3.3 **Injector**, with a capacity of 1 ml to 20 ml, the outside diameter of the needle measuring 1 mm to 2 mm and the length of the needle 50 mm to 100 mm. A rubber plug shall be fixed at the end of the needle for sealing; a round-type silicone rubber, 13 mm to 15 mm in diameter and 5 mm to 7 mm in thickness, may be used for this purpose.

5.3.3.4 **Balance**, accurate to 0,1 mg. An electrobalance may conveniently be used if it is connected to an automatic titration device through a suitable interface.

5.3.4 Sampling

The laboratory sample of surface active agent or detergent shall have been prepared and stored in accordance with the instructions given in ISO 607.

5.3.5 Procedure

5.3.5.1 Calibration of titration device

Make the inside of an electrolytic cell anhydrous by coulometric titration until an end point is reached while stirring anolyte in the electrolytic cell. Inject 5 µl of water into the anolyte using a microsyringe and carry out coulometric titration until an end point is reached in order to find water content. Repeat this procedure two times or more, and confirm that the water content found by this procedure falls within the range of 5 000 µg ± 250 µg. If it is out of this range, after renewing the electrolytic solution or washing an electrode or diaphragm, carry out a calibration.

5.3.5.2 Determination of water content

Place about 100 ml of anolyte into the anode room of an electrolytic cell and about 5 ml of catholyte in the cathode room. Closely seal the electrolytic cell and mount it on the titration apparatus after it has been set up.

While stirring anolyte in the electrolytic cell, coulometrically titrate it to get an end point, and make the inside anhydrous. The volume of titrant in this case does not need to be read.

The existence of free iodine in the anolyte prevents the electrolytic current from flowing, so it is preferable to add water, water-alcohol solution or water-propylene carbonate solution to obtain an excess of water of 2 000 µg to 3 000 µg.

Wash the injector (5.3.3.3) before use, dry it and keep it in a desiccator.

Take the injector out of the desiccator and attach a rubber plug to the end of the injector needle.

Remove the plug, stick the injector needle as deeply as possible into the sample (which preferably contains 0,5 mg to 5 mg of water) in the sample container, gently draw the plunger, and take the sample up by about 1/10 of the volume of the injector.

Take the injector out of the sample container, invert the injector so that the needle is pointing upwards to remove air, attach a rubber plug to the tip of the needle, slightly apply pressure in order to let the sample moisten the ground surfaces, then discharge the sample.

Carry out this operation (washing the injector with the sample) two or three times.

Gradually take the sample over to the scale of the injector, discharge excessive sample to adjust it to the scale while orienting the injector upward, and simultaneously expel the air kept inside. Immediately attach the rubber plug to the tip of the needle. Weigh the mass of the injector with sample to the nearest 0,1 mg.

Remove the rubber plug attached to the injector and inject the sample through the stopper into the anolyte in the electrolytic cell or after opening the aperture plug. Then, attach the same rubber plug to the tip of the needle, and weigh its mass to the nearest 0,1 mg.

Stir the anolyte sufficiently to dissolve the sample. While stirring the anolyte, coulometrically titrate to get an end point, and read the indication of water content.

5.3.5.3 Expression of results

The water content of the product, w_P, expressed as a percentage by mass, is given by the formula:

$$w_P = \frac{m_{in} \times 100}{m_S \times 10^6}$$

where

m_{in} the indicated value of water content, in micrograms;

m_S the mass of the sample, in grams.

6 Precision

6.1 Precision of the volumetric titration method

6.1.1 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will not exceed the repeatability limit, r, in more than 5 % of cases.

According to the ring test results given in Annex B, the repeatability limit, r, shall be

- less than or equal to 0,013 g/100 g for a water content of approximately 0,24 % (mass fraction), or
- less than or equal to 1,74 g/100 g for a water content of approximately 28,8 % (mass fraction).

6.1.2 Reproducibility

The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will not exceed the reproducibility limit, R, in more than 5 % of cases.

According to the ring test results given in Annex B, the reproducibility limit, R, shall be

- less than or equal to 0,026 g/100 g for a water content of approximately 0,24 % (mass fraction), or
- less than or equal to 3,79 g/100 g for a water content of approximately 28,8 % (mass fraction).

6.2 Precision of the coulometric titration method

6.2.1 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will not exceed the repeatability limit, r, in more than 5 % of cases.

According to the ring test results given in Annex B, the repeatability limit, r, shall be

- less than or equal to 0,007 g/100 g for a water content of approximately 0,04 % (mass fraction), or
- less than or equal to 0,016 g/100 g for a water content of approximately 0,23 % (mass fraction).

6.2.2 Reproducibility

The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories by different operators using different equipment, will not exceed the reproducibility limit, R, in more than 5 % of cases.

According to the ring test results given in Annex B, the reproducibility limit, R, shall be

- less than or equal to 0,016 g/100 g for a water content of approximately 0,04 % (mass fraction), or
- less than or equal to 0,058 g/100 g for a water content of approximately 0,23 % (mass fraction).

7 Test report

The test report shall include the following:

a) all information necessary for the complete identification of the sample;

b) a reference to this International Standard and to the type of solvent and Karl Fischer reagent used;

c) the results obtained and the units in which they have been expressed;

d) the test conditions;

e) any operational details not specified in this International Standard or in the International Standards to which reference is made, as well as any operational details regarded as optional, plus details of any incidents likely to have affected the results.

Annex A
(informative)

Preparation of Karl Fischer reagent containing pyridine

A.1 Place 670 ml of methanol or 2-methoxyethanol (5.2.2.4) in a dry brown-glass flask, of capacity 1 l, fitted with a ground-glass stopper.

A.2 Add about 85 g of iodine. Stopper the flask and shake it occasionally until the iodine is completely dissolved. Then add approximately 270 ml of pyridine containing not more than 500 mg of water per kilogram, stopper the flask again and mix thoroughly. As the reaction will be exothermic, maintain the flask at about 0 °C, for example by immersing it in an ice bath. Using the method described below, dissolve 65 g of sulfur dioxide in this solution, ensuring that the temperature of the liquid does not exceed 20 °C.

A.3 Replace the ground-glass stopper by an attachment for introducing sulfur dioxide (consisting of a cork with a thermometer and a glass inlet tube passing through it, thermometer and inlet tube reaching to within 10 mm of the bottom of the flask) and a small capillary tube for connecting to the atmosphere.

A.4 Place the whole assembly with the cooling bath on a balance and weigh to the nearest 1 g. Then connect the inlet tube to a cylinder of sulfur dioxide by means of a flexible connection and a drying tube filled with drying agent and gently open the tap on the cylinder.

A.5 Adjust the rate of flow of sulfur dioxide so that all the gas is absorbed but the liquid does not rise up the inlet tube.

A.6 Then maintain the balance in equilibrium by gradually increasing the tare and ensure that the temperature of the liquid does not rise above 20 °C. Close the tap on the cylinder as soon as the increase in mass reaches 65 g.

A.7 Immediately remove the flexible connection and reweigh the flask with the ice bath and inlet tube. The mass of dissolved sulfur dioxide should be between 60 g (minimum) and 70 g. A slight excess is not serious.

A.8 Stopper the flask, mix the solution and leave for at least 24 h before using it (as the result of imperfectly understood reactions which occur in the fresh reagent, the water equivalent of the reagent decreases rapidly to begin with and then much more slowly).

A.9 This water equivalent shall be between 3,5 mg/ml and 4,5 mg/ml. It shall be determined daily if methanol has been used, but may be determined less frequently if 2-methoxyethanol has been used.

A.10 It is possible to prepare Karl Fischer reagent that has a lower water content by diluting the solution prepared as described above with dry methanol.

A.11 Store the reagent in the dark and protect from atmospheric moisture. It should preferably be stored in the reagent bottle of the Karl Fischer apparatus (5.2.3.1).

Annex B
(informative)

Interlaboratory test results

B.1 General

The statistical data presented in Tables B.1 to B.4 were obtained by a ring test carried out by JISC/TC 91 WG “Analysis” in 2010.

The evaluation was performed in accordance with ISO 5725-2.

B.2 Volumetric titration method

Table B.1 — Samples and methods used

Sample	Description	Method used
A	Oleyl bis(2-hydroxyethyl) methylammonium chloride	General (5.2.5.3.2)
B	Sodium polyoxyethylene (2EO) lauryl ether sulfate	Dissolution (5.2.5.3.3)

Table B.2 — Precision data

Designation	Sample A	Sample B
Number of laboratories participating	9	9
Number of laboratories not eliminated	9	9
Number of individual values of all laboratories	45	45
Mean value (g/100 g)	0,24	28,8
Repeatability standard deviation, S_r (g/100 g)	0,005	0,62
Repeatability limit, r: $r = (2{,}8 \times S_r)$ (g/100 g)	0,013	1,74
Coefficient of variation repeatability, V_r (%)	1,9	2,2
Reproducibility standard deviation, S_R (g/100 g)	0,009	1,35
Reproducibility limit, R: $R = (2{,}8 \times S_R)$ (g/100 g)	0,026	3,79
Coefficient of variation reproducibility, V_R (%)	3,8	4,7

B.3 Coulometric titration method

Table B.3 — Samples and methods used

Sample	Description
C	Polyoxyethylene (2EO) lauryl ether
D	Oleyl bis(2-hydroxyethyl) methylammonium chloride

Table B.4 — Precision data

Designation	Sample C	Sample D
Number of laboratories participating	10	10
Number of laboratories not eliminated	10	10
Number of individual values of all laboratories	50	50
Mean value (g/100 g)	0,041	0,23
Repeatability standard deviation, S_r (g/100 g)	0,002 6	0,006
Repeatability limit, r: $r = (2{,}8 \times S_r)$ (g/100g)	0,007 3	0,016
Coefficient of variation repeatability, V_r (%)	6,3	2,5
Reproducibility standard deviation, S_R (g/100 g)	0,005 8	0,021
Reproducibility limit, R: $R = (2{,}8 \times S_R)$ (g/100 g)	0,016 3	0,058
Coefficient of variation reproducibility, V_R (%)	14,0	9,1

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[1] JIS K 0068, *Test methods for water content of chemical products*

[2] ISO 760, *Determination of water — Karl Fischer method (General method)*

[3] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[4] EN 13267:2001, *Surface active agents — Determination of water content — Karl Fischer method*

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