IS 2362 : 1993

(Reaffirmed 1998)

भारतीय मानक

कार्ल फिशर पद्धति द्वारा जल ज्ञात करने की परीक्षण पद्धति

(दूसरा पुनरीक्षण)

Indian Standard

DETERMINATION OF WATER BY KARL FISCHER METHOD — TEST METHOD

(Second Revision)

UDC 543·812

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 4

Chemical Standards Sectional Committee, CHD 001

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

This standard was originally published in 1963 and revised in 1973. The present revision has been undertaken to update the standard incorporating latest analytical techniques developed in this field.

A small percentage of water in certain liquids including volatile liquids can be conveniently measured using Karl Fischer method. This method is increasingly used in various chemical standards prepared by BIS. Therefore, this standard is intended to assist the various technical committees of BIS preparing chemical standards in avoiding unnecessary variations in the details of the method.

In this revision, the method involving visual detection of end point has been deleted in view of the problems encountered during the end point detection of colour change. Wide range of electrometric Karl Fischer titrators and Karl Fischer reagents including non-pyridene based Karl Fischer reagents are now available and therefore, reference to non-pyridine based Karl Fischer reagents is given.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

The Committee responsible for the preparation of the standard is given in Annex B.

Indian Standard
DETERMINATION OF WATER BY KARL FISCHER METHOD — TEST METHOD
(*Second Revision*)

1 SCOPE

1.1 This Indian Standard describes the Karl Fischer method for determination of free water or water of crystallization or moisture in solid or liquid chemical products, both organic and inorganic.

1.1.1 The method is not applicable to all the organic and inorganic chemical products. The limitations are:

a) oxidizing agents, such as chromates, dichromates, cupric and ferric salts, and higher oxides and peroxides;
b) reducing agents, such as sulphides, thiosulphates and stannous salts;
c) compounds which may form water with components of Karl Fischer reagent, namely, basic zinc oxide, salts of weak oxyacids, alkali carbonates and bicarbonates, and borates; and
d) organic compounds, such as quinines, ascorbic acid, peroxy compounds, active carbonyl compounds, nitrogen compounds containing amino or hydrozo nitrogen, and sulphur compounds where sulphur is not bonded with oxygen. In some cases the method can be employed with minor modification before operating the final titration.

1.1.2 This standard describes electrometric method involving direct titration and the back titration.

NOTE — Certain reagents used, may cause health hazard and therefore they should be handled carefully.

2 REFERENCE

IS 1070 : 1992 Specification for reagent grade water (*third revision*) is a necessary adjunct to this standard.

3 PRINCIPLE OF THE METHOD

The determination of water is based on the reaction of water while oxidizing R-sulphite anion to R-sulphate by iodine. The reaction of sulphur dioxide with alcohol (methanol) producing a mono-alkyl ester of the sulphurous acid is a basic requirement for the Karl Fischer reaction. The mono-alkyl ester of sulphurous acid in turn reacts with water in presence of iodine and amine to form stable salts as per following equations :

$$ROH + SO_2 + Rn \longrightarrow (Rn\,H^+)\,SO_3R^-$$

$$H_2O + I_2 + (RnH)SO_3R + 2Rn \longrightarrow (RnH)SO_4R + 2\,(RnH)I$$

(Rn = Amine and ROH = alcohol).

Methanol used above may be replaced by isopropyl alcohol or 2-methoxy-ethanol (ethylene glycol monomethyl ether). While using 2-methoxy-ethanol as solvent the more constant titration volumes can be obtained and the reagent is suitable for aldehydes and ketones.

NOTE — According to second equation one molecule of iodine should be equivalent to one molecule of water, in practice this stoiciometric ratio is not attained and the Karl Fischer reagent has to be standardized against a known mass of water.

4 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

5 REAGENTS

5.1 Methanol

Shall not contain more than 0.05 percent of water. If the reagent contains more than this quantity of water, dry it by distillation from magnesium turnings activated with iodine. Collect the distillate in a bottle protected from atmospheric moisture by means of a guard tube filled with anhydrous aluminium sodium silicate or activated silica gel (**5.11**).

5.2 2-Methoxyethanol (Ethylene Glycol Monomethyl Ether)

Shall not contain more than 0.05 percent of water. If the reagent contains more than this quantity of water, dry it by distillation, rejecting the first portion of distillate which will contain water. It is preferable to methanol due to its stabilizing effect on the final reagent.

5.3 Iodine

5.4 Pyridine

Shall not contain more than 0.05 percent of water. If the reagent contains more than this quantity of water, dry it by distillation, rejecting the first portion of distillate which will contain the water.

5.5 Sample Solvent

Either methanol or a mixture containing 4 parts of methanol and 1 part of pyridine (by volume) or (preferably for determination with compounds containing carbonyl groups) a mixture containing 4 parts of 2-methoxyethanol and 1 part of pyridine (by volume). In special cases, other solvents may be recommended, for example, acetic acid, pyridine or a mixture containing 1 part of methanol and 3 parts of chloroform (by volume).

5.6 Sulphur Dioxide

5.7 Karl Fischer Reagent

Place 670 ml of methanol or 2-methoxyethanol in a previously dried flask, coloured brown or painted black on the outside, fitted with a ground-glass stopper and having a capacity slightly more than 1 litre. 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, stopper the flask again and mix thoroughly. Dissolve 65 g of sulphur dioxide in this solution, cool to ensure that the temperature of the liquid does not exceed 20°C.

NOTE — The reaction being exothermic, it is necessary to cool the flask from the beginning and to maintain it at about 0°C, for example, by immersing in an ice-bath or in crushed solid carbon dioxide.

Replace the ground-glass stopper by an attachment for introducing sulphur dioxide. This consists of a cork with a thermometer and an inlet glass tube of 6 to 8 mm, reaching to within 10 mm of the bottom of the flask, and a small capillary tube for connecting to the atmosphere. Place the whole assembly with the ice-bath on a balance and weigh to the nearest 1 g. Connect the inlet tube to a siphon of sulphur dioxide by means of a flexible connection and drying tube filled with anhydrous aluminium sodium silicate as desiccant and gently open the tap on the siphon.

Adjust the rate of flow of sulphur dioxide so that all the gas is absorbed without the liquid showing any sign of rising in the inlet tube. Maintain the equilibrium of the balance by gradually increasing the rate and ensure that the temperature of the liquid does not rise above 20°C. Close the tap on the siphon as soon as the increase in mass reaches 65 g.

Immediately remove the flexible connection and reweigh the flask and its inlet attachment. The mass of dissolved sulphur dioxide shall be between 60 to 70 g. A slight excess is not harmful. Stopper the flask, mix the solution and leave for at least 24 hours before using it. In fact, as a 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 slowly. This water equivalent is between 3.5 and 4.5 mg/ml. It shall be determined daily if methanol has been used, but may be done less frequently if 2-methoxyethanol has been used.

It is possible to prepare the Karl Fischer reagent with a lower water content by diluting the solution prepared as described above with the sample solvent. Store the reagent out of the light and protected from atmospheric moisture. It should preferably be stored in a reagent bottle of brown or black-painted glass.

NOTE — Non-pyridine based Karl Fischer reagent

Despite the long, successful use of pyridine based Karl Fischer reagent for determination of water, its constant use has been questioned because of the toxicity of pyridine, especially irritation of the respiratory tract, its disagreeable odour and reagent instability, the latter largely overcome by replacing methanol by 2-methoxy ethanol (methyl cellosolve). Non-pyridine based Karl Fischer reagents have, therefore, been developed recently. Amongst the alternatives, those based on aliphatic amines have successfully been used and results obtained are quite comparable with the conventional Karl Fischer reagent. The alternative non-pyridine based Karl Fischer reagents are available indigenously from reputed laboratory reagent manufacturers. These reagents may be used after standardization.

5.8 Sodium Tartarate, Crystalline ($Na_2C_4H_4O_6.2H_2O$)

Sodium tartarate shall be of such quality that when dried at 150°C for 3 hours, it gives out 15.66 ± 0.5 percent water.

5.9 Water-Methanol Standard Solution — 10 mg/ml

Using a microburette or a pipette, place 1 ml of water in a dry 100-ml one-mark volumetric flask, containing approximately 50 ml of methanol. Dilute to the mark with methanol and mix. For standardization of this solution (*see* **A-1.1**).

5.10 Water-Methanol Solution — Approximately 2 g/l

Using a microburette or a pipette, place 1 ml of water into a perfectly dry 500-ml one-mark, volumetric flask containing approximately 100 ml of methanol. Dilute to the mark with methanol and mix (*see* the correspondence by volume of this solution with the Karl Fischer reagent in **8.2.3**).

5.11 Aluminium Sodium Silicate/Activated Silica Gel

Aluminium sodium silicate anhydrous, granules of diameter 1.7 mm for use as a desiccant. These granules may be regenerated by washing with water and drying at 350°C for at least 48 hours. Alternatively, activated silica gel, indicating type, may be used.

5.12 Silicone Base Grease

For lubricating the ground-glass joints.

6 APPARATUS

All glasswares used, should be previously dried in oven and cooled in desiccator. While using such glasswares for estimation of moisture content, care should be taken to protect them from absorbing moisture from the surrounding atmosphere.

Any commercial Karl Fischer titrator with electrometric end point detection will be suitable. The instrument should incorporate the following:

a) Automatic Burette — 10 to 25 ml capacity with a fine pointed tip and graduations of 0.05 ml (*see* Fig. 1). For back titration method a two burette system (*see* Fig. 2) would be required. In both the cases the system should be protected from atmospheric moisture by guard tube filled with a desiccant.

b) Titration Vessel — 100 ml capacity having provision for inserting burette tip (two burette tips in case of back titration method) platinum electrodes and also for the introduction of liquid samples (suitable ground glass joint stopper) with the help of syringe or pipette, without removing the vessel from apparatus. A similar arrangement for introduction of solid samples with least exposure is preferred.

c) Reagent bottle for Karl Fischer Reagent — Amber coloured connected to automatic burette through ground glass joint.

d) Double platinum electrode.

e) Magnetic stirrer with PTFE coated stirring bar.

f) Electrometric end point detection device utilizing a micrometer.

g) Glass syringe — suitable capacity.

h) A small glass tube — closed at one end and fitted at the other with a rubber stopper, used for weighing and introducing into the titration vessel for example, the mass of crystalline sodium tartarate (approximately 200 mg) used to standardize the Karl Fischer reagent or possibly test samples of solid products.

7 DIRECT ELECTROMETRIC TITRATION

7.1 Outline of the Method

Indication of the end point of titration by the depolarization of the cathode accompanied by a sudden increase in current intensity (which is shown by a suitable electrical device), the two platinum electrodes being immersed in the solution and subjected to a potential difference, but while water is present in the solution polarization of the cathode opposes the passage of a current.

7.2 Standardization of Karl Fischer Reagent

7.2.1 Assemble the apparatus as recommended in Fig. 1 lubricating the joints with grease. Introduce by

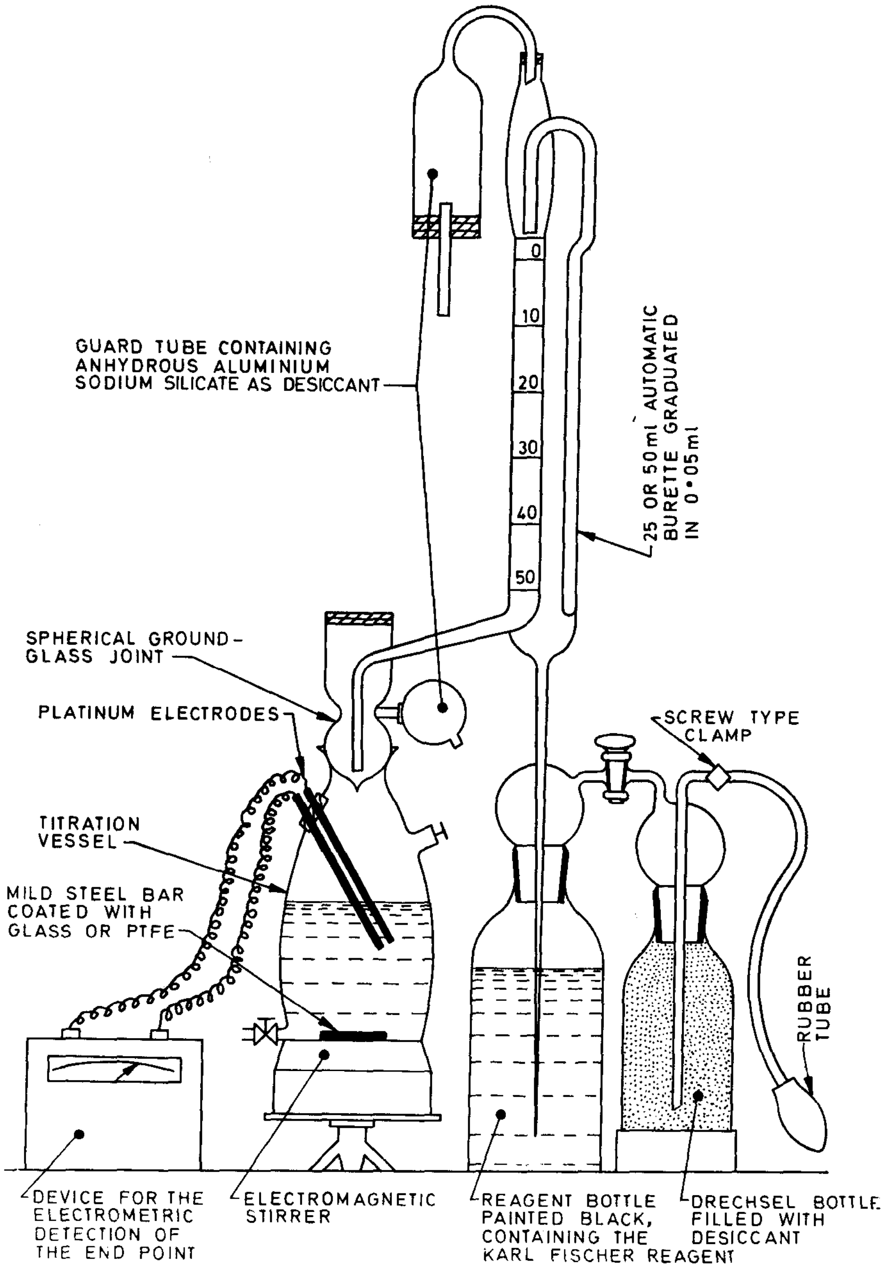

FIG. 1 APPARATUS FOR DIRECT ELECTROMETRIC TITRATION

means of a syringe 25 ml of methanol into the titration vessel through the ground glass stopper. Switch on the electromagnetic stirrer and close the circuit of the device for the electrometric detection of the end point.

Adjust the apparatus so that a voltage of 1 to 2 V is applied to the electrodes and the galvanometer shows a low current, usually a few microamperes. Add the Karl Fischer reagent until the galvanometer shows a sudden increase in current of about 10 to 20 µA, which remains stable for at least 30 seconds.

7.2.2 In the small glass tube [*see* **6** (h)] weigh 250 mg of crystalline sodium tartarate to the nearest 0.1 mg. Place this in the titration vessel very quickly, removing the ground glass stopper for a few seconds. Weigh the small glass tube empty, so as to determine, by difference, the mass of crystalline sodium tartarate used. (For standardization with water-methanol standard solution, *see* **A-1.1**)

Titrate the known quantity of water introduced in this way with the Karl Fischer reagent to be standardized, until the same deflection to the pointer of the galvanometer is reached and remains stable for at least 1 minute. Note the volume of the reagent used.

7.2.3 *Calculation*

$$\text{Water equivalent (T) of the Karl Fischer reagent (mg } H_2O\text{/ml)} = \frac{M_1}{A} \text{ or } \frac{M_2}{A}$$

where

M_1 = mass in mg of water used (*see* **7.2.2**),

A = volume in ml of Karl Fischer reagent used, and

M_2 = mass in mg of sodium tartarate introduced multiplied by 0.156 6.

7.3 Procedure

7.3.1 Empty the titration vessel by mean of the emptying tap. Place in it 25 ml of methanol or other solvent, or any other solvent, or any other suitable volume indicated in the procedure for the products to be analysed, using a syringe passing through the ground glass stopper. Switch on the electromagnetic stirrer. Add Karl Fischer reagent, proceeding as described in **7.2** until there is a sudden and constant deflection lasting for at least 30 seconds. Then introduce the required amount of test portion taken by means of a syringe in the case of a liquid or weighed to the nearest 0.1 mg in a small weighing tube [*see* **6** (h)] in the case of a solid powder. Titrate with Karl Fischer reagent using the same electrometric procedure for detecting the end point of the reaction. Note the volume of Karl Fischer reagent for the determination.

NOTE — It is advisable to use a quantity of test portion the water content of which corresponds to a volume of Karl Fischer reagent that can be measured with sufficient accuracy. If necessary, increase in proportion the quantities of solvent and test sample used and then use a titration vessel of suitable capacity.

7.3.2 *Calculation*

$$\text{Water content of the sample } (H_2O)\text{, percent } (m/m) = \frac{B \times T}{E \times 10} \text{ or } \frac{B \times T}{V \times d \times 10}$$

where

B = volume in ml of Karl Fischer reagent used for the test,

T = water equivalent in mg/ml of the Karl Fischer reagent (*see* **7.2.3**),

E = mass in g of the test portion (for solid products),

V = volume in ml of the test portion (for liquid products), and

d = density of the sample in g/ml at measurement temperature (for liquid products only).

8 ELECTROMETRIC BACK-TITRATION

8.1 Outline of the Method

Addition of an excess of Karl Fischer reagent which is then back-titrated with a water-methanol standard solution. Indication of the end point of the titration by the polarization of the cathode accompanied by the sudden interruption of the current (which is shown by a suitable electrical device) the electrodes being subjected to a very slight potential difference but sufficient to cause a large deflection of the galvanometer pointer at the start of the back-titration.

8.2 Standardization of Karl Fischer Reagent

8.2.1 Assemble the apparatus as recommended in Fig. 2 lubricating the joints with grease. Place in the titration vessel sufficient quantity of Karl Fischer reagent from one of the automatic burettes to cover the electrodes. Switch on the electromagnetic stirrer and the circuit of the device for the electrical detection of the end point. Allow the water-methanol standard solution to flow from the second automatic burette [*see* **6** (a)] until the pointer of the galvanometer moves suddenly to zero.

8.2.2 In the small glass tube [*see* **6** (h)] weigh approximately 250 mg of crystalline sodium tartarate to the nearest 0.1 mg. Place this in the titration vessel very quickly, removing the ground glass stopper for a few seconds. Weigh the small glass tube empty so as to determine by difference the mass of crystalline sodium tartarate used.

8.2.3 *Correspondence Between the Karl Fischer Reagent and the Standard Solution of Water in Methanol*

Partially empty the titration vessel leaving the electrodes submerged in the liquid neutralized as described

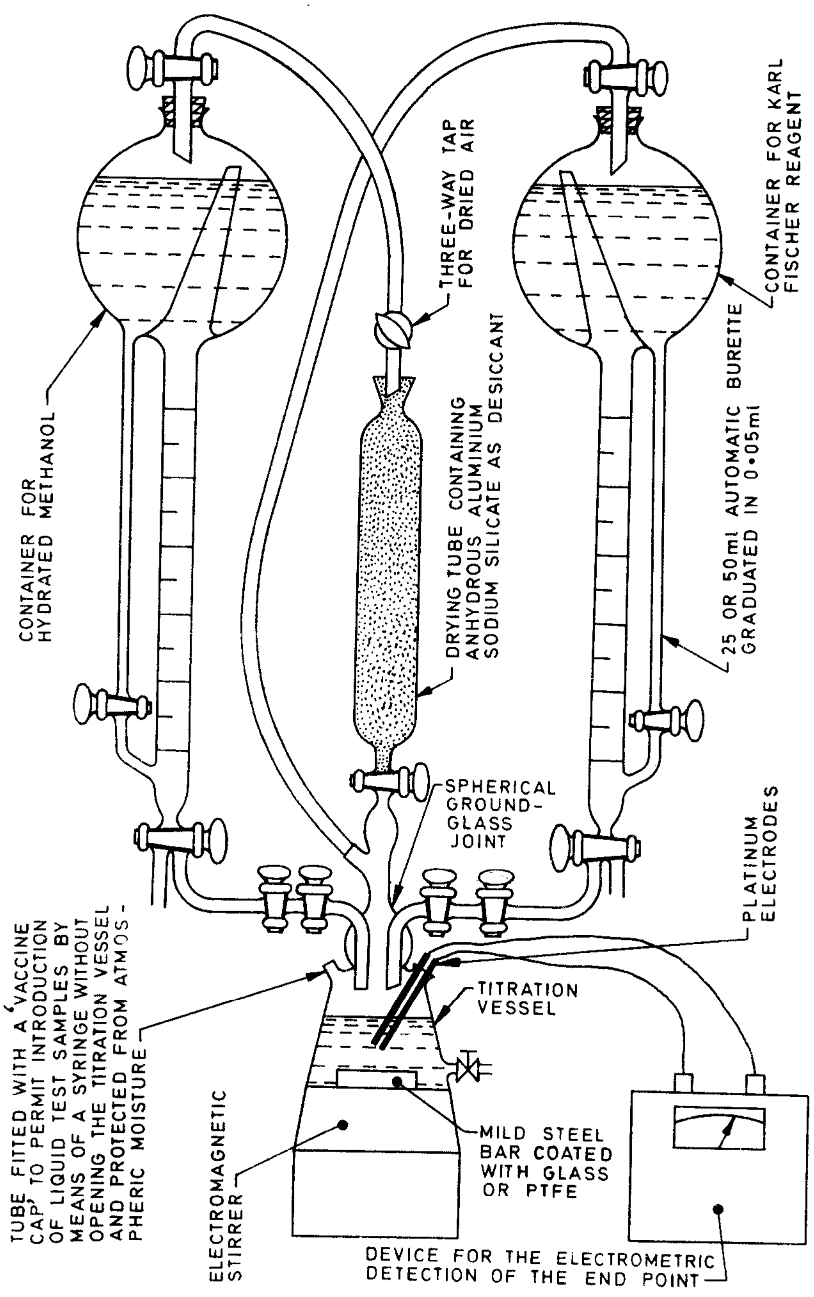

FIG. 2 APPARATUS FOR ELECTROMETRIC BACK-TITRATION

in **8.2.1**. Add 20 ml of Karl Fischer reagent, measured in the first automatic burette and titrate with the standard water-methanol solution contained in the second automatic burette until the pointer of the galvanometer moves suddenly to zero. Note the volume of this solution used.

8.2.4 *Calculation*

$$\text{Water equivalent } (T) \text{ of the Karl Fischer reagent (mg } H_2O\text{/ml)} = \frac{M_1}{A - \frac{A_1 \times 20}{A_2}} \text{ or } \frac{M_2}{A - \frac{A_1 \times 20}{A_2}}$$

where

M_1 = mass in mg of water used in **8.2.2**,

A = volume in ml of Karl Fischer reagent (used in **8.2.1**),

A_1 = volume in ml of water-methanol standard solution used in **8.2.1** for the back-titration,

A_2 = volume in ml of water-methanol standard solution used in **8.2.3** (correspondence with the Karl Fischer reagent), and

M_2 = mass in mg of crystalline sodium tartrate multiplied by 0.156 6.

8.3 Procedure

8.3.1 Empty the titration vessel by means of the emptying cap. Place in it 25 ml of methanol or any other appropriate volume indicated in the procedure for the product to be analysed, using a syringe passing through the ground glass stopper. Switch on electromagnetic stirrer.

8.3.2 Add a slight excess (approximately 2 ml of Karl Fischer reagent) and then add water-methanol standard solution until the pointer of the galvanometer moves suddenly to zero. Introduce the required amount of test portion by means of a syringe in the case of a liquid or weighed to the nearest 0.1 mg in a small glass tube [*see* **6.1** (h)] in the case of a solid powder.

8.3.3 Add a known excess volume of Karl Fischer reagent stopping when the solution becomes brown in colour. Wait for 30 seconds and back titrate this excess with the water-methanol standard solution until the pointer of the galvanometer moves suddenly to zero.

NOTE — It is advisable to use a quantity of test portion the water content of which corresponds to a volume of Karl Fischer reagent that can be measured with sufficient accuracy. If necessary, increase in proportion the quantities of solvent and test samples used and then use a titration vessel of suitable capacity.

8.4 Calculation

Water content of the sample introduced (H_2O), percent (*m/m*)

$$= \left(B - B_1 \times \frac{20}{A2}\right) \times \frac{T}{E \times 10} \text{ or } \left(B - B_1 \times \frac{20}{A_2}\right) \times \frac{T}{V \times d \times 10}$$

where

B = volume in ml of Karl Fischer reagent (used in **8.3**),

B_1 = volume in ml of water-methanol standard solution used in **8.3** for the back-titration,

T = water equivalent in mg/ml of the Karl Fischer reagent,

E = mass in g of the test portion (for liquid products),

V = volume in ml of the test portion (for liquid products), and

d = density of the sample at measurement temperature in g/ml (for liquid products only).

ANNEX A

(*Clause* 7.2.2)

STANDARDIZATION OF KARL FISCHER REAGENT WITH WATER-METHANOL STANDARD SOLUTION

A-1 PROCEDURE

A-1.1 If the water-methanol standard solution (*see* **5.10**) is used instead of crystalline sodium tartarate to standardize the Karl Fischer reagent clauses **7.2.1** and **7.2.2** relating to direct electrometric titration shall be amended as follows:

> Using a syringe add 10.0 ml of methanol to the titration vessel, titrate with the Karl Fischer reagent until the same deflection of the pointer of the galvanometer is reached and remains stable for at least 30 minutes. Note the volume of reagent used.

In the same way add 10.0 ml of water-methanol standard solution (**5.10**). Titrate the known quantity of water thus introduced with the Karl Fischer reagent until the same deflection of the pointer of the galvanometer is reached and remains stable for at least 30 minutes. Note the volume of reagent used.

In addition, **7.2.3** shall be amended as follows:

$$\text{Water equivalent } (T) \text{ of the Karl Fischer reagent (mg } H_2O\text{/ml)} = \frac{100}{A - V}$$

where

100 = mass of water in mg used in 10 ml of water-methanol standard solution,

A = volume in ml of Karl Fischer reagent used for the titration of 10 ml of water-methanol standard solution, and

V = volume in ml of Karl Fischer reagent used for the titration of 10 ml of pure methanol.

ANNEX B

(Foreword)

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