INTERNATIONAL STANDARD 760

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Determination of water — Karl Fischer method (General method)

Dosage de l'eau — Méthode de Karl Fischer (Méthode générale)

First edition — 1978-12-01

UDC 661 : (543.71 + 543.81) **Ref. No. ISO 760-1978 (E)**

Descriptors : chemical compounds, chemical analysis, determination of content, water, volumetric analysis, electrometric analysis, Karl Fischer reagent.

Price based on 12 pages

ISO 760-1978 (E)

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; those documents have subsequently been transformed into International Standards. As part of that process, Technical Committee ISO/TC 47, reviewed ISO Recommendation R 760-9168 and found it technically suitable for transformation. International Standard ISO 760-1978 therefore replaced ISO Recommendation R 760-1968.

ISO Recommendation R 760-1968 had been approved by the member bodies of the following countries :

Australia	Germany, F.R.	Poland
Austria	Hungary	Portugal
Belgium	India	Romania
Chile	Israel	Spain
Colombia	Italy	United Kingdom
Czechoslovakia	Japan	U.S.S.R.
Egypt, Arab Rep. of	Korea, Rep. of	Yugoslavia
France	Netherlands	

The member bodies of the following countries had expressed disapproval of the Recommendation on technical grounds :

New Zealand
U.S.A.

The member body of the following country disapproved the transformation of ISO/R 760 into an International Standard :

Netherlands

Printed in Switzerland

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Determination of water — Karl Fischer method (General method)

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a general method known as the Karl Fischer method[1], suitable for the determination of free water or water of crystallization in most solid or liquid chemical products, both organic and inorganic.

Precautions are necessary in certain cases and these are specified in the appropriate International Standards.

Two methods of titration, depending on whether the end-point is detected visually or electrometrically, are specified. The visual method can be used when no electrometric apparatus is available but is applicable only to colourless solutions; it is always a direct titration. The electrometric method, on the other hand, may involve either a direct titration or a back-titration. The electrometric method, whether by direct titration or back-titration, is the more accurate, and for this reason is recommended.

2 PRINCIPLE

Reaction of any water present in a test portion with a solution of iodine and sulphur dioxide in a pyridine/methanol mixture (Karl Fischer reagent), previously standardized by titration with an exactly known mass of water (see 6.1, 7.1 and 8.1).

NOTE — Methanol may be replaced by 2-methoxyethanol (ethylene glycol monomethyl ether). With this solvent, a more constant titration volume is obtained and the reagent can be used with aldehydes and ketones, without using any special technique.[2]

3 REACTIONS[3]

$$H_2O + I_2 + SO_2 + 3\,C_5H_5N \rightarrow 2\,C_5H_5N.HI + C_5H_5N.SO_3$$

$$C_5H_5N.SO_3 + ROH \rightarrow C_5H_5NH.OSO_2\,OR$$

4 REAGENTS AND MATERIALS

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Methanol, not containing more than 0,05 % (*m/m*) 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 receiver protected from atmospheric moisture by means of a guard tube filled with the desiccant (4.9).

4.2 2-Methoxyethanol (Ethylene glycol monomethyl ether), not containing more than 0,05 % (*m/m*) of water. If the reagent contains more than this quantity of water, dry it by distillation, rejecting the first portion of distillate, which contains the water.

4.3 Pyridine, not containing more than 0,05 % (*m/m*) of water. If the reagent contains more than this quantity of water, dry it by distillation, rejecting the first portion of distillate, which contains the water.

4.4 Sample solvent : either a mixture containing 4 parts by volume of the methanol (4.1) and 1 part by volume of the pyridine (4.3), or (preferably for determinations with compounds containing carbonyl groups) a mixture containing 4 parts by volume of the 2-methoxyethanol (4.2) and 1 part by volume of the pyridine (4.3). In special cases, other solvents may be recommended, for example acetic acid, pyridine or a mixture containing 1 part by volume of the methanol (4.1) and 3 parts by volume of chloroform.

4.5 Karl Fischer reagent

Place 670 ml of the methanol (4.1) or the 2-methoxyethanol (4.2) in a dry brown glass flask, fitted with a ground glass stopper and having a capacity slightly greater 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 the pyridine (4.3), stopper the flask again and mix thoroughly. Using the method described below, dissolve 65 g of sulphur dioxide in this solution, cooling to ensure that the temperature of the liquid does not exceed 20 °C.

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

Replace the ground glass stopper by an attachment for introducing sulphur dioxide, consisting of a cork bearing a thermometer and a glass inlet tube 6 mm × 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 cylinder of sulphur dioxide by means of a flexible connection and a drying tube filled with the desiccant (4.9) and gently open the tap on the cylinder.