INTERNATIONAL STANDARD 910

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

Sulphuric acid and oleum for industrial use — Determination of total acidity, and calculation of free sulphur trioxide content of oleum — Titrimetric method

Acide sulfurique et oléums à usage industriel — Détermination de l'acidité totale et calcul de la teneur en trioxyde de soufre libre des oléums — Méthode titrimétrique

First edition — 1977-11-15

UDC 661.25 : 543.241

Ref. No. ISO 910-1977 (E)

Descriptors : sulphuric acid, chemical analysis, determination of content, acidity, volumetric analysis.

Price based on 3 pages

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; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47, *Chemistry*, has reviewed ISO Recommendation R 910-1968 and found it technically suitable for transformation. International Standard ISO 910 therefore replaces ISO Recommendation R 910-1968 to which it is technically identical.

ISO Recommendation R 910 had been approved by the member bodies of the following countries :

Austria	Hungary	Portugal
Belgium	India	Romania
Brazil	Iran	South Africa, Rep. of
Chile	Ireland	Spain
Cuba	Italy	Switzerland
Czechoslovakia	Japan	Thailand
Egypt, Arab Rep. of	Netherlands	Turkey
France	New Zealand	U.S.S.R.
Germany	Poland	Yugoslavia

The member body of the following country had expressed disapproval of the Recommendation on technical grounds :

United Kingdom

The member body of the United Kingdom also disapproved the transformation of the Recommendation into an International Standard.

Printed in Switzerland

Sulphuric acid and oleum for industrial use — Determination of total acidity, and calculation of free sulphur trioxide content of oleum — Titrimetric method

1 SCOPE

This International Standard specifies a titrimetric method for the determination of the total acidity of sulphuric acid for industrial use, conventionally expressed as H_2SO_4, and a method for the calculation of the free sulphur trioxide content of oleum.

2 FIELD OF APPLICATION

Two cases are considered :

a) H_2SO_4 contents equal to or lower than 98 % (*m/m*);

b) H_2SO_4 contents higher than 98 % (*m/m*).

3 PRINCIPLE

Oxidation of a test portion with hydrogen peroxide and titration of the total acidity with a standard volumetric sodium hydroxide solution, in the presence of methyl red as indicator.

4 REAGENTS

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

4.1 Hydrogen peroxide, 60 g/l solution, neutral to methyl red.

4.2 Sodium hydroxide, 1 N standard volumetric solution.

4.3 Methyl red, 1 g/l solution in 95 % (*V/V*) ethanol.

5 APPARATUS

Ordinary laboratory apparatus and

5.1 Flask, capacity approximately 500 ml, with neck of diameter about 30 mm, with ground glass stopper.

5.2 Spherical glass ampoule, of suitable shape and capacity, for example about 20 mm in diameter, having one capillary end of length about 50 mm (see the example shown in the figure).

5.3 Burette, graduated in 0,05 ml, complying with ISO 385.

5.4 Conical flask, capacity 500 ml, with ground glass stopper.

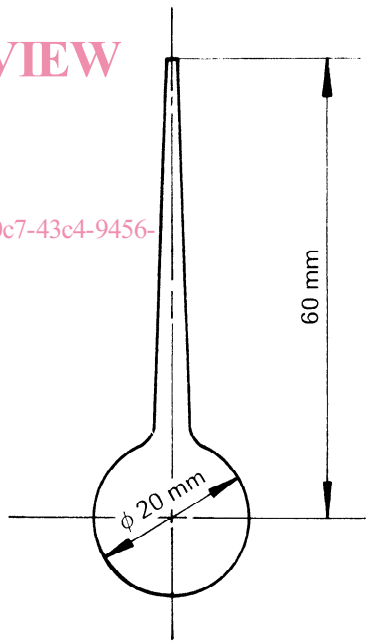

FIGURE — **Spherical glass ampoule**

6 PROCEDURE

6.1 H_2SO_4 contents equal to or lower than 98 % (*m/m*)

6.1.1 *Test portion*

In a weighing bottle, previously tared to the nearest 0,000 1 g, weigh, to the nearest 0,000 1 g, approximately 2 g of the test sample.

6.1.2 *Determination*

Transfer the test portion (6.1.1) quantitatively to a 500 ml conical flask containing approximately 300 ml of water.

Add 5 ml of the hydrogen peroxide solution (4.1), heat to boiling and boil gently for 10 min.

Allow to cool, add 2 drops of the methyl red solution (4.3) and titrate with the sodium hydroxide solution (4.2) until the colour changes from red to yellow.

6.2 H_2SO_4 contents higher than 98 % (*m/m*)

6.2.1 *Test portion*

Carefully mix the test sample by shaking the container. If the acid is partially crystallized, slightly heat the container until the sample is dissolved, then carefully mix again.

Nearly fill the ground glass stoppered flask (5.1) with the test sample. Slightly heat in a flame the bulb of the glass ampoule (5.2), previously weighed to the nearest 0,000 1 g.

Immerse the capillary end of the ampoule into the flask (5.1) containing the test sample and ensure that the bulb is filled to about two-thirds of its volume during cooling (2 to 3 ml approximately).

Withdraw the ampoule and carefully wipe the capillary end with filter paper.

Seal the capillary end in an oxidizing flame, **without loss of glass**. Remove from the flame and allow to cool. Wash the capillary and wipe carefully with filter paper.

Weigh the ampoule to the nearest 0,000 1 g and calculate by difference the mass of the test portion.

6.2.2 *Preparation of test solution*

Carefully place the ampoule containing the test portion (6.2.1) in the conical flask (5.4) containing 300 ml of cold water. Stopper the flask and shake to break the ampoule containing the test portion; cool the flask during this operation.

Keep cooling and shaking the flask until the vapours are completely absorbed.

Remove the stopper and rinse it with water, collecting the washings in the same flask. By means of a glass rod, grind the fragments of the ampoule and in particular the capillary, which may have remained intact in spite of shaking.

Withdraw the glass rod and wash it with water, collecting the washings in the same flask.

Transfer the solution quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

6.2.3 *Determination*

Transfer a 100,0 ml aliquot portion of the test solution (6.2.2) to a 500 ml conical flask.

Add 5 ml of the hydrogen peroxide solution (4.1), heat to boiling and boil gently for 10 min.

Allow to cool, add 2 drops of the methyl red solution (4.3) and titrate with the sodium hydroxide solution (4.2) until the colour changes from red to yellow.

7 EXPRESSION OF RESULTS

7.1 H_2SO_4 contents equal to or lower than 98 % (*m/m*)

The total acidity, expressed as a percentage by mass of sulphuric acid (H_2SO_4), is given by the formula

$$\frac{V \times 0{,}049\ 04 \times 100}{m}$$

$$= \frac{4{,}904\ V}{m}$$

where

V is the volume, in millilitres, of the sodium hydroxide solution (4.2) used for the titration;

m is the mass, in grams, of the test portion (6.1.1);

0,049 04 is the mass, in grams, of sulphuric acid corresponding to 1 ml of exactly 1 N sodium hydroxide solution.

7.2 H_2SO_4 contents higher than 98 % (*m/m*)

7.2.1 *Calculation of total acidity*

The total acidity, A, expressed as a percentage by mass of sulphuric acid (H_2SO_4), is given by the formula

$$A = \frac{V \times 0{,}049\ 04 \times R \times 100}{m}$$

$$= \frac{4{,}904 \times V \times R}{m}$$

where

R is the ratio of the volume of the test solution to the volume of the aliquot portion taken for the determination;

V and m have the same meaning as in 7.1.

NOTE – If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.

7.2.2 *Calculation of free sulphur trioxide content of oleum*

Before calculating the free sulphur trioxide (SO_3) content of oleum, it will be necessary to evaluate

– the total acidity expressed as SO_3 (see 7.2.2.1);

– the water combined as sulphuric acid (see 7.2.2.2);

– the sulphuric acid content (see 7.2.2.3).

7.2.2.1 EVALUATION OF TOTAL ACIDITY EXPRESSED AS A PERCENTAGE BY MASS OF SULPHUR TRIOXIDE (SO_3)

The total acidity, B, expressed as a percentage by mass of sulphur trioxide (SO_3), is given by the formula

$$B = A \times 0{,}8162$$

where

A is the total acidity, expressed as a percentage by mass of sulphuric acid (H_2SO_4) (see 7.2.1);

0,8162 is the conversion factor for H_2SO_4 to SO_3.

7.2.2.2 EVALUATION OF WATER COMBINED AS SULPHURIC ACID (H_2SO_4)

The water combined as sulphuric acid, C, expressed as a percentage by mass, is given by the formula

$$C = 100 - B$$

where B is the total acidity, expressed as a percentage by mass of sulphur trioxide (SO_3) (see 7.2.2.1).

7.2.2.3 EVALUATION OF SULPHURIC ACID (H_2SO_4) CONTENT

The sulphuric acid content, D, expressed as a percentage by mass, is given by the formula

$$D = C \times 5{,}444$$

where

C is the water combined as sulphuric acid, expressed as a percentage by mass (see 7.2.2.2);

5,444 is the conversion factor for H_2O to H_2SO_4.

The free sulphur trioxide (SO_3) content of oleum, expressed as a percentage by mass, is given by the formula

$$100 - D$$

where D is the sulphuric acid content, expressed as a percentage by mass (see 7.2.2.3).

8 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO SULPHURIC ACID AND OLEUM FOR INDUSTRIAL USE

ISO 910 – Determination of total acidity, and calculation of free sulphur trioxide content of oleum – Titrimetric method.

ISO 911 – Evaluation of sulphuric acid concentration by measurement of density.*

ISO 912 – Determination of sulphur dioxide content – Barium sulphate gravimetric method.

ISO 913 – Determination of ash – Gravimetric method.

ISO 914 – Determination of total nitrogen content – Titrimetric method after distillation.

ISO/R 915 – Determination of iron content – 2,2′-Bipyridyl spectrophotometric method.

ISO 2363 – Determination of oxides of nitrogen – 2,4-Xylenol spectrophotometric method.

ISO 2717 – Determination of lead content – Dithizone photometric method.

ISO 2877 – Determination of chlorides content – Potentiometric method.*

ISO 2899 – Determination of ammoniacal nitrogen content – Spectrophotometric method.

ISO 3423 – Determination of sulphur dioxide content – Iodometric method.

ISO 5792 – Determination of arsenic content – Silver diethyldithiocarbamate photometric method.*

* Applicable only to sulphuric acid.

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