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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 910

SULPHURIC ACID AND OLEUM FOR INDUSTRIAL USE

DETERMINATION OF TOTAL ACIDITY AND CALCULATION OF FREE SO_3 CONTENT OF OLEUM

Volumetric method

1st EDITION
December 1968

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 910, *Sulphuric acid and oleum for industrial use – Determination of total acidity and calculation of free SO_3 content of oleum – Volumetric method*, was drawn up by Technical Committee ISO/TC 47, *Chemistry*, the Secretariat of which is held by the Ente Nazionale Italiano di Unificazione (UNI).

Based on detailed work on this question carried out by the Technical Committee, a Draft ISO Recommendation was adopted in 1965.

In June 1967, this Draft ISO Recommendation (No. 1180) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

One Member Body opposed the approval of the Draft :

United Kingdom

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in December 1968, to accept it as an ISO RECOMMENDATION.

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[*] **Instituto Centroamericano de Investigación y Tecnologia Industrial (Costa Rica, Guatemala, Honduras, Nicaragua, El Salvador, Panama).**

ISO Recommendation R 910 December 1968

SULPHURIC ACID AND OLEUM FOR INDUSTRIAL USE

DETERMINATION OF TOTAL ACIDITY AND CALCULATION OF FREE SO_3 CONTENT OF OLEUM

Volumetric method

1. SCOPE

This ISO Recommendation describes a volumetric method for the determination of the total acidity of sulphuric acid for industrial use, conventionally expressed as H_2SO_4, and for the calculation of free SO_3 content of oleum.

2. FIELD OF APPLICATION

The method is applicable to sulphuric acid and oleum for industrial use. Two cases are considered :

2.1 H_2SO_4 content equal to, or lower than, 98 %.

2.2 H_2SO_4 content higher than 98 %.

3. PRINCIPLE

Preliminary oxidation with hydrogen peroxide and titration of total acidity by means of a sodium hydroxide standard volumetric solution, in the presence of methyl red.

4. REAGENTS

Distilled water or water of equivalent purity, neutral to methyl red, should be used in the test.

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

4.2 *Sodium hydroxide,* N standard volumetric solution.
(see Note, section 7).

4.3 *Methyl red,* 1 g/l ethanolic solution.
Dissolve 0.1 g of methyl red in 95 % (v/v) ethanol and dilute to 100 ml with the same ethanol.

5. APPARATUS

Ordinary laboratory apparatus and

5.1 *Ground glass stoppered flask,* capacity approximately 500 ml, with neck of about 30 mm diameter.

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

5.3 *Burette,* precision at least 0.05 ml.

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

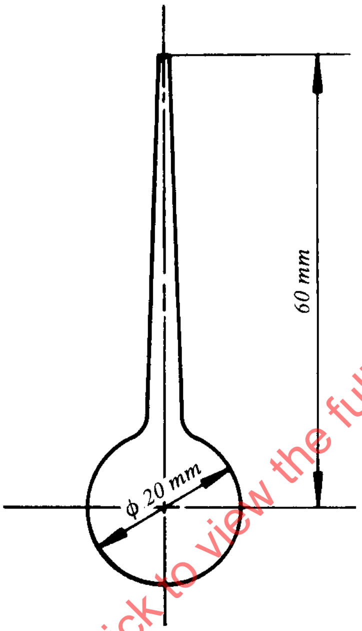

FIGURE – Spherical glass ampoule

6. PROCEDURE

6.1 H_2SO_4 content equal to or lower than 98 %

6.1.1 *Test portion.* In a weighing bottle, previously tared, weigh, to the nearest 0.1 mg, approximately 2 g of the test sample.

6.1.2 *Determination.* Transfer quantitatively the test portion (6.1.1) in a 500 ml conical flask containing approximately 300 ml of water.

Add 5 ml of the hydrogen peroxide solution (4.1) and gently boil for 10 minutes.

Allow to cool, add two drops of the methyl red solution (4.3) and titrate to the end-point by means of the sodium hydroxide solution (4.2).

6.2 H_2SO_4 content higher than 98 %

6.2.1 *Test portion.* Carefully mix the test sample by shaking the container. If the acid is partially crystallised, 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 0.1 mg.

Immerse the capillary end of the ampoule into the flask (5.1) containing the test sample and ensure that the bulb is filled up 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 at the 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 0.1 mg and calculate by difference the mass of the test portion.

6.2.2 *Preparation of sample solution.* Carefully place the ampoule containing the test portion (6.2.1) into 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 during this operation.

Keep cooling and shaking until the vapours are completely absorbed. Remove the stopper and rinse it with water, collecting the washings in the conical flask (5.4).

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 conical flask (5.4).

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

6.2.3 *Determination.* Transfer 100.0 ml of the sample solution (6.2.2) to a 500 ml conical flask.

Add 5 ml of the hydrogen peroxide solution (4.1) and gently boil for 10 minutes.

Allow to cool, add two drops of the methyl red solution (4.3) and titrate to the end-point by means of the sodium hydroxide solution (4.2).

7. EXPRESSION OF RESULTS

7.1 H_2SO_4 content equal to or lower than 98 %

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

$$\frac{V \times S \times 100}{E}$$

where

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

S is the mass, in grammes, of sulphuric acid corresponding to 1 ml of sodium hydroxide N solution (theoretical value 1 ml $\triangleq$ 0.04904 g of H_2SO_4, see Note below),

E is the mass, in grammes, of the test portion.

NOTE. – If the sodium hydroxide standard volumetric solution (4.3) is not of exactly the strength indicated in the list of reagents, a suitable correction factor should be employed in calculating the results.

7.2 H_2SO_4 content higher than 98 %

7.2.1 *Calculation of total acidity.* The total acidity, expressed as sulphuric acid (H_2SO_4), is given as a percentage, by mass, by the following formula :

$$A = \frac{V \times S \times 100}{E}$$

where the symbols have the same meaning as those of clause 7.1.

7.2.2 *Calculation of free SO_3 content of oleum.* Before calculating the free SO_3 content of oleum, it will be necessary to evaluate

- the total acidity expressed in SO_3 (see clause 7.2.2.1),
- water combined as sulphuric acid (see clause 7.2.2.2),
- sulphuric acid content (see clause 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, expressed as sulphur trioxide (SO_3), is given as a percentage, by mass, by the following formula :

$$B = A \times 0.8162$$

where

A is the total acidity expressed as a percentage by mass of H_2SO_4 (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).* Water combined as sulphuric acid is given as a percentage, by mass, by the following formula :

$$C = 100 - B$$

where

B is the total acidity expressed as a percentage by mass of SO_3 (7.2.2.1).