INTERNATIONAL STANDARD 3423

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

Sulphuric acid and oleums for industrial use — Determination of sulphur dioxide content — Iodometric method

Acide sulfurique et oléums à usage industriel — Dosage du dioxyde de soufre — Méthode iodométrique

First edition — 1975-11-01

UDC 661.25 : 546.224 : 543.242.3 **Ref. No. ISO 3423-1975 (E)**

Descriptors : sulphuric acid, chemical analysis, determination of content, sulphur dioxide, 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.

International Standard ISO 3423 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in February 1974.

It has been approved by the Member Bodies of the following countries :

Austria	India	South Africa, Rep. of
Belgium	Israel	Switzerland
Bulgaria	Italy	Thailand
Chile	Netherlands	Turkey
Egypt, Arab Rep. of	New Zealand	United Kingdom
France	Poland	U.S.S.R.
Germany	Portugal	Yugoslavia
Hungary	Romania	

No Member Body expressed disapproval of the document.

Printed in Switzerland

3423-75 4851903 0025166 7

Sulphuric acid and oleums for industrial use — Determination of sulphur dioxide content — Iodometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an iodometric method for the determination of the sulphur dioxide content of sulphuric acid and oleums for industrial use. The method is applicable to products of which the sulphur dioxide content is equal to or greater than 2 mg/kg.

NOTE — For sulphur dioxide contents higher than 50 mg/kg, see also ISO/R 912.

2 PRINCIPLE

Displacement of the sulphur dioxide present in a test portion by a current of pure nitrogen and absorption in a known volume of iodine solution.

Titration of the excess of iodine with standard volumetric sodium thiosulphate solution.

3 REAGENTS

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

3.1 Hydrazinium sulphate.

3.2 Iodine, approximately 0,1 N, 0,05 N or 0,01 N solution, as required (see 5.3).

Prepare this solution at the time of use.

3.3 Sodium thiosulphate, 0,1 N, 0,05 N or 0,01 N standard volumetric solution, as required.

Prepare this solution at the time of use.

3.4 Starch, 5 g/l solution.

3.5 Nitrogen, containing less than 0,001 % of oxygen.

It is recommended that the reduction valve from the nitrogen cylinder be connected to a gas-washing bottle containing a 150 g/l solution of titanium(III) chloride.

3.6 Crushed ice, prepared from distilled water or from water of equivalent purity.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Glass apparatus, with ground joints, comprising (see example in figure) :

A : **3-necked flask,** 500 ml or 250 ml capacity;

B : **cylindrical dropping funnel,** fitting into one of the two side necks of the flask;

C : **tube, fitted with a stopcock,** fitting into the central neck of the flask and terminating in a fritted disc at a level of about 1 cm above the bottom of the flask.

D_1, D_2, D_3 : **three gas-washing bottles,** Drechsel type, 100 or 125 ml capacity, with the inlet tubes terminating in fritted discs (porosity P16 (pore diameter 4 to 16 μm)) to disperse the gas.

4.2 Glass flask with ground glass stopper.

4.3 Flowmeter or **bubble-counter.**

5 PROCEDURE

5.1 Test portion

Fill the flask or weighing pipette (4.2) with the test sample and take, weighing by difference to the nearest 0,05 g, a test portion of approximately 200 g.

If the sample is of acid with a density higher than about 1,70 g/ml or is of oleum, slowly pour the test portion, cooling so that the temperature remains below 10 °C, onto a quantity of crushed ice (3.6) such that the density of the solution obtained is about 1,70 g/ml.

5.2 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all reagents as used in the determination.

5.3 Preparation of the apparatus

Place 50,00 ml of the iodine solution (3.2), of suitable concentration as indicated in the table, appropriate to the expected SO_2 content, in the first gas-washing bottle (D_1).

Divide 25,00 ml of the corresponding standard volumetric sodium thiosulphate solution (3.3) of concentration corresponding to that of the iodine solution, between the second and third gas-washing bottles (D_2, D_3), if necessary adding a little water so as to ensure sufficient scrubbing of the gas.

Expected sulphur dioxide content	Concentration of iodine solution
mg/kg	N
2 to 30	approximately 0,01
> 30 to 150	approximately 0,05
> 150	approximately 0,1

NOTE – The two bottles D_2 and D_3 are intended to retain any entrained iodine.

Place bottle D_1 in a cooling bath and check that, during all subsequent operations, the temperature of its contents do not rise above 10 °C and that it is not exposed to a bright light.

If the sample contains nitrite or nitrate ions, place an excess of the hydrazinium sulphate (3.1) in the flask (A). Connect the various parts of the apparatus together and displace the air by means of a rapid current of the nitrogen (3.5).

5.4 Determination

Run the test portion (5.1) into the flask (A) through the dropping funnel (B) and pass a current of the nitrogen (3.5) at a rate, measured by the flowmeter or bubble-counter (4.3), of about 20 l/h for 3 h. At the end of this time, there should still be an excess of iodine in the gas-washing bottle D_1.

Disconnect the three gas-washing bottles D_1, D_2 and D_3 and transfer their contents quantitatively into a beaker. Rinse the bottles with a few millilitres of water, collecting the washings in the beaker. Titrate the excess of iodine with the appropriate standard volumetric sodium thiosulphate solution (3.3) in the presence of the starch solution (3.4).

6 EXPRESSION OF RESULTS

The sulphur dioxide content, expressed as milligrams of sulphur dioxide (SO_2) per kilogram, is given by the formula

$$\frac{32 \times T \times 1\,000}{m}[(50{,}00 - 25{,}00 - V_1) - (50{,}00 - 25{,}00 - V_0)] = \frac{32\,000\,(V_0 - V_1) \times T}{m}$$

where

m is the mass, in grams, of the test portion;

T is the exact normality of the standard volumetric sodium thiosulphate solution used (3.3);

32 is the mass, in milligrams, of sulphur dioxide (SO_2) corresponding to 1 ml of exactly 1 N standard volumetric sodium thiosulphate solution;

V_0 is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (3.3) used for the blank test;

V_1 is the volume, in millilitres, of the standard volumetric sodium thiosulphate solution (3.3) used for the determination.

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 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.

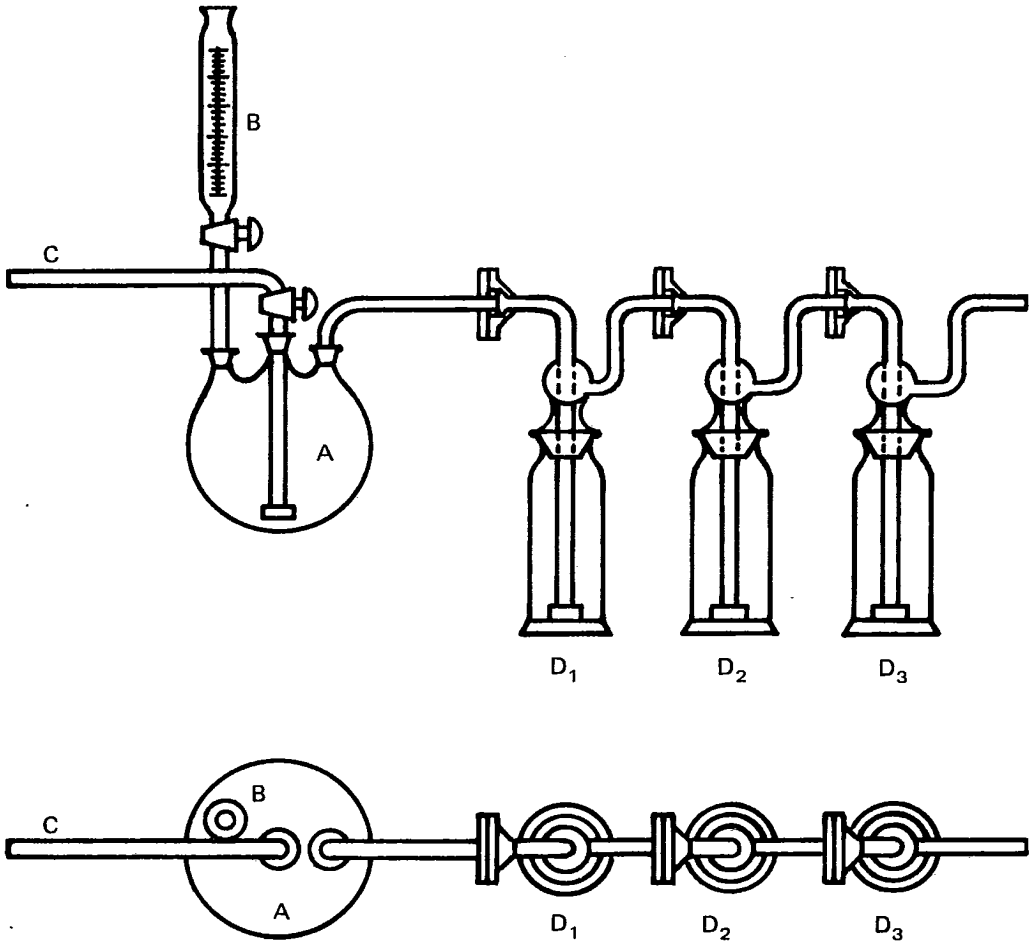

FIGURE — **Diagram of the glass apparatus**