
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

Sample Document

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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.

FOREWORD

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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
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Germany	Portugal	Yugoslavia
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No Member Body expressed disapproval of the document.

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.

D₁, D₂, D₃ : **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 SO₂ content, in the first gas-washing bottle (D₁).