INTERNATIONAL STANDARD ISO 5792

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Sulphuric acid for industrial use — Determination of arsenic content — Silver diethyldithiocarbamate photometric method

Acide sulfurique à usage industriel — Dosage de l'arsenic — Méthode photométrique au diéthyldithiocarbamate d'argent

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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 5792 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in February 1977.

It has been approved by the member bodies of the following countries :

Australia	Germany	Romania
Belgium	Hungary	South Africa, Rep. of
Brazil	Israel	Switzerland
Bulgaria	Italy	Turkey
Chile	Mexico	United Kingdom
Czechoslovakia	Netherlands	U.S.S.R.
France	Poland	Yugoslavia

No member body expressed disapproval of the document.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Printed in Switzerland

Sulphuric acid for industrial use — Determination of arsenic content — Silver diethyldithiocarbamate photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a silver diethyldithiocarbamate photometric method for the determination of the arsenic content of sulphuric acid for industrial use.

The method is applicable to products having arsenic (As) contents equal to or greater than 0,1 mg/kg.

2 REFERENCE

ISO 2590, *General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method.*

3 PRINCIPLE

See ISO 2590, clause 3. The reduction of arsenic by zinc is, however, carried out in sulphuric acid medium.

4 REAGENTS

See ISO 2590, clause 4; replace "4.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution, or approximately 12 N." by

4.1 Sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution, or approximately 36 N.

5 APPARATUS

See ISO 2590, clause 5; replace "5.1.1 Conical flask, 100 ml capacity, for the liberation of the arsine." by

5.1.1 Conical flask, 125 ml capacity, for the liberation of the arsine.

6 PROCEDURE

WARNING — See ISO 2590, clause 6.

6.1 Test portion

Determine the volume of the test sample that corresponds to 250 to 260 milliequivalents of sulphuric acid (generally this volume corresponds to a mass between 10 and 20 g). Weigh this quantity by difference, to the nearest 0,001 g, using, for example, a weighing pipette.

6.2 Preparation of the test solution

6.2.1 If the test portion (6.1) contains from 1 to 20 µg of As, introduce it slowly, while cooling to ambient temperature, into the conical flask (5.1.1) of the apparatus (5.1), containing sufficient water to give a final volume of about 65 ml. The resulting solution should have an acidity of between 3,8 N and 4 N.

6.2.2 If the test portion (6.1) contains more than 20 µg of As, add it slowly to water, while cooling to ambient temperature. Transfer the solution obtained quantitatively to a one-mark volumetric flask of suitable capacity, dilute to the mark and mix. Take an aliquot portion of accurately known volume not exceeding 50 ml and containing not more than 20 µg of As, and introduce it into the conical flask (5.1.1) of the apparatus (5.1). Add water or the sulphuric acid solution (4.1) so as to give a final volume of about 65 ml. The resulting solution should have an acidity of between 3,8 N and 4 N.

6.3 Blank test

See ISO 2590, sub-clause 6.2.

6.4 Preparation of the calibration graph

See ISO 2590, sub-clause 6.3.

6.4.1 *Preparation of the standard matching solutions*

See ISO 2590, sub-clause 6.3.1; however, use the 125 ml conical flask (5.1.1) and 7 ml of the sulphuric acid solution (4.1) instead of 10 ml of the hydrochloric acid solution, and the quantity of water necessary to obtain a final volume of about 65 ml; the addition shall be carried out while cooling.

6.4.2 *Photometric measurements*

See ISO 2590, sub-clause 6.3.2.

6.4.3 *Preparation of the calibration graph*

See ISO 2590, sub-clause 6.3.3.

6.5 Determination

To the test solution (6.2.1 or 6.2.2), contained in the conical flask (5.1.1), add 2 ml of the potassium iodide solution (4.6) and 2 ml of the tin(II) chloride solution (4.7); swirl and allow to stand for 15 min. Continue in accordance with the procedure specified in ISO 2590, sub-clause 6.3.1, starting from the third paragraph ("Place a little of the absorbent cotton wool . . .").

6.5.1 *Photometric measurements*

Carry out the photometric measurements on the test solution and the blank test solution according to the procedure specified in ISO 2590, sub-clause 6.4.1, after having, however, adjusted the instrument to zero absorbance against the silver diethyldithiocarbamate solution (4.2).

7 EXPRESSION OF RESULTS

By means of the calibration graph (see ISO 2590, sub-clause 6.3.3), determine the masses of arsenic corresponding to the value of the photometric measurement of the test solution and to that of the blank test solution.

The arsenic content, expressed in milligrams of arsenic (As) per kilogram, is given by the formula

$$\frac{m_1 - m_2}{m_0} \times D$$

where

m_0 is the mass, in grams, of the test portion (6.1);

m_1 is the mass, in micrograms, of As found in the test solution;

m_2 is the mass, in micrograms, of As found in the blank test solution;

D is the ratio of the volume of the test solution to the volume of the aliquot portion taken for the determination. (If the determination has been carried out on the whole of the test solution, D is equal to 1.)

8 TEST REPORT

The test report shall include the following particulars :

a) an identification of the sample;

b) the reference of the method used;

c) the results and the method of expression used;

d) any unusual features noted during the determination;

e) any operation not included in this International Standard or in the International Standard to which reference is made, 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 residue on ignition — 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.