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Ammonium sulphate for industrial use — Determination of arsenic content — Silver diethyldithiocarbamate photometric method

Sulfate d'ammonium à usage industriel — Dosage de l'arsenic — Méthode photométrique au diéthyldithiocarbamate d'argent

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FOREWORD

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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
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Ammonium sulphate 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 ammonium sulphate 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

Dissolution of a test portion, acidification with hydrochloric acid solution and application of the general method (see ISO 2590, clause 3).

4 REAGENTS

See ISO 2590, clause 4.

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

Weigh, to the nearest 0,001 g, about 20 g of the test sample.

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 into the conical flask (5.1.1) of the apparatus (5.1). Add 50 ml of water, stir until dissolution is complete and add 15 ml of the hydrochloric acid solution (4.1). The resulting solution should have an acidity of about 3 N.

6.2.2 If the test portion (6.1) contains more than 20 µg of As, dissolve it in water, 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 15 ml of the hydrochloric acid solution (4.1) and, if necessary, dilute to about 65 ml with water. The resulting solution should have an acidity of about 3 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.

6.5 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).

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7 EXPRESSION OF RESULTS

By means of the calibration graph (see ISO 2590, subclause 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 AMMONIUM SULPHATE FOR INDUSTRIAL USE

ISO 2992 – Determination of iron content – 2,2′-Bipyridyl photometric method.

ISO 2993 – Determination of free acidity – Titrimetric method.

ISO 2994 – Determination of matter insoluble in water – Gravimetric method.

ISO 3332 – Determination of ammoniacal nitrogen content – Titrimetric method after distillation.

ISO 3333 – Determination of copper content – Zinc dibenzyldithiocarbamate photometric method.

ISO 3694 – Determination of chloride ions content – Potentiometric method.

ISO 5786 – Determination of arsenic content – Silver diethyldithiocarbamate photometric method.

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