

INTERNATIONAL STANDARD**5372**

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

Condensed phosphates for industrial use (including foodstuffs) — Determination of arsenic content — Silver diethyldithiocarbamate photometric method*Phosphates condensés à usage industriel (y compris les industries alimentaires) — Dosage de l'arsenic — Méthode photométrique au diethyldithiocarbamate 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 5372 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	Hungary	Portugal
Belgium	India	Romania
Brazil	Israel	South Africa, Rep. of
Bulgaria	Italy	Switzerland
Czechoslovakia	Korea, Rep. of	Turkey
France	Mexico	United Kingdom
Germany	Poland	U.S.S.R.

The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

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

Condensed phosphates for industrial use (including foodstuffs) — 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 condensed phosphates for industrial use (including foodstuffs).

The method is applicable to products having arsenic (As) contents equal to or greater than 0,2 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 oxidation with potassium bromide-bromate solution, followed by hydrolysis. Determination of arsenic in accordance with the general method (see ISO 2590, clause 3).

4 REAGENTS

The reagents listed in clause 4 of ISO 2590 and

4.9 Potassium bromide-bromate solution, approximately 2 N.

Dissolve 20 g of potassium bromide (KBr) and 5,20 g of potassium bromate (KBrO₃) in water. Quantitatively transfer the solution obtained to a 100 one-mark volumetric flask, dilute to the mark and mix.

5 APPARATUS

See ISO 2590, clause 5.

6 PROCEDURE

WARNING — See ISO 2590, clause 6.

6.1 Test portion

Weigh, to the nearest 0,001 g, about 5 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 20 ml of water and the quantity of the hydrochloric acid solution (4.1) necessary to obtain a solution of pH 4. Dilute to about 30 ml and add 12 ml of the hydrochloric acid solution (4.1) and 1 ml of the potassium bromide-bromate solution (4.9). Fit a reflux condenser to the flask and boil the solution for 20 min; continue heating the solution on a boiling water bath until the bromine is completely eliminated. Cool the solution.

6.2.2 If the test portion (6.1) contains more than 20 µg of As, introduce it into a conical flask having a ground glass neck and of suitable capacity. Dissolve in approximately 20 ml of water and add the hydrochloric acid solution (4.1) so as to obtain a solution of pH approximately 4. Add a further 10 ml of the hydrochloric acid solution (4.1) and 1 ml of the potassium bromide-bromate solution (4.9). Fit a reflux condenser having a ground glass joint to the flask and boil the solution for 20 min; continue heating the solution on a boiling water bath until the bromine is completely eliminated. Cool, transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark and mix.

Take an aliquot portion of accurately known volume not exceeding 30 ml, and containing 1 to 20 µg of As, and introduce it into the conical flask (5.1.1) of the apparatus (5.1). Dilute to 30 ml, if necessary, and add 10 ml of the hydrochloric acid solution (4.1). The resulting solution should have an acidity of 3 N to 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.

6.5 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 (As) corresponding to the values of the photometric measurements of the test solution and 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 CONDENSED PHOSPHATES FOR INDUSTRIAL USE
(INCLUDING FOODSTUFFS)**

ISO 5372 – Determination of arsenic content – Silver diethyldithiocarbamate photometric method.

ISO 5373 – Determination of calcium content – Flame atomic absorption method.

ISO 5374 – Determination of chloride ions content – Potentiometric method.

ISO 5375 – Determination of oxides of nitrogen content – 3,4-Xylenol spectrophotometric method.