INTERNATIONAL STANDARD 5372

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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

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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).

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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 ($KBrO_3$) 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 . . .").