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INTERNATIONAL STANDARD



INTERNATIONAL ORGANIZATION FOR STANDARDIZATION MEЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ ORGANISATION INTERNATIONALE DE NORMALISATION

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

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

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FOREWORD

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International Standard ISO 3359. was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in April 1974.

It has been approved by the Member Bodies of the following countries:

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Phosphoric 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 phosphoric acid for industrial use.

The method can be used for the determination of quantities of arsenic, expressed as As, greater than 0,1 mg/kg.

2 REFERENCES

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

ISO 4285, Phosphoric acid for industrial use — Sampling technique.1)

3 PRINCIPLE

See ISO 2590, clause 3.

4 REAGENTS

The reagents listed in clause 4 of ISO 2590 and

4.9 Bromide-bromate solution, approximately 2 N.

Dissolve 20 g of potassium bromide (KBr) and 5,20 g of potassium bromate (KBrO₃) in water and dilute to 100 ml.

5 APPARATUS

See ISO 2590, clause 5.

6 PROCEDURE

WARNING - See ISO 2590, clause 6.

6.1 Test portion and preparation of the test solution

Weigh, to the nearest 1 mg, into the conical flask (5.1.1) a quantity of the test sample containing between 1 and 20 μg of As. This quantity shall not be greater than 10 g. Dilute with 10 ml of water, add 1 ml of the bromide-bromate

solution (4.9), and warm the solution on a boiling water bath. Then add to the hot solution, in small portions, the bromide-bromate solution until the yellow colour from the released bromine persists for about 5 min. Add 1 ml of the bromide-bromate solution in excess and continue heating on the boiling water bath until the disappearance of the yellow bromine colour. Cool the solution to room temperature, dilute to about 30 ml and add 10 ml of the hydrochloric acid solution (4.1).

NOTE — If hydrochloric acid solution of concentration approximately 12 N is not available, a solution of lower concentration may be used, by adding the corresponding stoichiometric quantity of HCl and adjusting the dilution to reach the same final volume of of 40 ml.

6.2 Blank test

See ISO 2590, sub-clause 6.2.

6.3 Preparation of the calibration curve

See ISO 2590, sub-clause 6.3.

6.4 Determination

Follow the procedure specified in sub-clause 6.4 of ISO 2590.

7 EXPRESSION OF RESULTS

By means of the calibration curve (see 6.3.3), determine the quantity of arsenic (As) corresponding to the value of the photometric measurement of the test solution.

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

$$\frac{m_1}{m_0}$$

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.

¹⁾ At present at the stage of draft.

8 TEST REPORT

The test report shall include the following particulars:

- a) the reference of the method used;
- b) the results and the method of expression used;
- c) any unusual features noted during the determination;
- d) any operation not included in this International Standard or in the International Standards to which reference is made, or regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO PHOSPHORIC ACID FOR INDUSTRIAL USE

ISO/R 848 — Determination of calcium content — Volumetric method.

ISO/R 849 - Determination of iron content - 2,2'-bipyridyl spectrophotometric method.

ISO 2997 - Determination of sulphate content -- Method by reduction and titrimetry.

ISO 3359 — Determination of arsenic content — Silver diethyldithiocarbamate photometric method.

ISO 3360 — Determination of fluorine content — Alizarin complexone and cerium chloride photometric method.

ISO 3361 - Determination of silica content - Reduced molybdosilicate spectrophotometric method.

ISO 3706 — Determination of phosphorus(V) oxide content.

ISO 3707 — Determination of calcium content — Flame atomic absorption spectrophotometric method.

ISO 3708 — Determination of chlorides content — Potentiometric method.

ISO 3709 - Determination of nitrogen oxides.

ISO 4285 — Sampling technique.