INTERNATIONAL STANDARD ISO 2590

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

General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method

First edition — 1973-05-01

UDC 546.19 : 543.42

Ref. No. ISO 2590-1973 (E)

Descriptors : chemical analysis, determination of content, arsenic, photometry.

Price based on 5 pages

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 2590 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in November 1971.

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

Austria	Netherlands	Switzerland
Belgium	New Zealand	Thailand
Egypt, Arab Rep. of	Poland	Turkey
France	Portugal	United Kingdom
Hungary	Romania	U.S.A.
India	South Africa, Rep. of	U.S.S.R.
Israel	Spain	
Italy	Sweden	

The Member Body of the following country expressed disapproval of the document on technical grounds :

Germany

Printed in Switzerland

General method for the determination of arsenic — Silver diethyldithiocarbamate photometric method

1 SCOPE

This International Standard specifies a general method for the photometric determination of arsenic, using silver diethyldithiocarbamate.

2 FIELD OF APPLICATION

The method is applicable to the determination of quantities of arsenic (As), contained either in all the test solution or in the aliquot portion taken for the determination, of between 1 and 20 μg.

3 PRINCIPLE

Reduction of the arsenic by zinc in a hydrochloric acid medium, with the formation of arsine.

Absorption of the arsine in a solution of silver diethyldithiocarbamate in pyridine.

Photometric measurement of the purplish-red colour produced by the colloidally dispersed silver at the maximum of the absorption curve (wavelength approximately 540 nm).

NOTE — The reaction of the formation of the colloidal silver is :

$$AsH_3 + 6\ Ag(DDTC) = 6\ Ag + 3\ H(DDTC) + As(DDTC)_3$$

4 REAGENTS

All the reagents, and the zinc in particular, shall be free from arsenic or have a very low arsenic content.

Distilled water, or water of equivalent purity, shall be used in the test.

4.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution, or approximately 12 N.

4.2 Silver diethyldithiocarbamate, [Ag(DDTC)], 5 g/l solution in pyridine.

Dissolve 1 g of silver diethyldithiocarbamate in pyridine (ρ approximately 0,980 g/ml) and dilute to 200 ml with the same pyridine.

Store the solution in a tightly sealed, dark glass bottle, protected from light.

This solution remains stable for about 2 weeks.

4.3 Arsenic standard solution, 0,100 g/l.

Weigh, to the nearest 0,000 1 g, 0,132 0 g of arsenic trioxide (As_2O_3) and transfer it to a beaker of suitable capacity (for example, 100 ml). Dissolve the arsenic trioxide in about 2 ml of 50 g/l sodium hydroxide solution. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask. Wash the beaker several times, collecting the wash water in the volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 100 μg of As.

4.4 Arsenic standard solution, 2,50 mg/l.

Transfer 25,0 ml of the arsenic standard solution (4.3) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. Prepare this solution immediately before use.

1 ml of this standard solution contains 2,5 μg of As.

4.5 Absorbent cotton wool saturated with lead acetate.

Dissolve 50 g of lead acetate trihydrate [$Pb(C_2H_3O_2)_2.3H_2O$] in 250 ml of water. Saturate the absorbent cotton wool with this solution, remove the excess solution by allowing it to drain and dry the cotton wool under vacuum at room temperature.

Store it in an air-tight container.

4.6 Potassium iodide, 150 g/l solution.

Dissolve 15 g of potassium iodide in water, dilute to 100 ml and mix.

4.7 Tin(II) chloride, hydrochloric acid solution.

Dissolve 40 g of tin(II) chloride dihydrate ($SnCl_2.2H_2O$) in a mixture of 25 ml of water and 75 ml of the hydrochloric acid solution (4.1).

4.8 Zinc shot, 0,5 to 1 mm, or any other form of zinc which has been shown, by experiment, to give equivalent results under the specified test conditions (see Annex).

5 APPARATUS

All the glass containers used for the determination of arsenic shall be washed with hot concentrated sulphuric acid, taking the necessary precautions, rinsed thoroughly with water and dried completely.

Ordinary laboratory apparatus and

5.1 Glass apparatus with ground glass joints, for the liberation and total absorption of the arsine.

NOTE – See ISO/R 383 and ISO/R 641 for details of conical and spherical ground glass joints.

A suitable apparatus is shown in the Figure, and consists of :

5.1.1 Conical flask, 100 ml capacity, for the liberation of the arsine.

5.1.2 Connecting tube, to trap hydrogen sulphide.

5.1.3 15-Bulb absorption vessel.

5.2 Spectrophotometer, or

5.3 Photoelectric absorptiometer, fitted with filters giving a maximum transmission between 520 and 560 nm.

6 PROCEDURE

WARNING – Because of the toxicity and unpleasant odour of pyridine, it is recommended that it should be handled with care and in a well-ventilated fume cupboard.

6.1 Test portion and preparation of the test solution

Weigh the quantity of the test sample indicated in the International Standard relating to the product concerned and treat it in the appropriate manner so as to obtain a test solution according to the conditions described below.

In general, the test solution should be prepared so that its total volume, or that of the aliquot portion taken for the determination, is 40 ml, and so that it contains between 1 and 20 µg of As and 10 ml of the hydrochloric acid solution (4.1). The resulting acidity is about 3 N before the addition of the potassium iodide solution. The test solution must be absolutely free from nitrate ions; moreover, certain elements (cobalt, mercury, silver, copper, molybdenum, palladium, etc.) will reduce the yield of arsine. The procedure should, if necessary, indicate how to take account of their presence.

NOTE – In the case where the test solution is necessarily a sulphuric acid medium, the acidity of the 40 ml of test solution should be about 3,8 to 4 N (containing 10 ml of an approximately 15 N sulphuric acid solution).

6.2 Blank test

Carry out a blank test in parallel with the determination, following the same procedure and using the same quantities of all the reagents used for the determination.

6.3 Preparation of the calibration curve

The calibration curve shall be prepared each time that a new batch of zinc is used and each time that a new solution of silver diethyldithiocarbamate is prepared.

6.3.1 *Preparation of the standard matching solutions* for photometric measurements with a 1 cm cell.

Transfer into six separate conical flasks (5.1.1) the volumes of the arsenic standard solution (4.4) indicated in the following table :

Volume of the arsenic standard solution (4.4)	Corresponding mass of arsenic
ml	µg
0 *	0
1,00	2,5
2,00	5
4,00	10
6,00	15
8,00	20

* Compensation solution

Add to each flask 10 ml of the hydrochloric acid solution (4.1) and the quantity of water necessary to make up to about 40 ml. Add 2 ml of the potassium iodide solution (4.6) and 2 ml of the tin(II) chloride solution (4.7); swirl and then allow to stand for 15 min.

NOTE – In the case of test solutions in a sulphuric acid medium, instead of adding 10 ml of the hydrochloric acid solution, add 10 ml of an approximately 15 N solution of sulphuric acid.

Place a little of the absorbent cotton wool (4.5) in the connecting tube (5.1.2) in order to absorb any hydrogen sulphide which may be released with the arsine.

Grease the ground glass joints with a grease that is insoluble in pyridine, transfer 5,0 ml of the silver diethyldithiocarbamate solution (4.2) to the absorption vessel (5.1.3) and attach the connecting tube (5.1.2) to the absorption vessel by means of a safety clip.

After allowing to stand for 15 min, add to the conical flask (5.1.1), with the aid of a powder funnel, 5 g of the zinc (4.8) and rapidly assemble the apparatus as indicated in the Figure.

Allow approximately 45 min for the reaction to take place.

Detach the absorption vessel (5.1.3), swirl in order to dissolve the red deposit formed in the lower part and completely mix the solution.

The colour of the solution is stable, in the absence of light, for about 2 h and the measurements shall be carried out within this period.

6.3.2 *Photometric measurements*

Carry out the measurements using the spectrophotometer (5.2) at the maximum of the absorption curve (wavelength approximately 540 nm), or with the photoelectric absorptiometer (5.3), fitted with suitable filters, after having adjusted the instrument, in each case, to zero absorbance against the compensation solution.

6.3.3 *Preparation of the calibration chart*

Plot a graph having, for example, the arsenic contents in micrograms per 5 ml of standard matching solution as abscissae, and the corresponding values of absorbance as ordinates.

6.4 **Determination**

To 40 ml of the test solution (6.1), 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.

Complete the operations according to the procedure described in 6.3.1.

6.4.1 *Photometric measurement*

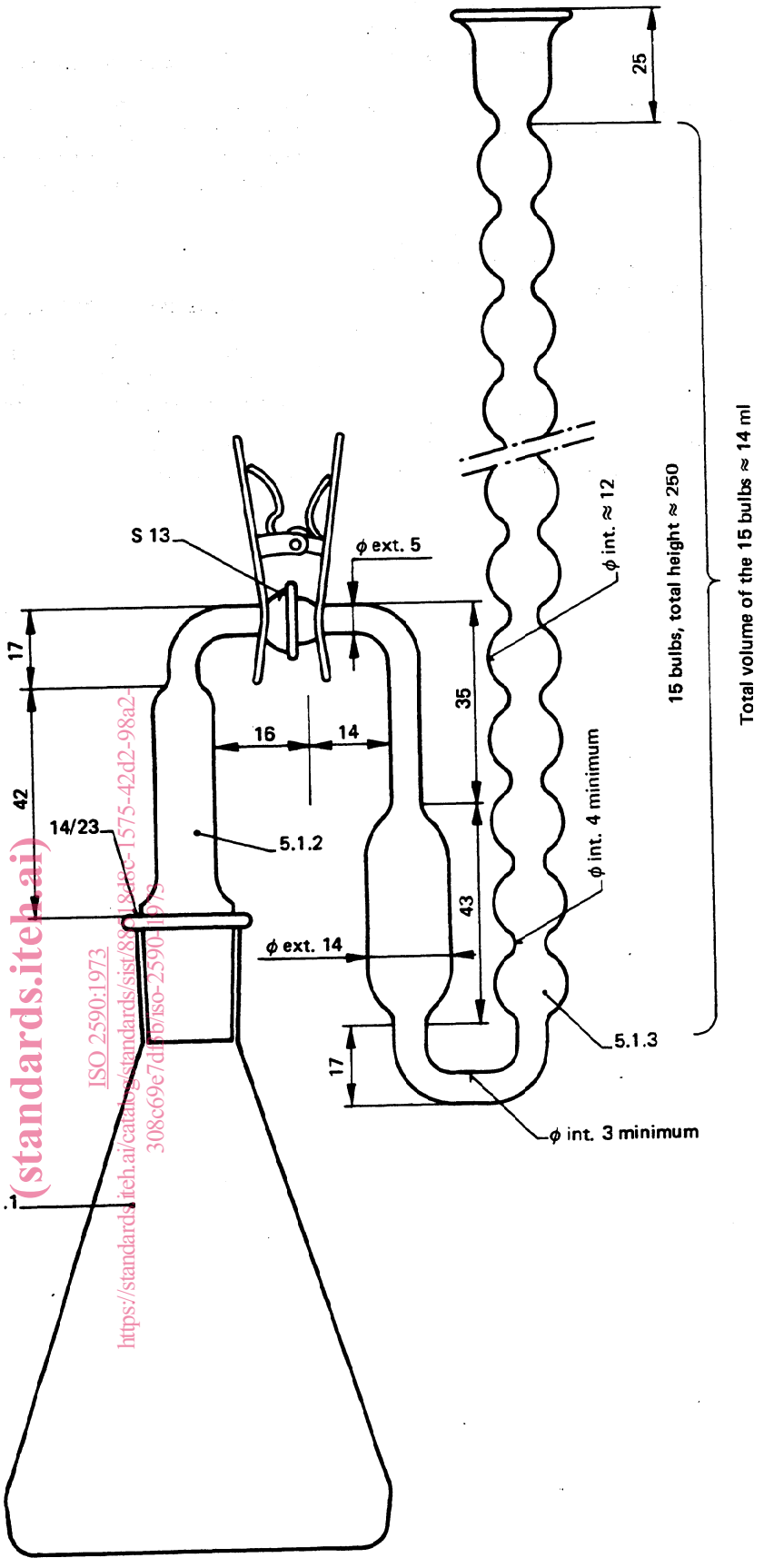
Carry out the photometric measurement on the test solution according to the method described in 6.3.2, after having adjusted the instrument to zero absorbance against the blank test solution (6.2).

7 EXPRESSION OF RESULTS

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

The International Standards relating to the product in question will give the formulae to be applied for the final calculation.

Dimensions in millimetres



FIGURE

ANNEX

USE OF ZINC IN NEEDLE FORM

When, for any reason, zinc in the form of needles is used in place of zinc shot, the following modifications shall be made to the method :

4.8 read : "Zinc, in needles 2 to 3 mm in diameter."

6.1 2nd paragraph – read : "In general, the test solution should be prepared so that its total volume, or that of the aliquot portion taken for the determination, is 30 ml, and so that it contains 1 to 20 μg of As and 10 ml of the hydrochloric acid solution (4.1). The acidity is about 4 N before the addition of the potassium iodide solution...."

6.3.1 2nd paragraph – read : "Add to each flask 10 ml of the hydrochloric acid solution (4.1) and the quantity of water necessary to make up the volume to about 30 ml. Add 2 ml of the potassium iodide solution (4.6) and 2 ml of the tin(II) chloride solution (4.7); ..."

Penultimate paragraph – read : "Allow 1 h for the reaction to take place."

This page intentionally left blank