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INTERNATIONAL STANDARD ISO 2465

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

Glycérines à usage industriel — Dosage de l'arsenic — Méthode photométrique au diéthyldithiocarbamate 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.

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

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

No Member Body expressed disapproval of the document.

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INTERNATIONAL STANDARD ISO 2465-1974 (E)

Glycerols 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 glycerols for industrial use.

1.1 General case

The method is applicable to the analysis of glycerols having an arsenic content greater than 0,1 mg/kg. It is applied successively to a test portion as it is, and to another identical portion with a known quantity of arsenic added. The determination is considered to be valid if the amount added is recovered.

1.2 Special case

If the arsenic added is not recovered or is recovered only partially, it is advisable to recommence the determination, after first igniting the test portion in an oxidizing medium in order to destroy the organic substances capable of inhibiting the arsine formation, without loss of arsenic.

A control test is also carried out in this case, after ignition, on an identical sample enriched by a known amount of arsenic, which should be recovered.

NOTE — Ignition should also be applied to the standard matching samples prepared with the aid of double-distilled glycerol in order to prepare the calibration curve, otherwise the results obtained will be slightly below the correct values.

2 REFERENCES

ISO 2096, *Glycerols for industrial use — Methods of sampling.*

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

3 PRINCIPLE

See ISO 2590, clause 3.

4 REAGENTS

See ISO 2590, clause 4.

5 APPARATUS

See ISO 2590, clause 5.

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 portions

Weigh, to the nearest 0,1 g, two identical portions of 10 g of the test sample (see ISO 2096), directly into each of two of the conical flasks (5.1.1).

6.2 Blank test

Carry out a blank test at the same time as the determination and following the same procedure, using the same quantities of all the reagents.

6.3 Preparation of the calibration curve

See ISO 2590, sub-clause 6.3.

6.3.1 *Preparation of standard matching solutions*

See ISO 2590, sub-clause 6.3.1, modifying the text as follows :

- Delete the note.
- Amend the third paragraph after the note to read : "After allowing to stand for 15 min, dilute the test solution to 90 ml with water, add to the conical flask (5.1.1) ... "

6.3.2 *Photometric measurements*

See ISO 2590, sub-clause 6.3.2.

6.3.3 *Plotting the calibration curve*

See ISO 2590, sub-clause 6.3.3.

6.4 Control test

Add 5 μg of As in the form of 2,00 ml of the arsenic standard solution (4.4) to the first test portion (6.1).

6.5 Determination

Add 10 ml of water and 10 ml of the hydrochloric acid solution (4.1) to both of the conical flasks, containing the control test (6.4) and the second test portion (6.1), and treat each solution as follows :

Dilute to 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 allow to stand for 15 min.

Continue as indicated in 6.3.1.

6.5.1 *Photometric measurements*

See ISO 2590, sub-clause 6.4.1.

7 EXPRESSION OF RESULTS

By means of the calibration curve (see 6.3.3), determine the quantities of arsenic (As) corresponding to the determination and to the control test, i.e.

the mass m_1, in micrograms, of As found in the determination (test portion as it is);

the mass m_2, in micrograms, of As found in the control test (test portion with 5 μg of As added);

the mass m_0, in grams, of the second test portion.

If the difference, $m_2 - m_1$, corresponds to the quantity of arsenic added to the first test portion (i.e. 5 μg of As), the determination is considered to be valid. In this case, the arsenic content of the sample, expressed as As, is given, in milligrams per kilogram, by the formula

$$m_1 \times \frac{1}{1\,000} \times \frac{1\,000}{m_0} = \frac{m_1}{m_0}$$

Express the result to one place of decimals.

8 SPECIAL CASE

Determination after ignition of the test portion. This particular case is applicable when the difference, $m_2 - m_1$, obtained according to clause 7 of the general case, differs by at least 2 μg from the quantity of As added to the first test portion.

8.1 Principle

Combustion of the glycerol in the presence of magnesium oxide. Ignition at 600 °C of the residue to which magnesium nitrate has been added. Dissolution in hydrochloric acid.

Application of the principle given in clause 3.

8.2 Reagents

Use the reagents specified in clause 4, adding the reagents listed below, the arsenic content of which should also be nil or very low.

8.2.1 *Glycerol,* double-distilled, ρ 1,23 g/ml.

8.2.2 *Magnesium oxide.*

8.2.3 *Magnesium nitrate hexahydrate* $[Mg(NO_3)_2.6H_2O]$, 100 g/l solution.

8.3 Apparatus

Use the apparatus specified in clause 5 and

8.3.1 *Porcelain dishes,* about 40 mm high, 100 mm upper diameter and 50 mm lower diameter.

8.3.2 *Electric oven,* capable of being controlled at 150 ± 10 °C.

8.3.3 *Muffle furnace,* capable of being controlled between 550 and 600 °C.

8.4 Procedure

8.4.1 *Test portions*

Weigh, to the nearest 0,1 g, two identical portions of 10 g of the test sample (see ISO 2096) directly into each of two of the porcelain dishes (8.3.1).

8.4.2 *Blank test*

Carry out a blank test at the same time as the determination and following the same procedure, using the same quantities of all the reagents.

8.4.3 *Preparation of calibration curve*

8.4.3.1 PREPARATION OF THE STANDARD MATCHING SOLUTIONS, for photometric measurements with a 1-cm cell.

Transfer to a series of six of the porcelain dishes (8.3.1) the volumes of the arsenic standard solution (4.4) indicated in the table in sub-clause 6.3.1 of ISO 2590. Add 10 g of the glycerol (8.2.1) and 2 g of the magnesium oxide (8.2.2), mix with a glass rod and heat gently, on a hot-plate for example, until an apparently dry residue is obtained. Increase the heat and ignite the glycerol with which the magnesium oxide is coated.

After the combustion, allow to cool, add 10 ml of the magnesium nitrate solution (8.2.3) and evaporate to dryness in the oven (8.3.2), controlled at 150 ± 10 °C. Place the dishes in the furnace (8.3.3), controlled at between 550 °C and 600 °C, and leave them for 1 h.

After cooling, dissolve the residue in 20 ml of water and 20 ml of the hydrochloric acid solution (4.1), stirring the solution with a glass rod. Transfer the solutions quantitatively to two of the conical flasks (5.1.1), add 2 ml of the potassium iodide solution (4.6), followed by 2 ml of the tin(II) chloride solution (4.7), swirl and leave to stand for 15 min.

Follow the procedure described in 6.3.1 of ISO 2590 starting from the first paragraph after the note : "Place a little of the absorbent cotton wool (4.5) . . . "

8.4.3.2 PHOTOMETRIC MEASUREMENTS

Proceed according to 6.3.2.

8.4.3.3 PLOTTING THE CALIBRATION CURVE

Proceed according to 6.3.3.

8.4.4 *Control test*

Add 5 μg of As in the form of 2,00 ml of the arsenic standard solution (4.4) to the first test portion (8.4.1).

8.4.5 *Determination*

Add 2 g of the magnesium oxide (8.2.2) to both of the porcelain dishes containing the control test (8.4.4) and the second test portion (8.4.1) and treat the contents of each dish as follows :

Mix using a glass rod and heat gently on a hot plate for example, until an apparently dry residue is obtained. Then increase the heat andignite the glycerol with which the magnesium oxide is coated.

Follow the procedure specified in 8.4.3.1, starting from the second paragraph : "After the combustion, allow . . ."

8.4.6 *Photometric measurements*

Proceed according to 6.5.1.

8.4.7 *Expression of results*

Calculate the results as indicated in clause 7.

9 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 the International Standards to which reference is made, or regarded as optional.