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Ammonium sulphate for industrial use — Determination of copper content — Zinc dibenzyldithiocarbamate photometric method

Sulfate d'ammonium à usage industriel — Dosage du cuivre — Méthode photométrique au dibenzyldithiocarbamate de zinc

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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 3333 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in March 1974.

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

Austria	Hungary	South Africa, Rep. of
Belgium	India	Spain
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No Member Body expressed disapproval of the document.

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Ammonium sulphate for industrial use — Determination of copper content — Zinc dibenzyldithiocarbamate photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a zinc dibenzyldithiocarbamate photometric method for the determination of the copper content of ammonium sulphate for industrial use.

The method is applicable to products of which the copper content is greater than 0,1 mg/kg.

2 PRINCIPLE

Formation of a coloured complex between the copper and zinc dibenzyldithiocarbamate and photometric measurement at a wavelength of about 435 nm.

3 REAGENTS

During the analysis, use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

3.1 Carbon tetrachloride, redistilled.

3.2 Hydrochloric acid, approximately 2 N solution, freed from copper by extraction with the zinc dibenzyldithiocarbamate solution (3.3).

3.3 Zinc dibenzyldithiocarbamate, 0,5 g/l solution in carbon tetrachloride.

Dissolve 0,05 g of zinc dibenzyldithiocarbamate ($[(C_6H_5CH_2)_2NCSS]_2Zn$) in the carbon tetrachloride (3.1) and dilute to 100 ml with the same carbon tetrachloride.

3.4 Copper, standard solution corresponding to 0,100 g of Cu per litre.

Weigh, to the nearest 0,000 1 g, 0,1 g of electrolytic copper and dissolve in 10 ml of approximately 8 N nitric acid solution. Heat the solution on a hot-plate until the fumes evolved are no longer brown, cool and add about 100 ml of water. Transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,10 mg of Cu.

3.5 Copper, standard solution corresponding to 0,001 0 g of Cu per litre.

Transfer 10,0 ml of the standard copper solution (3.4) to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 1 μg of Cu.

Prepare this solution at the time of use.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Spectrophotometer, or

4.2 Photoelectric absorptiometer, fitted with filters giving a maximum transmission between 430 and 440 nm.

4.3 Burette, graduated in 0,02 ml.

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0,01 g, a mass of the test sample containing not more than 10 μg of copper.

5.2 Blank test

Carry out a blank test under the same conditions as the determination, using the same procedure and the same quantities of all the reagents as used for the determination.

5.3 Preparation of the calibration curve

5.3.1 *Preparation of standard colorimetric solutions* relating to measurements carried out with cells of 4 cm or 5 cm optical path length.

Into a series of five 250 ml separating funnels, each containing 10 ml of the hydrochloric acid solution (3.2), transfer, by means of the burette (4.3), the volumes of the

standard copper solution (3.5) indicated in the following table :

Standard copper solution (3.5)	Corresponding mass of copper
ml	µg
0*	0
2,5	2,5
5,0	5,0
7,5	7,5
10,0	10,0

* Compensation solution.

5.3.2 *Colour development*

Treat the contents of each separating funnel as follows. Dilute to approximately 100 ml with water and mix. Add 10,0 ml of the zinc dibenzyldithiocarbamate solution (3.3), shake the funnel vigorously for 1 min and allow the layers to separate. Run off the lower, organic, layer and filter through a retentive filter paper into a 25 ml one-mark volumetric flask. Repeat the extraction with 5 ml of the zinc dibenzyldithiocarbamate solution (3.3) and filter the organic layer into the same flask. Repeat this operation once more. Wash the filter paper with the carbon tetrachloride (3.1), adding the washings to the flask. Dilute to the mark with the carbon tetrachloride (3.1) and mix.

5.3.3 *Photometric measurements*

Carry out the photometric measurements using the spectrophotometer (4.1), at a wavelength of about 435 nm, or the photoelectric absorptiometer (4.2) fitted with suitable filters, after having adjusted the instrument to zero absorbance against the carbon tetrachloride (3.1). Deduct the absorbance of the compensation solution from those of the standard colorimetric solutions.

5.3.4 *Preparation of the calibration chart*

Plot a graph having, for example, the numbers of micrograms of copper in 25 ml of the standard colorimetric solutions as abscissae and the corresponding values of the absorbance as ordinates.

5.4 Determination

5.4.1 *Preparation of sample solution*

Dissolve the test portion (5.1) in 50 ml of water and add 10 ml of the hydrochloric acid solution (3.2). Transfer quantitatively to a 250 ml separating funnel and dilute to a volume of about 100 ml.

5.4.2 *Colour development*

Add 10,0 ml of the zinc dibenzyldithiocarbamate solution (3.3) and proceed as specified in 5.3.2.

5.4.3 *Photometric measurements*

Carry out the photometric measurements on the test solution and on the blank test solution using the spectrophotometer (4.1), at a wavelength of about 435 nm, or the photoelectric absorptiometer (4.2) fitted with suitable filters, after having adjusted the instrument to zero absorbance against the carbon tetrachloride (3.1).

6 EXPRESSION OF RESULTS

By reference to the calibration chart (see 5.3.4), determine the masses of copper corresponding to the absorbances of the test solution and that of the blank test solution.

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

$$\frac{m_1 - m_2}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (5.1);

m_1 is the mass, in micrograms, of copper found in the test solution;

m_2 is the mass, in micrograms, of copper found in the blank test solution.

7 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 regarded as optional.

ANNEX

ISO PUBLICATIONS RELATING TO AMMONIUM SULPHATE FOR INDUSTRIAL USE

ISO 2992 — Determination of iron content — 2,2′-bipyridyl photometric method.

ISO 2993 — Determination of free acidity — Titrimetric method.

ISO 2994 — Determination of matter insoluble in water — Gravimetric method.

ISO 3332 — Determination of ammoniacal nitrogen content — Volumetric method after distillation.

ISO 3333 — Determination of copper content — Zinc dibenzyldithiocarbamate photometric method.

ISO 3694 — Determination of chlorides content — Potentiometric method.