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Boric acid, boric oxide and *Di*sodium tetraborates for industrial use — Determination of copper content — Zinc dibenzyldithiocarbamate photometric method

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FOREWORD

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Austria	Israel	Sweden
Belgium	Italy	Switzerland
Czechoslovakia	Netherlands	Thailand
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Boric acid, boric oxide and *Di*sodium tetraborates for industrial use — Determination of copper content — Zinc dibenzyldithiocarbamate photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a zinc dibenzyldithiocarbamate method for the photometric determination of the copper content of boric acid, boric oxide and *di*sodium tetraborates for industrial use.

2 PRINCIPLE

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

3 REAGENTS

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

3.1 Carbon tetrachloride, redistilled.

3.2 Hydrochloric acid, approximately 6 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 in the carbon tetrachloride (3.1) and dilute to 100 ml with the same carbon tetrachloride.

3.4 Copper standard solution, containing 0.10 g/l of Cu.

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 solution contains 0.10 mg of Cu.

3.5 Copper standard solution, containing 0.010 g/l of Cu.

Transfer 10.0 ml of the copper standard solution (3.4) to a 100 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 10 μg of Cu.

Prepare this solution just before use.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Spectrophotometer, with 4 cm cells, or

4.2 Photoelectric absorptiometer, with 4 cm cells.

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0.1 g, a mass of the test sample as indicated in the following table :

Material		Mass, in grams, of test portion
boric acid	(H_3BO_3)	10
boric oxide	(B_2O_3)	5
*di*sodium tetraborate decahydrate	($Na_2B_4O_7 \cdot 10H_2O$)	15
*di*sodium tetraborate pentahydrate	($Na_2B_4O_7 \cdot 5H_2O$)	10
anhydrous disodium tetraborate	($Na_2B_4O_7$)	10

5.2 Blank test

At the same time as the analysis, carry out a blank test using the same procedure and quantities of all reagents employed in the determination.

5.3 Preparation of calibration curve

5.3.1 *Preparation of standard colorimetric solutions*

Into a series of five 250 ml separating funnels, each containing 50 ml of the hydrochloric acid solution (3.2), transfer the quantities of the copper standard solution (3.5) indicated in the following table :

Volume of copper standard solution (3.5)	Corresponding mass of copper
ml	µg
0[1)]	0
0.5	5
1.0	10
1.5	15
2.0	20

1) Compensation solution

5.3.2 *Development of the colour*

Dilute the contents of each separating funnel to approximately 200 ml with water and mix. Add 10 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 it through a retentive filter paper into a 25 ml one-mark volumetric flask. Treat the aqueous phase with a 10 ml portion of the zinc dibenzyldithiocarbamate solution (3.3) and filter the organic layer into the same flask. 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*

Using the spectrophotometer (4.1) at a wavelength of about 435 nm or the photoelectric absorptiometer (4.2) with suitable filters carry out the photometric measurements, adjusting 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 *Plotting of the calibration curve*

Plot a graph having, for example, the quantities in micrograms of copper in 25 ml of the standard colorimetric solution as abscissae and the corresponding values of the abosrbance as ordinates.

5.4 Determination

5.4.1 *Preparation of test solution*

Dissolve the test portion (5.1) in 140 ml of water and add 60 ml of the hydrochloric acid solution (3.2). Simmer the solution for 10 min, cool and transfer quantitatively to a 250 ml separating funnel.

5.4.2 *Development of the colour*

Develop the colour following the instructions given in 5.3.2.

5.4.3 *Photometric measurements*

Carry out the photometric measurements on the test solution and on the blank solution following the instructions given in 5.3.3, adjusting the instrument to zero absorbance against the carbon tetrachloride (3.1).

6 EXPRESSION OF RESULTS

By reference to the calibration curve (see 5.3.4) determine the mass of copper corresponding to the absorbance of the test solution and that of the blank test.

The copper content (Cu), expressed in parts per million by mass, is given by the formula :

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

where

m_0 is the mass, in grams, of the test portion;

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.