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Copper and copper alloys — Determination of manganese — Spectrophotometric method

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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 :

Australia	Germany	South Africa, Rep. of
Belgium	India	Spain
Canada	Italy	Sweden
Czechoslovakia	Netherlands	Switzerland
Denmark	New Zealand	Thailand
Egypt, Arab Rep, of	Norway	Turkey
Finland	Poland	United Kingdom
France	Romania	U.S.A.

No Member Body expressed disapproval of the document.

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Copper and copper alloys — Determination of manganese — Spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a spectrophotometric method for the determination of manganese in copper and copper alloys.

The method is applicable to copper and all copper alloys, bronzes, brasses, copper-nickels and copper-aluminiums having manganese contents equal to or less than 6 %, which can be attacked under the conditions given in this International Standard.

2 PRINCIPLE

Fluoroboric-nitric acid attack of the test portion. Oxidation of manganese to permanganic acid by potassium periodate.

Spectrophotometry in comparison with a background colour prepared by selective reduction of the permanganic acid by sodium nitrite.

3 REAGENTS

All the reagents shall be of the analytical grade. Use distilled or deionized water.

3.1 Boric acid, 40 g/l solution.

3.2 Attack reagent.[1)]

Mix together :

boric acid, 40 g/l	300 ml
hydrofluoric acid, 40 %, ρ 1,14 g/ml	30 ml
nitric acid, ρ 1,40 g/ml	500 ml
water	150 ml

3.3 Dilution solution.[2)]

For the dilutions, use a boric acid solution, 40 g/l, acidified to 1 % with sulphuric acid, ρ 1,84 g/ml, of which the organic materials likely to reduce the permanganate are oxidized by the addition, when boiling, of a few crystals of potassium periodate.

3.4 Potassium periodate, 50 g/l solution in nitric acid, ρ 1,40 g/ml diluted 1 + 3.

3.5 Sodium nitrite solution, 20 g/l, freshly prepared.

3.6 Sulphuric acid, ρ 1,26 g/ml.

Dilute 250 ml of sulphuric acid, ρ 1,84 g/ml, with water, while cooling, and make up to 1 000 ml.

3.7 Manganese standard solution, 1 g/l (1 ml contains 1 mg of manganese) prepared from manganese metal as follows.

3.7.1 Free the electrolytic manganese used to prepare the solution from any surface oxide which may be present by placing a few grams of metal in a 250 ml glass beaker containing 60 to 80 ml of sulphuric acid (3.6) and about 100 ml of water. Stir and after a few minutes, decant the acid solution and pour water into the beaker. Repeat the decantation and washing with water several times. Then place the manganese metal in acetone and stir. Decant the acetone, dry the metal in a hot air oven at 100 °C for about 2 min and allow it to cool in a desiccator.

3.7.2 In a 600 ml tall-form beaker, dissolve 1 ± 0,001 g of electrolytic manganese (purity ⩾ 99,9 %) with 40 ml of sulphuric acid (3.6) and about 80 ml of water. Boil the solution for several minutes. Cool, transfer to a 1 000 ml volumetric flask and make up to volume.

3.8 Manganese standard solution, 0,1 g/l (1 ml contains 0,1 mg of manganese).

Transfer 100 ml of the manganese standard solution (3.7) to a 1 000 ml volumetric flask and make up to the mark with water.

4 APPARATUS

4.1 Ordinary laboratory equipment, particularly 100 ml **volumetric flasks** and 300 ml **conical flasks,** and

4.2 Spectrophotometer.

1) This solution attacks glass to a limited extent; it should be stored temporarily in a plastics container. Long term storage is not recommended.

2) Dilution in the cold with this solution does not give rise to any precipitation of tin. It makes it possible to avoid attack of the glass of the colorimetric cells. In spite of this precaution, it is recommended that the solutions should not be allowed to remain in the cells.

5 SAMPLING

Follow the procedure given in ISO... .[1)]

6 PROCEDURE

6.1 Preparation of the calibration curve

6.1.1 Into a series of seven 300 ml conical flasks introduce approximately the volumes of water and exactly the volumes of manganese standard solution (3.8) shown in the following table :

Water volume	Manganese standard solution (3.8)		Corresponding manganese content, % for a test portion of		
	Volume	Mass of Mn contained			
ml	ml	mg	0,400 g	0,080 g	0,032 g
20	0	0	0	0	0
19	1	0,1	0,025	0,125	0,3125
18	2	0,2	0,05	0,250	0,625
16	4	0,4	0,1	0,5	1,25
14	6	0,6	0,15	0,75	1,875
10	10	1	0,25	1,25	3,125
0	20	2	0,50	2,50	6,25

Add 50 ml of the attack reagent (3.2). Boil for 5 min in order to eliminate any oxides of nitrogen which may be present.

6.1.2 Introduce 5 ml of potassium periodate solution (3.4) into the boiling solution. Maintain at boiling for 5 min then immerse the flasks in a boiling water bath for 20 min. Cool and transfer to a 100 ml volumetric flask. For rinsing and making up to the mark, use the dilution solution (3.3). Mix.

6.1.3 Carry out the spectrophotometric measurement at the maximum of the absorption curve in 1, 2 or 4 cm cells[2)] (wavelength generally in the neighbourhood of 530 nm) in comparison with a background colour formed by pouring into a colorimetric cell first a drop of sodium nitrite solution, then one or other of the coloured samples.

Plot the calibration curves.

6.2 Test portion

$m = 0{,}4 \pm 0{,}001$ g.

6.3 Blank test

The blank test, due to the reagents, is normally insignificant. In any case, if the operations are carried out with the same lot of products, the same quantities of reagents being used for all the calibration tests and those on the test portions, the blank test is automatically compensated.

6.4 Determination

6.4.1 *Attack*

Attack the test portion with 50 ml of the attack reagent (3.2). Warm, if necessary, to accelerate the attack. When dissolution is complete, add 20 ml of water. Boil for 5 min to remove the oxides of nitrogen.

6.4.1.1 Contents equal to or less than 0,5 %.

Proceed as indicated in 6.1.2.

6.4.1.2 Contents between 0,5 and 2,5 %.

Cool and transfer to a 100 ml volumetric flask. Make up to volume. Transfer a 20 ml aliquot to a 300 ml conical flask, then add 40 ml of the attack reagent (3.2) and 10 ml of water in order to create the same conditions of dilution as in the preceding case.

Boil for 5 min and proceed as described in 6.1.2.

6.4.1.3 Contents between 2 and 6 %.

Cool and transfer to a 250 ml volumetric flask. Make up to volume. Transfer a 20 ml aliquot to a 300 ml conical flask, then add 46 ml of the attack reagent (3.2).

Boil for 5 min and proceed as described in 6.1.2.

6.4.2 *Spectrophotometric measurements*

Measure the absorbance at the maximum of the absorption curve in 1, 2 or 4 cm cells in comparison with the background colour obtained by decolorizing the coloured samples with a drop of sodium nitrite (see 6.1.3).

7 EXPRESSION OF RESULTS

Deduce from the calibration curve the manganese content, as a percentage, in the sample.

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 regarded as optional.

1) In preparation.

2) The solution containing 1 mg of manganese has an absorbance of about 0,5 in a 1 cm cell.