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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.

International Standard ISO 2543 was drawn up by Technical Committee ISO/TC 26, *Copper and copper alloys*, and circulated to the Member Bodies in August 1971.

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.

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-aluminums 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 $1 \pm 0,001$ g of electrolytic manganese (purity $\geq 99,9$ %) 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.