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**INTERNATIONAL STANDARD**



**2214**

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**Boric acid, boric oxide and *Disodium* tetraborates for industrial use — Determination of manganese content — Formaldehyde oxime photometric 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 2214 was drawn up by Technical Committee ISO/TC 47, *Chemistry*.

It was approved in August 1971 by the Member Bodies of the following countries:

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

No Member Body expressed disapproval of the document.

# Boric acid, boric oxide and Disodium tetraborates for industrial use – Determination of manganese content – Formaldehyde oxime photometric method

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a formaldehyde oxime method for the photometric determination of the manganese content of boric acid, boric oxide and disodium tetraborates for industrial use.

## 2 PRINCIPLE

Removal of the boron by evaporation with methanol and separation of manganese and other metals from the residue, by extraction with chloroform, as their diethyldithiocarbamates. Wet destruction of the diethyldithiocarbamates with a sulphuric acid/hydrogen peroxide mixture. Removal of any interfering iron by extraction as the thiocyanate.

Formation of the coloured manganese formaldehyde oxime complex in alkaline solution and photometric measurement at a wavelength of approximately 450 nm.

## 3 REAGENTS

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

### 3.1 Organic extraction solvent

Mix equal volumes of amyl alcohol (mixed isomers) and amyl acetate.

### 3.2 Chloroform, redistilled.

### 3.3 Methanol, redistilled.

**3.4 Hydrochloric acid**,  $\rho$  1.19 g/ml approximately, 38 % (m/m) solution or approximately 12 N.

**3.5 Ammonia solution**,  $\rho$  0.88 g/ml approximately, 35 % (m/m) solution or approximately 18 N.

**3.6 Sulphuric acid**, approximately 16 N solution.

**3.7 Sodium hydroxide**, approximately 6 N solution.

**3.8 Ammonium thiocyanate**, 100 g/l solution.

**3.9 Formaldehyde oxime hydrochloride**, 60 g/l solution.

**3.10 Hydrogen peroxide**, 500 g/l solution.

**3.11 Hydroxylammonium chloride**, 100 g/l solution.

NOTE – Solid hydroxylammonium chloride should not be allowed to come into contact with the skin. The solution shall be dispensed from a burette or safety pipette.

**3.12 Potassium sodium tartrate**, 100 g/l solution.

**3.13 Sodium diethyldithiocarbamate**, 10 g/l solution.

Dissolve 1 g of sodium diethyldithiocarbamate in 100 ml of water and filter before use.

**3.14 Manganese standard solution**, containing 0.10 g/l of Mn.

Measure by means of a burette 45.51 ml of exactly 0.1 N potassium permanganate solution into a 250 ml conical flask.

NOTE – If the normality of the potassium permanganate solution used is not exactly 0.1 N, use a volume of solution calculated to be equivalent to 45.51 ml of 0.1 N solution.

Add to the flask, drop by drop, a saturated solution of sulphur dioxide until the contents are clear and colourless. Boil the solution to remove excess sulphur dioxide and then cool. Transfer quantitatively to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 100  $\mu$ g of manganese.

**3.15 Manganese standard solution**, containing 0.01 g/l of Mn.

Transfer 10.0 ml of the manganese standard solution (3.14) to a 100 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 10  $\mu$ g of manganese.

Prepare this solution just before use.

**3.16 Cresol red**, 0.2 g/l solution.