International Standard



5934

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Crude sodium borates for industrial use — Determination of alkali-soluble copper and manganese contents — Zinc bis(dibenzyldithiocarbamate) and formaldehyde oxime photometric methods

Borates de sodium bruts à usage industriel — Dosage du cuivre et du manganèse solubles en milieu alcalin — Méthodes photométriques au bis(dibenzyldithiocarbamate) de zinc et à la formaldéhyde oxime

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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:

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The member body of the following country expressed disapproval of the document on technical grounds:

Netherlands

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

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Crude sodium borates for industrial use — Determination of alkali-soluble copper and manganese contents — Zinc bis(dibenzyldithiocarbamate) and formaldehyde oxime photometric methods

1 Scope and field of application

This International Standard specifies zinc bis(dibenzyldithiocarbamate) and formaldehyde oxime photometric methods for the successive determinations of the alkali-soluble copper and manganese contents in crude sodium borates for industrial use.

The methods are applicable to products in which the alkalisoluble copper and manganese contents exceed 0,25 and 0,5 mg/kg respectively.

Reference

ISO 2217, Crude sodium borates for industrial use - Determination of matter insoluble in alkaline medium and preparation of test solutions.

Principle

3.1 Determination of copper content

Formation of a coloured complex by reaction of zinc bis(dibenzyldithiocarbamate) with the copper contained in an aliquot portion of solution A (see ISO 2217).

Extraction of this complex with carbon tetrachloride and photometric measurement of the coloured complex in the organic phase at a wavelength of about 435 nm.

Retention of the aqueous phase for the determination of manganese.

3.2 Determination of manganese content

Addition of potassium sodium tartrate, to assist in the subsequent separation of manganese, to the aqueous solution from the copper determination and then of sodium diethyldithiocarbamate, at pH 6 to 7, to form manganese bis(diethyldithiocarbamate) (and to react similarly with other metals).

Extraction of these complexes with carbon tetrachloride followed by evaporation of the organic phase, in the presence of hydrochloric acid, to destroy the diethyldithiocarbamates.

Addition of ascorbic acid to suppress interference by iron.

Addition of formaldehyde oxime in alkaline medium, to form the manganese-formaldehyde oxime coloured complex and photometric measurement at a wavelength of about 450 nm.

Reagents

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

Carbon tetrachloride.

WARNING - Carbon tetrachloride is toxic. Avoid breathing the vapour and contact with eyes. Carry out all operations involving its use in a fume cupboard, away from intense heat.

- Hydrochloric acid, approximately 73 g/l solution.
- Sodium hydroxide, approximately 80 g/l solution.
- Ascorbic acid, 200 g/l solution, freshly prepared.
- 4.5 Potassium sodium tartrate, 200 g/l solution.

Discard the solution if it becomes cloudy.

4.6 Zinc bis(dibenzyldithiocarbamate), 0,5 g/l solution in the carbon tetrachloride (4.1).

Discard the solution if it becomes cloudy.

Sodium diethyldithiocarbamate, 20 g/l solution.

Discard this solution after 1 week.

4.8 Formaldehyde oxime, approximately 5 mol/l solution.

Dissolve 350 g of hydroxylammonium chloride in about 500 ml of water, add 375 ml of 40 % (V/V) formaldehyde solution and dilute to 1 000 ml.

Discard the solution if it becomes cloudy.