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INTERNATIONAL STANDARD 2217

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Crude sodium borates for industrial use — Determination of matter insoluble in alkaline medium and preparation of test solutions

Borates de sodium bruts à usage industriel — Détermination des matières insolubles en milieu alcalin, et préparation des solutions d'essai

First edition — 1975-02-15

UDC 661.652 : 541.8 **Ref. No. ISO 2217-1975 (E)**

Descriptors : sodium borates, chemical analysis, determination of content, insoluble matter.

Price based on 2 pages

FOREWORD

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International Standard ISO 2217 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in February 1972.

It has been approved by the Member Bodies of the following countries :

Austria	India	South Africa, Rep. of
Belgium	Ireland	Spain
Chile	Israel	Switzerland
Czechoslovakia	Italy	Thailand
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No Member Body expressed disapproval of the document.

Printed in Switzerland

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Crude sodium borates for industrial use — Determination of matter insoluble in alkaline medium and preparation of test solutions

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the methods to apply in the analysis of crude sodium borates to determine the matter insoluble in alkaline medium and to prepare the solutions intended for the determination of impurities soluble under these conditions.

2 PRINCIPLE

Solution of a test portion in a weak excess of sodium hydroxide solution.

Filtration, washing of the insoluble matter, drying and weighing.

Dilution and acidification of the filtrate to obtain the test solution (solution A).

Preparation of an identical solution but without the test portion with a view to the carrying out of blank tests (solution B).

3 REAGENTS

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

3.1 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution or approximately 12 N solution.

3.2 Sodium hydroxide, ρ approximately 1,08 g/ml, about 7,5 % (*m/m*) solution or approximately 2 N solution.

3.3 Acetone.

3.4 pH indicator paper (universal)

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Fine grain filter papers (for fine precipitates), 110 mm diameter approximately.

4.2 Vacuum filtration apparatus, consisting of a Buchner funnel, approximately 90 mm internal diameter, fitted with a weighed filter paper (4.1), the perimeter of which extends up the walls of the funnel, and a filtration flask connected to a water pump and a guard flask.

NOTE — Apparatus which contacts the test solution shall not contain silicon or aluminium when these are to be determined subsequently. Where possible, such apparatus, particularly the beakers used, should be made of polypropylene, stainless steel or other suitable material.

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0,01 g, in a 500 ml beaker, 10 ± 0,1 g of the finely ground and mixed test sample.

5.2 Determination of matter insoluble in alkaline medium

Add 50 ml of the sodium hydroxide solution (3.2) to the beaker, warm to about 80 °C and stir for 5 min using a glass stirrer, flattened at one end, to break up the particles.

Wash a filter paper (4.1) with acetone (3.3) and, after the acetone has evaporated, dry in an oven at 110 °C for 30 min. Allow to cool in a desiccator for 15 min, weigh to the nearest 0,001 g and place in the filtration apparatus (4.2). Moisten the paper and operate the filter pump so as to maintain a slight vacuum in the filter flask. Filter the contents of the beaker using as much of the paper surface as possible, to accelerate the filtration, without allowing the liquid to go above the edge of the paper.

Wash the beaker, the insoluble matter and the filter with several small quantities of water, each time rinsing the perimeter of the filter, until the filtrate is no longer alkaline, as shown by the indicator paper (3.4).

Quantitatively transfer the filtrate into a 500 ml one-mark volumetric flask and re-assemble the filtration apparatus.

Wash the insoluble matter and the filter three times, with 10 ml portions of the acetone (3.3) each time, removing the rinse by applying the vacuum.

Remove the filter paper from the Buchner funnel and place it and its contents on a clock-glass in an oven at 110 °C for 30 min. Allow to cool for 15 min in a desiccator and then weigh the filter paper and insoluble matter to the nearest 0,001 g.

5.3 Preparation of solutions intended for determination of impurities soluble in alkaline medium and for the blank tests

5.3.1 *Solution intended for determinations* (solution A)

Add 20 ml of the hydrochloric acid solution (3.1) to the filtrate in the 500 ml one-mark volumetric flask (see 5.2), cool the solution to ambient temperature, dilute to the mark and mix.

This is solution A, from which are taken the various aliquots necessary for the determination of impurities soluble in alkaline medium.

5.3.2 *Solution intended for blank tests* (solution B)

Introduce into a 300 ml flask 5 ml of the hydrochloric acid solution (3.1), evaporate to dryness, cool, dissolve the contents in water, and transfer quantitatively to a 500 ml one-mark volumetric flask. Add 50 ml of the same sodium hydroxide solution (3.2) used to dissolve the test portion. Add 250 ml of water and 15 ml of the hydrochloric acid solution (3.1). Cool to ambient temperature, dilute to the mark and mix.

This is solution B, from which are taken the various aliquots necessary to carry out blank tests during the determination of different impurities.

6 EXPRESSION OF RESULTS

The content of matter insoluble in alkaline medium, expressed as a percentage by mass, is given by the formula

$$\frac{m_1 - m_2}{m_0} \times 100$$

where

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

m_1 is the mass, in grams, of the filter paper and insoluble matter after drying;

m_2 is the mass, in grams, of the dried filter paper alone.

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.