# INTERNATIONAL STANDARD 3121



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## Boric acid, boric oxide and *di*sodium tetraborates for industrial use — Determination of chloride content — Mercurimetric method

*Acide borique, oxyde borique et tétraborates disodiques à usage industriel — Dosage des chlorures — Méthode mercurimétrique*

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## FOREWORD

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International Standard ISO 3121 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in April 1973.

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

| | | |
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| Austria | India | Spain |
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No Member Body expressed disapproval of the document.



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# Boric acid, boric oxide and *di*sodium tetraborates for industrial use — Determination of chloride content — Mercurimetric method

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a mercurimetric titration method for the determination of the chloride content of boric oxide, boric acid and *di*sodium tetraborates for industrial use.

## 2 PRINCIPLE

Titration of the $Cl^-$ ion with standard volumetric mercury(II) nitrate solution, using diphenylcarbazone as indicator.

## 3 REAGENTS

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

**3.1 Nitric acid,** $\rho$ approximately 1,40 g/ml, about 68 % (*m/m*) solution or approximately 14 N, of which the chloride content, expressed as chlorine, is not greater than 1 mg/kg.

**3.2 Nitric acid,** approximately 2 N solution.

**3.3 Sodium hydroxide,** approximately 2 N solution.

**3.4 Sodium chloride,** 0,05 N standard reference solution.

Weigh, to the nearest 0,000 1 g, 2,922 1 g of sodium chloride, previously heated for 1 h at 500 °C and allowed to cool in a desiccator. Place in a beaker, dissolve in water, transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

**3.5 Standard end-point matching solution**

Pour 350 ml of water into a 500 ml conical flask, add 3 drops of the bromophenol blue solution (3.7), and the nitric acid solution (3.2), drop by drop, until the colour changes from blue to yellow. Add an excess of 3 drops of this acid, 0,5 to 1,0 ml of the diphenylcarbazone solution (3.8) and the volume of the mercury(II) nitrate solution (3.6) (from a burette) necessary to change the colour of the solution from yellow to mauve (about 2 drops).

Prepare this standard solution immediately before use.

**3.6 Mercury(II) nitrate,** 0,05 N standard volumetric solution.

**3.6.1** *Preparation of the solution*

Dissolve 5,43 ± 0,01 g of mercury(II) oxide (HgO) in 10 ml of the nitric acid solution (3.1) in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Standardize this solution following the procedure specified in 3.6.2.

**3.6.2** *Standardization of the solution*

Pipette 40,0 ml of the standard reference sodium chloride solution (3.4) into a 500 ml conical flask. Add 160 ml of water and 3 drops of the bromophenol blue solution (3.7). Add the nitric acid solution (3.2), drop by drop, until the colour of the indicator changes from blue to yellow.

Add 3 drops of this nitric acid solution in excess, followed by a volume of the diphenylcarbazone solution (3.8) identical to that added for the standard end-point matching solution (3.5).

Titrate with the mercury(II) nitrate solution to be standardized (3.6.1) until the colour matches the mauve of the standard end-point matching solution (3.5) and deduct the volume of the mercury(II) nitrate solution (3.6.1) added during the preparation of the standard end-point matching solution (about 2 drops).

The volume is 40,0 ml if the standard volumetric solution (3.6) is exactly 0,05 N.

**3.7 Bromophenol blue,** 1 g/l ethanolic solution.

Dissolve 0,1 g of bromophenol blue in 100 ml of 95 % (*V/V*) ethanol.

**3.8 Diphenylcarbazone,** 5 g/l ethanolic solution.

Dissolve 0,5 g of diphenylcarbazone in 100 ml of 95 % (*V/V*) ethanol.

Store this solution in a refrigerator and replace it when it no longer gives a sharp colour change.

## 4 APPARATUS

Ordinary laboratory apparatus.

## 5 PROCEDURE

### 5.1 Test portion

Weigh, to the nearest 0,01 g, approximately 10 g of the test sample into a 500 ml conical flask.

### 5.2 Determination

Add 350 ml of water to the test portion (5.1) and swirl to dissolve. Add 3 drops of the bromophenol blue solution (3.7), followed by the nitric acid solution (3.1) until the colour of the indicator changes from blue to yellow.

Add the sodium hydroxide solution (3.3) until the solution is again blue.

Finally add the nitric acid solution (3.2), drop by drop, until the colour of the indicator changes from blue to yellow.

Add 3 drops of this nitric acid solution in excess followed by a volume of the diphenylcarbazone solution (3.8) identical to that added for the standard end-point matching solution (3.5) and titrate with the standard volumetric mercury(II) nitrate solution (3.6) until the colour of the solution matches the mauve of the standard end-point matching solution (3.5).

## 6 EXPRESSION OF RESULTS

The chloride content, expressed as milligrams of chlorine (Cl) per kilogram, is given by the formula :

$$(V_0 - V_1) \times 1\,000 \times \frac{1\,000}{m} \times 0{,}001\,773 = \frac{1\,773\,(V_0 - V_1)}{m}$$

where

$V_0$ is the volume, in millilitres, of the standard volumetric mercury(II) nitrate solution (3.6) used for the titration;

$V_1$ is the volume, in millilitres, of the standard volumetric mercury(II) nitrate solution (3.6) used in the preparation of the standard end-point matching solution (3.5);

$m$ is the mass, in grams, of the test portion (5.1);

0,001 773 is the mass, in grams, of chlorine corresponding to 1 ml of exactly 0,05 N mercury(II) nitrate solution.

NOTE — If the concentration of the standard volumetric solution used is not exactly as specified in the list of reagents, an appropriate correction should be made.

## 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.

ANNEX

## ISO PUBLICATIONS RELATING TO (A) BORIC ACID, (B) BORIC OXIDE, (C) *D*/SODIUM TETRABORATES, (D) SODIUM PERBORATES, AND (E) CRUDE SODIUM BORATES, FOR INDUSTRIAL USE

| Applicability | |
|---|---|
| A | ISO 1914 — Determination of boric acid content — Volumetric method. |
| B | ISO 1915 — Determination of boric oxide content — Volumetric method. |
| C | ISO 1916 — Determination of sodium oxide and boric oxide contents and loss on ignition. |
| D | ISO 1917 — Determination of sodium oxide, boric oxide and available oxygen contents — Volumetric methods. |
| A B C E | ISO 1918 — Determination of sulphur compounds — Volumetric method. |
| A B C | ISO 2214 — Determination of manganese content — Formaldehyde oxime photometric method. |
| A B C | ISO 2215 — Determination of copper content — Zinc dibenzyldithiocarbamate photometric method. |
| E | ISO 2216 — Determination of sodium oxide and boric oxide contents — Volumetric method. |
| E | ISO 2217 — Determination of matter insoluble in alkaline medium and preparation of test solutions. |
| E | ISO 2218 — Determination of loss in mass after heating at 900 °C. |
| E | ISO 2760 — Determination of total aluminium content — Titrimetric method. |
| E | ISO 2761 — Determination of total titanium content — Photometric method. |
| D | ISO 3118 — Determination of particle size distribution by mechanical sieving. |
| A B C | ISO 3119 — Determination of chromium content — Diphenylcarbazide photometric method. |
| C E | ISO 3120 — Determination of water content — Gravimetric method. |
| A B C | ISO 3121 — Determination of chloride content — Mercurimetric method. |
| A B C D E | ISO 3122 — Determination of iron content — 2,2′-Bipyridyl photometric method. |
| D | ISO 3123 — Determination of rate of solution — Conductivity method. |
| E | ISO 3124 — Determination of iron soluble in alkaline medium — 2,2′-Bipyridyl photometric method. |
| E | ISO 3125 — Determination of aluminium soluble in alkaline medium — EDTA titrimetric method. |
| D | ISO 3424 — Determination of bulk density. |