

International Standard



5935

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**Crude sodium borates for industrial use —
Determination of total and alkali-soluble silica contents —**

Molybdsilicate spectrometric method

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 5935 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in January 1983.

It has been approved by the member bodies of the following countries:

| | | |
|----------------|-------------|-----------------------|
| Austria | Hungary | Portugal |
| Belgium | India | South Africa, Rep. of |
| China | Italy | Switzerland |
| Czechoslovakia | New Zealand | United Kingdom |
| France | Nigeria | USSR |
| Germany, F.R. | Poland | |

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

Australia

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

Crude sodium borates for industrial use — Determination of total and alkali-soluble silica contents — Molybdosilicate spectrometric method

1 Scope and field of application

This International Standard specifies a molybdosilicate spectrometric method for the determination of the total and alkali-soluble silica contents of crude sodium borates for industrial use.

The method is applicable to products having total silica contents in the range 0,4 to 4,0 % (*m/m*), and alkali-soluble silica contents in the range 0,01 to 0,05 % (*m/m*).

2 Principle

Preparation of test solutions by

- fusion of a test portion with sodium carbonate for the determination of the total silica content, and
- dissolution of the alkali-soluble part of a test portion in sodium hydroxide solution for the determination of the alkali-soluble silica content.

Formation of the molybdosilicate, followed by spectrometric measurement, at a wavelength of about 400 nm.

3 Reagents and materials

During the analysis, use only reagents of recognized analytical grade and only double-distilled water stored in a polyethylene bottle.

3.1 Sodium carbonate, anhydrous.

3.2 Sulfuric acid, approximately 36,5 % (*m/m*) solution prepared by diluting sulfuric acid, approximately 96 % (*m/m*) solution, 1 → 4.

Store in a polyethylene bottle.

3.3 Ammonium molybdate tetrahydrate [Hexa-ammonium heptamolybdate tetrahydrate] $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}]$, 80 g/l solution.

Store in a polyethylene bottle. Discard the solution if it becomes cloudy.

3.4 Ammonia

, approximately 87,5 g/l solution.

Store in a polyethylene bottle.

3.5 Sodium hydroxide

, approximately 80 g/l solution.

Store in a polyethylene bottle.

3.6 Mannitol

, approximately 100 g/l solution.

Store in a polyethylene bottle.

3.7 Silica

, standard solution corresponding to 0,200 g of silica (SiO_2) per litre.

Fuse 0,200 g of pure silica with 2 g of the sodium carbonate (3.1) in a platinum crucible until a clear melt is obtained. Cool, dissolve the melt in water, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Transfer the solution immediately to a polyethylene bottle. Discard after 1 month.

1 ml of this standard solution contains 0,200 mg of SiO_2 .

3.8 Phenolphthalein

, 1 g/l solution in 95 % (V/V) ethanol.

3.9 pH indicator paper

 (universal).

4 Apparatus

Apparatus which comes into contact with the test solution shall be free from silica (for example of polyethylene) to avoid any contamination. The volumetric flasks may, however, be of glass, provided that they are not scratched or matt.

4.1 Spectrometer with selectors for continuous wavelength variation, fitted with cells having optical path lengths of 4 cm or 5 cm for the alkali-soluble silica content and 1 cm for the total silica content, or

4.2 Spectrometer with selectors for discontinuous wavelength variation, fitted with similar cells.

4.3 pH meter, with glass measurement electrode and calomel reference electrode.

ISO 5935-1984 (E)

4.4 Platinum crucible, of capacity approximately 40 ml, with lid.

4.5 Platinum dish, of capacity approximately 80 ml.

4.6 Fine-grain filter paper (for fine precipitates), of diameter approximately 110 mm.

4.7 Vacuum filtration apparatus, comprising a Buchner funnel of approximately 90 mm internal diameter, fitted with one of the filter papers (4.6), the perimeter of which extends up the wall of the funnel, and a filtration flask connected to a water pump and a guard flask.

5 Procedure

5.1 Test portion

5.1.1 Determination of total silica content

Weigh, to the nearest 0,005 g, approximately 0,5 g of the finely ground and mixed laboratory sample into the platinum crucible (4.4).

5.1.2 Determination of alkali-soluble silica content

Weigh, to the nearest 0,01 g, into a 500 ml polyethylene beaker, $10 \pm 0,1$ g of the finely ground and mixed laboratory sample.

5.2 Blank tests

Carry out blank tests at the same time as the determinations using the same procedure and the same quantities of all the reagents used for the determinations, but omitting the test portions.

5.3 Preparation of calibration graph

5.3.1 Preparation of standard colorimetric solutions and colour development

Into a series of six 100 ml beakers, transfer the volumes of the standard silica solution (3.7) indicated in the following table and dilute each to about 40 ml with water.

Table

| Standard silica solution (3.7), ml | | Corresponding mass of silica, mg | |
|------------------------------------|-------------------------------|----------------------------------|-------------------------------|
| Total silica content | Alkali-soluble silica content | Total silica content | Alkali-soluble silica content |
| 0 * | 0 * | 0 * | 0 * |
| 1,0 | 0,50 | 0,200 | 0,100 |
| 3,0 | 1,00 | 0,600 | 0,200 |
| 4,0 | 1,50 | 0,800 | 0,300 |
| 7,0 | 2,00 | 1,400 | 0,400 |
| 10,0 | 2,50 | 2,000 | 0,500 |

* Blank test of reagents for calibration.

Treat the contents of each beaker as follows:

Dip the electrodes of the pH meter (4.3) into the solution and adjust the pH to 1,4 by adding the sulfuric acid solution (3.2) drop by drop. Add 10 ml of the ammonium molybdate solution (3.3) and readjust the pH to 1,4 by means of the sulfuric acid solution (3.2). Transfer the solution quantitatively to a 100 ml one-mark volumetric flask and allow to stand for 10 min. Add 10 ml of the mannitol solution (3.6) and dilute to the mark with water. Mix thoroughly and allow to stand for between 5 min and 15 min at room temperature.

5.3.2 Spectrometric measurement

Using the spectrometer (4.1) at a wavelength of approximately 400 nm, or the spectrometer (4.2) fitted with a suitable filter, and cells of appropriate path length (see 4.1 and 4.2), measure the absorbance of each solution after having adjusted the instrument to zero absorbance against water.

5.3.3 Plotting the graph

Deduct the absorbance of the blank test of reagents for calibration from that of the standard colorimetric solutions (see 5.3.1). Plot a graph having, for example, the masses, in milligrams, of silica in the standard colorimetric solutions as abscissae and the corresponding values of absorbance as ordinates.

5.4 Determination

5.4.1 Preparation of test solution for determination of total silica content

Add 1 g of the sodium carbonate (3.1) to the platinum crucible (4.4) containing the test portion (5.1.1), mix, cover the crucible with its lid, heat the contents carefully and maintain just at the fusion point until a clear melt is obtained. Cool, remove the lid and rinse it with 25 ml of hot water, collecting the rinse in the crucible. Stir until the melt is completely dissolved and transfer quantitatively to the platinum dish (4.5). Add 1 drop of the phenolphthalein solution (3.8) as indicator and neutralize the solution by addition of the sulfuric acid solution (3.2). Add a further 2 ml of this acid, boil gently to remove carbon dioxide and cool. Transfer the solution quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark and mix. Immediately transfer 10,0 ml of this solution to a 250 ml beaker and dilute to about 50 ml with water. This solution is the test solution for colour development (see 5.4.3).

5.4.2 Preparation of test solution for determination of alkali-soluble silica content

Add 50 ml of the sodium hydroxide solution (3.5) to the beaker used in 5.1.2, warm to about 80 °C and stir for 5 min, using a PTFE stirrer, to break up the particles. Place one of the filter papers (4.6) in the filtration apparatus (4.7), 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 until the filtrate is no longer

alkaline as shown by the indicator paper (3.9). Transfer the filtrate quantitatively to a 500 ml one-mark volumetric flask. Add 1 drop of the phenolphthalein solution (3.8) as indicator and neutralize the solution by addition of the sulfuric acid solution (3.2). Add a further 2 ml of this acid, dilute to the mark with water and mix. Immediately transfer 50,0 ml of this solution to a 250 ml beaker. This solution is the test solution for colour development (see 5.4.3).

5.4.3 Colour development

Dip the electrodes of the pH meter (4.3) into the freshly prepared test solution (5.4.1 or 5.4.2) and adjust the pH to 1,4 adding, drop by drop, either the sulfuric acid solution (3.2) or the ammonia solution (3.4), as appropriate. Follow the procedure specified in the last paragraph of 5.3.1, starting at the instruction "Add 10 ml of the ammonium molybdate solution (3.3)...".

5.4.4 Spectrometric measurement

Measure the absorbance of the solution following the procedure specified in 5.3.2.

6 Expression of results

By reference to the calibration graph (5.3.3), determine the masses of silica corresponding to the absorbances of the test solution and the blank test solution.

6.1 Total silica content

The total silica content, expressed as a percentage by mass of SiO_2 , is given by the formula

$$(m_1 - m_2) \times \frac{1}{1\,000} \times \frac{100}{10} \times \frac{100}{m_0}$$

$$= \frac{m_1 - m_2}{m_0}$$

where

m_0 is the mass, in grams, of the test portion (5.1.1);

m_1 is the mass, in milligrams, of silica found in the test solution (5.4.1);

m_2 is the mass, in milligrams, of silica found in the blank test solution (5.2).

6.2 Alkali-soluble silica content

The alkali-soluble silica content, expressed as a percentage by mass of SiO_2 , is given by the formula

$$(m_4 - m_5) \times \frac{1}{1\,000} \times \frac{500}{50} \times \frac{100}{m_3}$$

$$= \frac{m_4 - m_5}{m_3}$$

where

m_3 is the mass, in grams, of the test portion (5.1.2);

m_4 is the mass, in milligrams, of silica found in the test solution (5.4.2);

m_5 is the mass, in milligrams, of silica found in the blank test solution (5.2).

7 Test report

The test report shall include the following particulars:

- an identification of the sample;
- the reference of the method used;
- the results and the method of expression used;
- any unusual features noted during the determination;
- any operation not included in this International Standard or regarded as optional.

Annex

ISO publications relating to crude sodium borates for industrial use

- ISO 1918 — Determination of sulfur compounds — Volumetric method.
- ISO 2216 — Determination of sodium oxide and boric oxide contents — Volumetric method.
- ISO 2217 — Determination of matter insoluble in alkaline medium and preparation of test solutions.
- ISO 2218 — Determination of loss in mass after heating at 900 °C.
- ISO 2760 — Determination of total aluminium content — Titrimetric method.
- ISO 2761 — Determination of total titanium content — Photometric method.
- ISO 3120 — Determination of water content — Gravimetric method.
- ISO 3122 — Determination of iron content — 2,2'-Bipyridyl photometric method.
- ISO 3124 — Determination of iron soluble in alkaline medium — 2,2'-Bipyridyl photometric method.
- ISO 3125 — Determination of aluminium soluble in alkaline medium — EDTA titrimetric method.
- ISO 5933 — Determination of total nickel content of boric acid, boric oxide and *d*/sodium tetraborates and the alkali-soluble nickel content of crude sodium borates — Fural α -dioxime photometric method.
- ISO 5934 — Determination of alkali soluble copper and manganese contents — Zinc bis(dibenzylthiocarbamate) and formaldehyde oxime photometric methods.
- ISO 5935 — Determination of total and alkali-soluble silica contents — Molybdosilicate spectrometric method.
- ISO 5936 — Determination of carbonate content — Gravimetric method.
- ISO 6918 — Determination of total and alkali-soluble calcium and magnesium contents — Flame atomic absorption spectrometric method.
- ISO 6920 — Determination of total and alkali-soluble calcium and magnesium contents — Titrimetric method.