

International Standard



5936

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Crude sodium borates for industrial use — Determination of carbonate content — Gravimetric method

Borates de sodium bruts à usage industriel — Dosage des carbonates — Méthode gravimétrique

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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 5936 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in October 1978.

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

Australia	Germany, F. R.	Poland
Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Israel	Switzerland
Bulgaria	Italy	Thailand
China	Korea, Rep. of	United Kingdom
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No member body expressed disapproval of the document.

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 carbonate content — Gravimetric method

1 Scope and field of application

This International Standard specifies a gravimetric method for the determination of the carbonate content of crude sodium borates for industrial use.

2 Principle

Reaction of the carbonate in a test portion with hydrochloric acid, absorption of the released carbon dioxide in soda asbestos and measurement of the increase in mass.

3 Reagents

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

3.1 Soda asbestos, particle size 0,85 to 1,20 mm, self-indicating.

3.2 Calcium sulphate, anhydrous granules of particle size 0,85 to 1,20 mm, self-indicating.

This reagent can be re-activated but it gradually loses its colour through successive regenerations due to the migration of the coloured salt to the interior of the granules.

3.3 Sodium carbonate, anhydrous.

3.4 Chromic-sulphuric acid solution.

Dissolve cautiously $25 \pm 0,5$ g of potassium dichromate in 1 000 ml of the sulphuric acid solution (3.5).

3.5 Sulphuric acid, ρ 1,84 g/ml, about 96 % (m/m) solution.

3.6 Hydrochloric acid, approximately 219 g/l solution, to which has been added 2 drops of 1 g/l methyl orange indicator solution.

4 Apparatus

Ordinary laboratory apparatus and gas evolution and absorption train. It is important that the train be installed as shown in the figure. Its components are as follows.

4.1 Absorption tube, filled with the soda asbestos (3.1), and connected to the dropping funnel (4.2).

Replace the soda asbestos (3.1) before it is half exhausted, as indicated by the colour change.

4.2 Dropping funnel, approximately 25 ml capacity, with a stopcock and stem as shown in the figure.

4.3 Adaptor, to connect the funnel (4.2) and the condenser (4.5) to the reaction flask (4.4).

4.4 Reaction flask, flat-bottomed and of capacity 250 ml. A round-bottomed flask may be used if heated by means of a mantle.

4.5 Condenser, connected to the side limb of the adaptor (4.3). If desired, the Drechsel bottle (4.6.1) may be connected by means of a conical ground-glass joint.

4.6 Drechsel bottles, of capacity 50 ml. The bottles act as traps and prevent liquid being sucked back. Two of the bottles (4.6.1 and 4.6.2) are half-filled with the chromic-sulphuric acid solution (3.4) to remove water and sulphur dioxide, and the third (4.6.3) is half-filled with the sulphuric acid solution (3.5) to prevent access of atmospheric water vapour.

Recharge the bottles (4.6.1 and 4.6.2) after they have used 6 to 7 times, or more frequently if their contents show signs of exhaustion by development of a greenish colour.

4.7 Glass tube, approximately 150 mm in length and 10 mm nominal diameter, filled with the desiccant (3.2) which shall not be packed too tightly. If the desiccant tends to fuse and impede gas flow, glass beads or other inert material should be introduced in the middle of the tube to reduce this tendency.

4.8 Fleming absorber (two piece), with ground glass joints. The upper (smaller) chamber is filled with the desiccant (3.2) to retain the water formed by reaction of the carbon dioxide with the soda asbestos (3.1); the lower chamber is filled with the soda asbestos (3.1).

NOTE — If greater sensitivity is required, the Fleming absorber may be replaced by a device of lesser mass.

4.9 Glass tube, of suitable dimensions to replace the Fleming absorber (4.8) when the latter is not in the apparatus train.

4.10 Source of vacuum.

If a water pump is used, it shall be separated from the rest of the apparatus by means of the large vacuum receiver, of a least 2 000 ml capacity, to prevent water sucking back from the pump. The vacuum shall be controlled, preferably by means of a needle valve.

NOTE — The volume of the air space in the train should be kept to a minimum and the connecting tubing should be kept as short as possible.

4.11 Glass spheres, 2 to 4 mm diameter.

5 Procedure

5.1 Test portion

Depending on the expected carbonate content, weigh, to the nearest 0,1 g, into the flask (4.4), the mass of test sample specified in the following table.

Expected carbonate content % (<i>m/m</i>)	Mass of test portion g
<0,5	25,0
0,5 to 1,0	15,0
> 1,0	7,5

5.2 Determination

Ensure that the funnel (4.2) and its stem have been washed after the previous test until they are acid-free. Open the stopcock of the funnel and aspirate to remove carbon dioxide from the apparatus.

Check the assembled apparatus for leaks as follows. Close the stopcock of the funnel (4.2), evacuate the apparatus until no more bubbles are observed and then stop the aspiration. Examine the liquid in the Drechsel bottles (4.6.1, 4.6.2 and 4.6.3). If no movement occurs, open the stopcock of the funnel slowly so as to restore the pressure within the apparatus to that of the atmosphere and then close it. In the event of movement, find and repair the leak(s) and then retest as above.

Add several of the glass spheres (4.11) to the flask (4.4) and reconnect it to the apparatus. Close the inlet and outlet stopcocks of the Fleming absorber (4.8) and replace it by the glass tube (4.9). Draw a current of air through the train for a few minutes to sweep out all of the carbon dioxide. Regulate the suction, preferably by means of a needle valve, so that 2 to 4 bubbles per second pass through the apparatus.

With its inlet and outlet stopcocks closed, weigh the Fleming absorber (4.8) to the nearest 0,000 2 g.

Remove the glass tube (4.9) from the train, quickly re-insert the weighed Fleming absorber (4.8) and re-open its inlet and outlet stopcocks. Close the stopcock of the funnel (4.2), remove the absorption tube (4.1) and fill the funnel to capacity (to expel air containing carbon dioxide) with the hydrochloric acid solution (3.6). Replace the tube (4.1) and apply suction to the train.

Carefully open the stopcock of the funnel (4.2) and allow all the hydrochloric acid solution (3.6) to run slowly into the reaction flask (4.4). With the funnel stopcock still open allow air to bubble through the train at a rate of 2 to 4 bubbles per second. Heat the solution to boiling. A heating mantle may be used, but is not as easy to control as a burner or hot plate; too rapid boiling is best corrected by removing the heating mantle temporarily.

The solution should be distinctly red after boiling, indicating an excess of the acid. If it is not, allow the flask to cool slightly, close the stopcock of the funnel (4.2) and refill to capacity with the hydrochloric acid solution (3.6). Add the whole of the acid as before, and repeat the procedure from the acid addition.

After 15 min of boiling, close the outlet stopcock and then the inlet stopcock of the Fleming absorber (4.8) (closure in the reverse order will evacuate the absorber). Disconnect the Fleming absorber and reweigh to the nearest 0,000 2 g.

The mass of the absorber at the end of this determination may be used as the initial mass for the next determination if the elapsed time between determinations is less than 4 h.

6 Check test for efficiency of apparatus and procedure

Carry out the following check test whenever there is any question of the analysis giving doubtful results, which might be due to leaks in the apparatus, exhausted desiccant or soda asbestos, low grade reagents or unsatisfactory operation.

Dry approximately 25 g of the anhydrous sodium carbonate (3.3) for 2 h at 105 °C and allow to cool in a desiccator. Weigh $20 \pm 0,001$ g of the dried material in a weighing bottle and transfer it quantitatively to a 500 ml one-mark volumetric flask with cool, freshly-boiled distilled water. Allow to dissolve, dilute to the mark with the same water and mix.

Place 10,0 ml of this solution in the reaction flask (4.4), and follow the procedure specified in 5.2.

The percentage by mass of carbon dioxide, calculated as specified in clause 7, should be $41,5 \pm 0,8$ % (*m/m*).

7 Expression of results

The carbonate content, expressed as carbon dioxide (CO₂) as a percentage by mass, is given by the formula :

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

where

m_0 is the mass, in grams, of the test portion (5.1);

m_1 is the mass, in grams, of the Fleming absorber before the determination;

m_2 is the mass, in grams, of the Fleming absorber after the determination.

8 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.

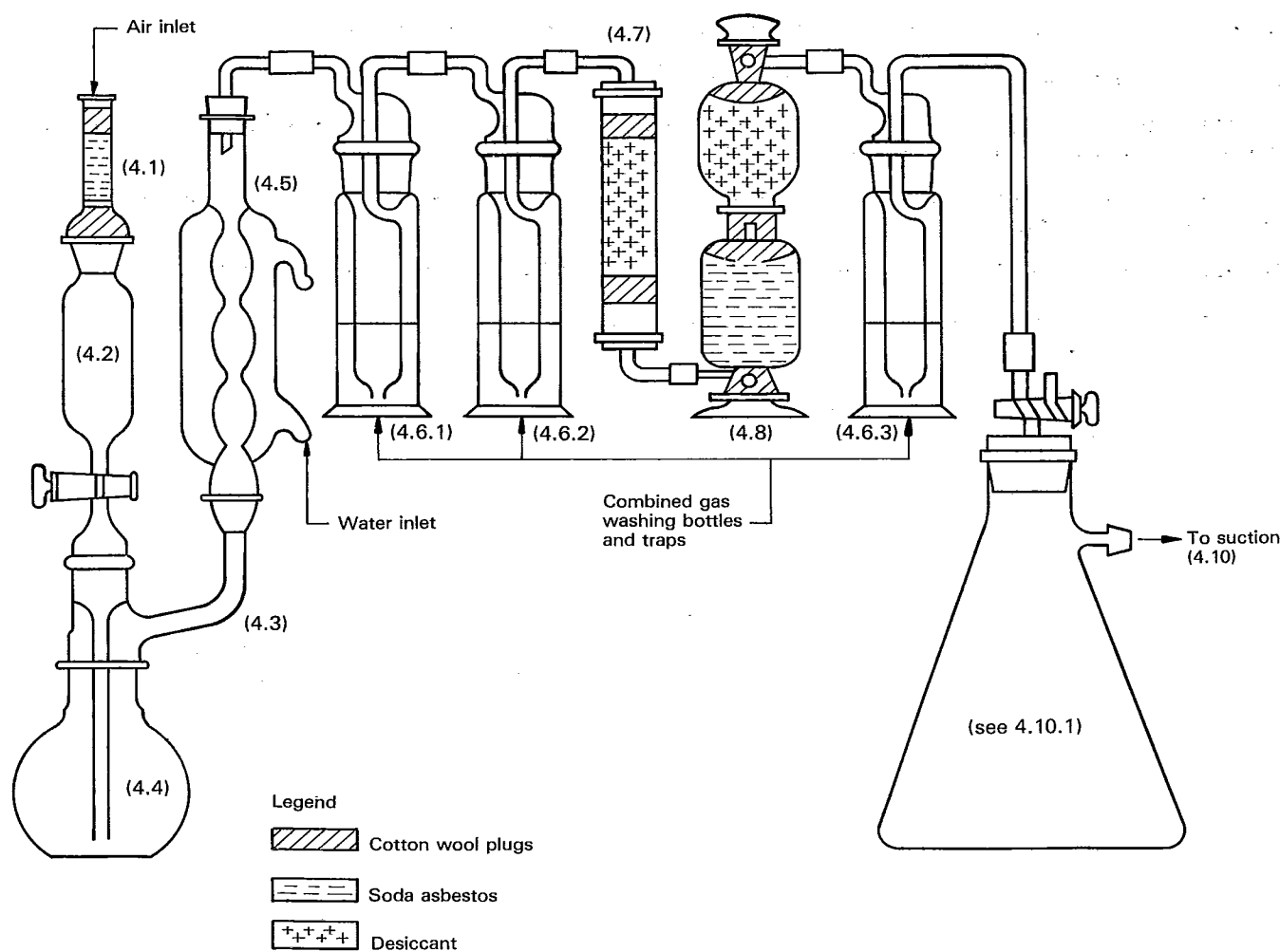


Figure — Apparatus train for the gravimetric determination of carbon dioxide (Schematic representation, not to scale)

Annex

ISO publications relating to crude sodium borates for industrial use

- ISO 1918 — Determination of sulphur 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 silica content and content of silica soluble in alkaline medium — Photometric method.
- ISO 5936 — Determination of carbonate content — Gravimetric method.
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