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Hydrated sodium perborates for industrial use – Determination of sodium oxide, boric oxide and available oxygen contents – Volumetric methods

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FOREWORD

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Hydrated sodium perborates for industrial use — Determination of sodium oxide, boric oxide and available oxygen contents — Volumetric methods

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies volumetric methods for the determination of the sodium oxide, boric oxide and available oxygen contents of hydrated sodium perborates for industrial use.

2 PRINCIPLE

Determination of the sodium oxide content by addition of an excess of a solution of hydrochloric acid and back titration with a standard volumetric solution of sodium hydroxide, using screened methyl red as indicator.

Subsequent titration of the boric oxide with the standard volumetric solution of sodium hydroxide in the presence of mannitol or sorbitol, using phenolphthalein as indicator.

Determination of the available oxygen content by titration in acid solution with a standard volumetric potassium permanganate solution.

3 REACTION

The reaction involved in the determination of available oxygen is the following:

$$2\,MnO_4^- + 6\,H^+ + 5\,O = 2\,Mn^{2+} + 5\,O_2 + 3\,H_2O$$

4 REAGENTS

Distilled water or water of equivalent purity, free from carbon dioxide, shall be used in the test.

4.1 Mannitol, neutral, or alternatively **sorbitol,** neutral.

These products shall satisfy the following condition:

5.0 g, dissolved in 50 ml of carbon dioxide-free water, requires for neutralization not more than 0.3 ml of 0.02 N sodium hydroxide solution using phenolphthalein solution as indicator.

4.2 Hydrochloric acid, 0.5 N standard volumetric solution.

4.3 Sulphuric acid, approximately 4 N solution.

4.4 Sodium hydroxide, 0.5 N standard volumetric solution, free from carbonate.

4.5 Potassium permanganate, 0.1 N standard volumetric solution.

4.6 Screened methyl red, indicator solution.

Dissolve 0.01 g of methyl red and 0.01 g of bromocresol green in 95 % (V/V) ethanol and dilute to 100 ml with the same ethanol.

4.7 Phenolphthalein, 10 g/l ethanolic solution.

Dissolve 1 g of phenolphthalein in 95 % (V/V) ethanol, dilute to 100 ml with the same ethanol and add 0.02 N sodium hydroxide solution until the first appearance of a pink colour.

5 APPARATUS

Ordinary laboratory apparatus.

6 SAMPLING

Follow the principles described in ISO ....[1].

7 PROCEDURE

7.1 Determination of sodium oxide and boric oxide contents

7.1.1 *Test portion*

Weigh, to the nearest 0.000 5 g, 2 g of sodium perborate tetrahydrate or 1.5 g of sodium perborate monohydrate.

7.1.2 *Determination of sodium oxide content*

Dissolve the test portion (7.1.1) in about 100 ml of water and add 0.4 ml of the screened methyl red indicator solution (4.6). Add 25.0 ml of the hydrochloric acid solution (4.2). Titrate with the sodium hydroxide solution (4.4) until the solution is just yellow. Retain this solution for the determination of boric oxide content (see 7.1.3).

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[1] Under study.

At the same time determine the equivalence of the two solutions (4.2) and (4.4) as follows:

Add 25.0 ml of the hydrochloric acid solution (4.2) to about 50 ml of water. Add 0.4 ml of the screened methyl red indicator solution (4.6) and titrate with the sodium hydroxide solution (4.4) until the solution is just yellow.

7.1.3 *Determination of boric oxide content*

To the solution retained from the determination of sodium oxide content (see 7.1.2), add approximately 15 g of the mannitol or sorbitol (4.1) and 0.4 ml of the phenolphthalein solution (4.7). Titrate the solution with the sodium hydroxide solution (4.4) to a distinct pink colour.

NOTE — The solution turns red on the addition of the mannitol or sorbitol; on titration with the sodium hydroxide solution the colour of the solution changes to yellow and subsequently to the phenolphthalein pink colour at the end point.

To ensure that the correct titration end point is obtained, the following standard colour matching solution may be used for comparison with the solution being titrated.

Mix

- 50 ml of a 3.81 g/l solution of disodium tetraborate decahydrate ($Na_2B_4O_7.10H_2O$).
- 100 ml of water,
- 1.0 ml of the hydrochloric acid solution (4.2),
- 0.4 ml of the screened methyl red indicator solution (4.6),
- 0.4 ml of the phenolphthalein solution (4.7).

Equal volumes of this solution and of the titrand shall be compared in similar beakers.

7.2 Determination of available oxygen

7.2.1 *Test portion*

Weigh, to the nearest 0.000 2 g, 0.3 g of sodium perborate tetrahydrate or 0.2 g of sodium perborate monohydrate.

7.2.2 *Determination*

Introduce into a 250 ml conical flask about 100 ml of the sulphuric acid solution (4.3) and add the potassium permanganate solution (4.5), drop by drop, until a faint pink colour persists. Transfer the test portion (7.2.1) quantitatively to the flask. Stir thoroughly to dissolve and titrate with the potassium permanganate solution (4.5) until the faint pink colour, present before the addition of the sample, again persists.

8 EXPRESSION OF RESULTS

8.1 Sodium oxide content (Na_2O) is given, as a percentage by mass, by the formula :

$$\frac{V_2 - V_1}{m_1} \times 1.549$$

where

V_1 is the volume, in millilitres, of the sodium hydroxide solution (4.4) used in the titration of the excess of the hydrochloric acid solution (4.2);

V_2 is the volume, in millilitres, of sodium hydroxide solution (4.4) used in the titration of the 25 ml of the hydrochloric acid solution (4.2);

m_1 is the mass, in grams, of the test portion (7.1.1)

8.2 Boric oxide content (B_2O_3) is given, as a percentage by mass, by the formula :

$$\frac{V_3}{m_1} \times 1.741$$

where

V_3 is the volume, in millilitres, of the sodium hydroxide solution (4.4) used in the titration, after addition of the mannitol or sorbitol (4.1);

m_1 is the mass, in grams, of the test portion (7.1.1).

8.3 Available oxygen (O_2) is given, as a percentage by mass, by the formula :

$$\frac{V_4}{m_2} \times 0.080$$

where

V_4 is the volume, in millilitres, of the potassium permanganate solution (4.5) used in the titration;

m_2 is the mass, in grams, of the test portion (7.2.1).

9 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.