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Crude sodium borates for industrial use — Determination of total titanium content — Photometric method

Borates de sodium bruts à usage industriel — Dosage du titane total — Méthode photométrique

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FOREWORD

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Crude sodium borates for industrial use — Determination of total titanium content — Photometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a photometric method for the determination of the total titanium content of crude sodium borates for industrial use, for material containing up to 0,1 % titanium.

The method, as specified, is not applicable in the presence of vanadium or high silica content. Molybdenum and chromium also interfere. (See clause 8, "Bibliographical reference"). The same reference also states that fluoride destroys the colour and that phosphates and alkali sulphates have a slight fading action.

2 PRINCIPLE

Fusion of a test portion with sodium carbonate, extraction of the cooled melt with sulphuric acid and the addition of hydrogen peroxide.

Photometric measurement, at a wavelength of approximately 420 nm, of the yellow-coloured complex ions formed.

3 REAGENTS

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

3.1 Sodium carbonate, anhydrous.

3.2 Sulphuric acid, approximately 9 M solution.

3.3 Hydrogen peroxide, approximately 30 % (*m/m*) solution.

3.4 Titanium, standard solution, corresponding to 0,100 g of TiO_2 per litre.

Weigh, to the nearest 0,001 g, 0,443 g of potassium titanium oxalate dihydrate [$K_2TiO(C_2O_4)_2.2H_2O$] and transfer it to a Kjeldahl flask. Add 1 g of ammonium sulphate and 100 ml of sulphuric acid, approximately 18 M solution. Heat the mixture gradually to boiling and keep it boiling gently for 10 min. Cool the solution and pour it slowly and quantitatively, with stirring and cooling, into 750 ml of cold water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,000 10 g of TiO_2.

3.5 Titanium, standard solution, corresponding to 0,020 g of TiO_2 per litre.

Take 100,0 ml of the standard titanium solution (3.4), transfer it to a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,000 020 g of TiO_2.

3.6 Universal indicator paper.

4 APPARATUS

Ordinary laboratory apparatus and

4.1 Platinum crucible, with platinum lid.

4.2 Spectrophotometer, or

4.3 Photoelectric absorptiometer.

5 PROCEDURE

5.1 Test portion

Weigh, to the nearest 0,001 g, approximately 1 g of the test sample into the platinum crucible (4.1).

5.2 Blank test

Carry out a blank test, at the same time as the determination, following the same procedure and using the same quantities of all the reagents used for the determination.

5.3 Preparation of the calibration curve

5.3.1 *Preparation of standard colorimetric solutions relating to photometric measurements carried out with cells of 4 cm optical path length*

Into each of a series of six 100 ml one-mark volumetric flasks, place the volumes of the standard titanium solution (3.5) shown in the following table :

Standard titanium solution (3.5)	Corresponding mass of TiO_2
ml	g
0[1]	0
5.0	0,000 10
10.0	0,000 20
15.0	0,000 30
20.0	0,000 40
25.0	0,000 50

[1] Blank test of the reagents used for the preparation of the calibration curve.

To each flask add the quantity of water necessary to make up the volume to 50 ml, then in turn, mixing after each addition, add 10 ml of the sulphuric acid solution (3.2) and 1 ml of the hydrogen peroxide solution (3.3). Dilute to the mark, mix and wait for 5 min.

5.3.2 *Photometric measurements*

Carry out the measurements with the aid of the spectrophotometer (4.2) at a wavelength of about 420 nm, or with the photoelectric absorptiometer (4.3) fitted with suitable filters, after having adjusted the instrument to zero absorbance against water.

5.3.3 *Preparation of the calibration chart*

Deduct the absorbance of the reagent blank from that of each standard colorimetric solution. Plot a graph having, for example, the values in grams of the quantities of TiO_2 contained in 100 ml of standard colorimetric solutions as abscissae, and the corresponding values of absorbance as ordinates.

5.4 Determination

5.4.1 *Preparation of the test solution*

Add 2 g of the sodium carbonate (3.1) to the platinum crucible (4.1) containing the test portion (5.1), mix, cover the crucible with its lid, heat the contents cautiously and maintain just at the point of fusion until a clear melt is obtained. Cool and add 50 ml of hot water. After digestion, neutralize the solution in the presence of the indicator paper (3.6), with the sulphuric acid solution (3.2). Boil gently to remove the carbon dioxide, cool and add a further 10 ml of the sulphuric acid solution (3.2).

5.4.2 *Development of the colour*

Transfer the test solution (5.4.1) quantitatively to a 100 ml one-mark volumetric flask and add 1 ml of the hydrogen peroxide solution (3.3). Dilute to the mark, mix and wait for 5 min.

5.4.3 *Photometric measurements*

Carry out the photometric measurements of the test and blank solutions, following the procedure specified in 5.3.2.

6 EXPRESSION OF RESULTS

By means of the calibration chart (5.3.3), determine the quantities of TiO_2 corresponding to the values of the photometric measurements.

The titanium content, expressed as titanium dioxide (TiO_2), is given, as a percentage by mass, by the formula :

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

where

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

m_1 is the mass, in grams, of titanium dioxide found in the test solution;

m_2 is the mass, in grams, of titanium dioxide found in the blank test solution.

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.

8 BIBLIOGRAPHICAL REFERENCE

Scott, *Standard Methods of Chemical Analysis*, 5th edition, page 987, D. Van Nostrand Company, Inc., 1939.