INTERNATIONAL STANDARD ISO 2366

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Cryolite, natural and artificial — Determination of sodium content — Flame emission and atomic absorption spectrophotometric methods

Cryolithe naturelle et artificielle — Dosage du sodium — Méthodes par spectrophotométrie de flamme (émission) et par absorption atomique

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FOREWORD

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International Standard ISO 2366 was drawn up by Technical Committee ISO/TC 47, *Chemistry,* and circulated to the Member Bodies in November 1972.

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

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

No Member Body expressed disapproval of the document.

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Cryolite, natural and artificial — Determination of sodium content — Flame emission and atomic absorption spectrophotometric methods

1 SCOPE

This International Standard specifies a flame emission spectrophotometric method and a flame atomic absorption spectrophotometric method for the determination of the sodium content of natural and artificial cryolite.

2 FIELD OF APPLICATION

The methods are applicable as alternatives to the determination of the sodium content of natural and artificial cryolite of normal composition, for which the molar ratio NaF/AlF_3 is equal to about 3.

3 REFERENCE

ISO/R 1619, *Cryolite (natural and artificial) — Preparation and storage of test samples.*

4 PRINCIPLE

Dissolution of a test portion in hydrochloric acid and water after attack by concentrated sulphuric acid.

— Flame emission spectrophotometric method : Atomization of the test solution in a flame (oxy-hydrogen, for example) and determination of sodium by measurement of the intensity of the radiation at 589 nm.

— Flame atomic absorption spectrophotometric method : Atomization of the solution in an air-acetylene flame and determination of sodium by spectrophotometric measurement of the absorption of the 589 nm line emitted by a sodium hollow-cathode lamp.

5 REAGENTS

Distilled water, or water of equivalent purity, shall be used in the test.

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

5.2 Hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*) solution.

5.3 Standard sodium solution corresponding to 1,00 g of sodium per litre.

Weigh, to the nearest 0,000 1 g, 3,089 2 g of anhydrous sodium sulphate, previously dried at about 120 °C and cooled in a desiccator. Place in a beaker of convenient capacity (250 ml, for example), dissolve in about 100 ml of water, add 10 ml of the hydrochloric acid solution (5.2), transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

Transfer the solution to a plastics bottle.[1)]

1 ml of this standard solution contains 1,00 mg of Na.

5.4 Standard sodium solution corresponding to 0,100 g of sodium per litre.

Place 50,0 ml of the standard sodium solution (5.3) in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this solution contains 0,100 mg of Na.

Prepare this solution at the time of use and transfer to a plastics bottle.[1)]

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Platinum dish, diameter about 75 mm, height about 30 mm.

6.2 Flame spectrophotometer, fitted with an atomizer-burner constructed so as to excite emission of the 589 nm sodium line.

or

6.3 Atomic absorption spectrophotometer, fitted with a burner fed by compressed air and acetylene.

6.4 Sodium lamp, of the hollow-cathode or discharge type.

1) Polyethylene, polytetrafluoroethylene and polypropylene are, among others, suitable materials.

7 PROCEDURE

7.1 Flame emission spectrophotometric method

7.1.1 *Test portion*

Weigh, to the nearest 0,000 1 g, 0,500 g of the dried test sample, prepared following the instructions given in 2.3 of ISO/R 1619.

7.1.2 *Preparation of the calibration curve*

7.1.2.1 PREPARATION OF THE STANDARD MATCHING SOLUTIONS

Into a series of eleven 1 000 ml one-mark volumetric flasks, place the volumes of the standard sodium solution (5.3) indicated in the following table.

Standard sodium solution (5.3)	Corresponding mass of sodium	Corresponding mass of sodium in 100 g of cryolite
ml	mg	g
0*	0	0
2,0	2,0	4
4,0	4,0	8
6,0	6,0	12
8,0	8,0	16
10,0	10,0	20
12,0	12,0	24
14,0	14,0	28
16,0	16,0	32
18,0	18,0	36
20,0	20,0	40

* Blank test on the reagents used for the preparation of the calibration curve.

Add to each flask 10 ml of the sulphuric acid solution (5.1) and 10 ml of the hydrochloric acid solution (5.2). Then dilute to the mark, mix and transfer immediately to plastics bottles.

Only use standard solutions which have been freshly prepared.

NOTE — Because of the different sensitivities of flame spectrophotometric instruments, the concentrations of the standard matching solutions, of the test solution and of the blank test solution should be modified so that the measurement can be made in the field of highest sensitivity of the equipment used.

7.1.2.2 SPECTROPHOTOMETRIC MEASUREMENT

Switch on the spectrophotometer (6.2) in advance to allow sufficient time for its stabilization. Adjust the sensitivity of the apparatus and the opening of the slit according to the characteristics of the apparatus and to ensure a band pass of not more than 6 nm centred on the emission maximum (theoretical value 589 nm).

Atomize the standard matching solutions (7.1.2.1) into the flame and measure the intensities of the emitted radiations, after having adjusted the zero of the transmission scale to the blank test on the reagents used for the preparation of the calibration curve, and the 100 point on the scale to the solution containing 20 mg of Na per litre.

Take care to keep the quantity of the solutions atomized in the flame constant per unit of time throughout the preparation of the calibration curve.

7.1.2.3 PREPARATION OF CALIBRATION CHART

Plot a graph having, for example, the number of milligrams of Na contained in the standard matching solutions on the abscissa and the corresponding values of the intensities on the ordinate.

7.1.3 *Determination*

7.1.3.1 PREPARATION OF THE TEST SOLUTION

Place the test portion (7.1.1) in the platinum dish (6.1), add 5 ml of the sulphuric acid solution (5.1) and heat with care in a well-ventilated fume cupboard until the hydrogen fluoride has been completely eliminated (15 to 20 min).

Then increase the temperature and evaporate the excess sulphuric acid. Add 3 ml of the hydrochloric acid solution (5.2) to the dish, then 30 ml of water and warm to complete solution.

Allow to cool, transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark and mix. Take 10,0 ml of the solution and place in a 1 000 ml one-mark volumetric flask. Add 10 ml of the sulphuric acid solution (5.1) and 10 ml of the hydrochloric acid solution (5.2), dilute to the mark and mix.

Transfer the solution to a plastics bottle.

7.1.3.2 SPECTROPHOTOMETRIC MEASUREMENTS

7.1.3.2.1 Approximate measurement

Carry out an initial orientation measurement on the test solution (7.1.3.1), according to the procedure specified in 7.1.2.2, at the same time as the spectrophotometric measurements on the standard matching solutions (7.1.2.1) are carried out.

7.1.3.2.2 Bracketing measurement

Carry out a second measurement on the test solution (7.1.3.1) by bracketing between two solutions differing by not more than 2 g of Na (relative to 100 g of cryolite).

For the preparation of these bracketing solutions, follow the procedure specified in 7.1.2.1, using in all cases convenient quantities of the standard sodium solution (5.4). These quantities shall not differ by more than 10 ml.

Bestelformulier

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