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Cryolite, natural and artificial — Determination of aluminium content — Atomic absorption method

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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 2830 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and circulated to the Member Bodies in July 1972.

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

Australia	India	South Africa, Rep. of
Austria	Ireland	Sweden
Belgium	Israel	Switzerland
Canada	Italy	Thailand
Czechoslovakia	Mexico	Turkey
Egypt, Arab Rep. of	Netherlands	United Kingdom
France	Poland	U.S.S.R.
Germany	Portugal	
Hungary	Romania	

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

The Member Body of the following country expressed disapproval of the document on technical grounds :

New Zealand

Printed in Switzerland

Cryolite, natural and artificial — Determination of aluminium content — Atomic absorption method

1 SCOPE

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

2 FIELD OF APPLICATION

The method is applicable to the determination of aluminium in 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 concentrated sulphuric acid and treatment with hydrochloric acid and water. Atomization of the solution into an acetylene-*di*nitrogen monoxide flame and determination of the aluminium content by photometric measurement of the absorption of the 309,3 nm line emitted by an aluminium 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 Sodium chloride, 4,067 g/l solution.

Weigh, to the nearest 0,000 1 g, 0,406 7 g of sodium chloride, previously dried at 105 °C and cooled in a desiccator, place in a 100 ml one-mark volumetric flask, dissolve in a little water, dilute to the mark and mix.

5.4 Aluminium, standard solution, corresponding to 1,00 g of Al per litre.

Pickle 1,5 g of extra pure aluminium (99,999 % purity), in the form of shavings obtained by milling or drilling, in a little nitric acid, ρ approximately 1,40 g/ml, about 68 % (*m/m*) solution.

Wash the pickled shavings with water and then dry them by washing with acetone. Weigh, to the nearest 0,000 1 g, 1,000 g of the dried shavings, put them in a tall-form beaker of suitable capacity (for example, 250 ml) and add about 100 ml of water, 5 ml of the hydrochloric acid solution (5.2) and 10 ml of the sulphuric acid solution (5.1). Wait until the reaction subsides, then place the beaker on a sand bath and maintain a gentle heat until all the aluminium has dissolved. Allow to cool, 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 1,00 mg of Al.

6 APPARATUS

Ordinary laboratory apparatus and

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

6.2 Atomic absorption spectrophotometer, fitted with a burner fed from cylinders of acetylene and *di*nitrogen monoxide.

6.3 Aluminium hollow cathode lamp

7 PROCEDURE

7.1 Test portion

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

7.2 Preparation of the calibration curve

7.2.1 *Preparation of the standard matching solutions*

Into a series of nine 100 ml one-mark volumetric flasks, place the volumes of the standard aluminium solution (5.4) shown in the following table :

Standard aluminium solution (5.4)	Corresponding mass of aluminium	Corresponding mass of aluminium in 100 g of cryolite
ml	mg	g
0 *	—	—
1,0	1,0	2
2,0	2,0	4
3,0	3,0	6
4,0	4,0	8
5,0	5,0	10
6,0	6,0	12
7,0	7,0	14
8,0	8,0	16

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

Add to each flask 1 ml of the hydrochloric acid solution (5.2), 1 ml of the sulphuric acid solution (5.1)and 10 ml of the sodium chloride solution (5.3) (corresponding mass of sodium 0,016 g : this mass corresponds on average to the sodium present in a test portion of 0,05 g of cryolite containing 32 % of sodium). Dilute to the mark, mix and transfer to a plastics bottle.[1)]

Use only freshly prepared standard matching solutions.

7.2.2 *Spectrophotometric measurements*

7.2.2.1 ADJUSTMENT OF THE APPARATUS fitted with aluminium hollow cathode lamp (6.3)

Switch on the current to the apparatus (6.2) to allow sufficient time for its stabilization. Adjust the wavelength to about 309,3 nm and the sensitivity and the aperture of the slit according to the characteristics of the apparatus. Adjust the acetylene and *di*nitrogen monoxide pressures according to the characteristics of the burner so as to obtain a clear, non-luminous, oxidizing flame.

7.2.2.2 SPECTROPHOTOMETRIC MEASUREMENTS

Atomize the series of standard matching solutions (7.2.1) into the flame and, for each, measure the absorbance. Take care to keep the quantity of solutions atomized in the flame constant per unit of time throughout the preparation of the calibration curve. Carry out at least three measurements for each standard matching solution and calculate the mean value.

NOTE — Pass water through the burner after each series of measurements.

7.2.3 *Preparation of the calibration chart*

Plot a graph having, for example, the values, expressed in milligrams, of the quantities of aluminium contained in 1 000 ml of the standard matching solutions as abscissae and the corresponding values of the measured absorbances, minus the absorbance of the reagent blank, as ordinates.

7.3 Determination

7.3.1 *Preparation of the test solution*

Place the test portion (7.1) in the platinum dish (6.1), add 5 ml of the sulphuric acid solution (5.1) and heat carefully on a sand bath in a well-ventilated cup-board, until complete elimination of hydrogen fluoride (15 to 20 min). Then raise the temperature and evaporate the excess sulphuric acid. Add 3 ml of the hydrochloric acid solution (5.2) and 30 ml of water to the dish and heat until solution is complete. Allow to cool, transfer quantitatively to a 100 ml one-mark volumetric flask, dilute to the mark and mix.

Place 10,0 ml of this solution in a 100 ml one-mark volumetric flask, add 1 ml of the hydrochloric acid solution (5.2), 1 ml of the sulphuric acid solution (5.1), dilute to the mark and mix.

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

7.3.2 *Spectrophotometric measurements*

7.3.2.1 APPROXIMATE MEASUREMENT

Carry out a first measurement of the test solution (7.3.1), following the procedure described in 7.2.2, at the same time as the spectrophotometric measurements on the standard matching solutions (7.2.1) are carried out.

7.3.2.2 BRACKETING MEASUREMENT

Carry out a second measurement on the test solution (7.3.1) by bracketing between two standard matching solutions differing by only 1 mg of aluminium per 100 ml, one at a concentration above, and one at a concentration below, that of the test solution. For the preparation of these standard matching solutions, follow the procedure given in 7.2.1, using suitable quantities of the standard aluminium solution (5.4).

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

7.4 Blank test

7.4.1 *Preparation of the blank test solution*

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.

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

7.4.2 *Spectrophotometric measurements*

7.4.2.1 APPROXIMATE MEASUREMENT

Carry out a first measurement of the blank test solution (7.4.1), following the procedure given in 7.2.2, at the same time as the spectrophotometric measurements on the standard matching solutions (7.2.1) are carried out.

7.4.2.2 BRACKETING MEASUREMENT

Carry out a second measurement on the blank test solution (7.4.1), according to the procedure described in 7.3.2.2 at the same time as the measurements described in the same clause.

8 EXPRESSION OF RESULTS

The aluminium concentration C, expressed as milligrams per litre, of the solution measured by the spectrophotometer, is given by the formula

$$C = \left[C_1 + (C_2 - C_1) \frac{E - E_1}{E_2 - E_1} \right] - \left[C_3 + (C_4 - C_3) \frac{E_0 - E_3}{E_4 - E_3} \right]$$

where

C_1 is the concentration, in milligrams per litre, of the weaker standard matching solution used during the determination;

E_1 is the value of the corresponding measurement;

C_2 is the concentration, in milligrams per litre, of the stronger standard matching solution used during the determination;

E_2 is the value of the corresponding measurement;

E is the value of the measurement corresponding to the test solution;

C_3 is the concentration, in milligrams per litre, of the weaker standard matching solution used during the blank test;

E_3 is the value of the corresponding measurement;

C_4 is the concentration, in milligrams per litre, of the stronger standard matching solution used during the blank test;

E_4 is the value of the corresponding measurement;

E_0 is the value of the measurement corresponding to the blank test solution.

The aluminium content (Al) is given, as a percentage by mass, by the formula :

$$\frac{C \times 10 \times 100}{m \times 10 \times 1\,000} = \frac{C}{m \times 10}$$

where

m is the mass, in grams, of the test portion.

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 the document to which reference is made, or regarded as optional.

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

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