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Cryolite, natural and artificial, and aluminium fluoride for industrial use — Determination of water content — Electrometric method

Cryolithe, naturelle et artificielle, et fluorure d'aluminium à usage industriel — Dosage de l'eau — Méthode électrométrique

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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 3392 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the Member Bodies in May 1974.

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

Austria	Hungary	South Africa, Rep. of
Belgium	India	Spain
Bulgaria	Ireland	Sweden
Chile	Israel	Switzerland
Czechoslovakia	Italy	Thailand
Egypt, Arab Rep. of	Netherlands	Turkey
France	Poland	United Kingdom
Germany	Portugal	Yugoslavia

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

U.S.S.R.

Cryolite, natural and artificial, and aluminium fluoride for industrial use – Determination of water content – Electrometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies an electrometric method for determination of the water content of natural and artificial cryolite and aluminium fluoride for industrial use.

2 REFERENCES

ISO/R 760, *Determination of water by the Karl Fischer method*.

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

ISO 2925, *Aluminium fluoride for industrial use – Preparation and storage of test samples*.

3 PRINCIPLE

Simultaneous determinations of :

- a) loss of mass at 550 °C, which is the sum of the masses of water which has not undergone any reaction, and of hydrogen fluoride formed by pyrohydrolysis;
- b) water released at 550 °C, determined by the Karl Fischer method (direct electrometric titration).

The difference between these two determinations is due to the formation of hydrogen fluoride by pyrohydrolysis (2 HF corresponds to 1 H₂O).

Calculation of the water content from the two results obtained.

4 REAGENTS

During the analysis use only reagents of recognized analytical grade.

4.1 Nitrogen.

4.2 Calcium chloride, anhydrous.

4.3 Pyridine, anhydrous (water content less than 0,05 % (m/m)) (see ISO/R 760 sub-clause 4.3).

4.4 Karl Fischer reagent (of which 1 ml corresponds to about 1 mg of water).

Prepare the reagent as specified in 4.5 of ISO/R 760 and standardize it as specified in 7.2.1 of ISO/R 760, using the sodium tartrate (4.5) as water standard.

4.5 Sodium tartrate, crystalline, dihydrate (Na₂C₄H₄O₆·2H₂O), for use as a water standard (see ISO/R 760 clause 4.6) or, alternatively, water.

NOTE – If water is used, prepare the standard solution as specified in 4.7 of ISO/R 760 (with suitable dilution in a one-mark volumetric flask with methanol) and standardize the Karl Fischer reagent as specified in A.2 of ISO/R 760.

4.6 Magnesium perchlorate [Mg(ClO₄)₂].

5 APPARATUS

All the glassware used shall be previously dried for 30 min in an oven, controlled at approximately 130 °C, and allowed to cool and stored in a dry atmosphere.

Ordinary laboratory apparatus and

5.1 Steel cylinders, containing the nitrogen (4.1), fitted with a pressure reducer and a flowmeter covering the range 0 to 90 l/h.

5.2 Absorption tower, filled with the calcium chloride (4.2).

5.3 Two gas washing bottles, Drechsel type, containing concentrated sulphuric acid (ρ approximately 1,84 g/ml).

5.4 Tubular electric furnace, capable of being controlled at 550 ± 5 °C. Length of the heated part 400 mm, diameter of the tube 50 mm.

5.5 Quartz tube, consisting of a 450 mm length of internal diameter 25 mm, extended by a short length at right angles of length 150 mm and internal diameter 4 mm.

5.6 Nickel combustion boat, 80 mm long, 15 mm wide and 10 mm high.