INTERNATIONAL STANDARD ISO 1693

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Cryolite, natural and artificial — Determination of fluorine content — Modified Willard-Winter method

Cryolithe, naturelle et artificielle — Dosage du fluor — Méthode de Willard-Winter modifiée

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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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 47 has reviewed ISO Recommendation R 1693 and found it technically suitable for transformation. International Standard ISO 1693 therefore replaces ISO Recommendation R 1693-1970 to which it is technically identical.

ISO Recommendation R 1693 was approved by the Member Bodies of the following countries :

Australia	Hungary	Romania
Austria	India	South Africa, Rep. of
Belgium	Iran	Spain
Brazil	Israel	Switzerland
Canada	Italy	Thailand
Czechoslovakia	Netherlands	Turkey
Egypt, Arab Rep. of	New Zealand	United Kingdom
France	Norway	U.S.S.R.
Germany	Peru	Yugoslavia
Greece	Poland	

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1693 into an International Standard.

Printed in Switzerland

Cryolite, natural and artificial — Determination of fluorine content — Modified Willard-Winter method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a modified Willard-Winter method for the determination of the fluorine content of natural and artificial cryolite and of natural and synthetic materials having a molar ratio (NaF/AlF_3) between 3 and 1,7 approximately.

2 REFERENCE

ISO 1619, *Cryolite, natural and artificial — Preparation and storage of test samples.*

3 PRINCIPLE

Fusion of a test portion with sodium carbonate.

Separation of fluorine by distillation with sulphuric acid or perchloric acid. Titration with thorium nitrate solution using sodium alizarinsulphonate-methylene blue as indicator.

Alternatively, the thorium nitrate titration may be made using only sodium alizarinsulphonate, the end-point being spectrophotometrically determined under carefully defined conditions when the absorbance at 525 nm reaches the arbitrary value of 0,60.

4 REAGENTS

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

4.1 Sodium carbonate, anhydrous.

4.2 Hydrochloric acid, approximately 0,06 N solution.

Dilute 5 ml of hydrochloric acid, ρ approximately 1,19 g/ml, about 38 % (*m/m*), with water to 1 000 ml.

4.3 Sodium hydroxide, 20 g/l solution.

4.4 Sulphuric acid, approximately 24 N solution.

Carefully add, in small quantities, 200 ml of sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution, to approximately 100 ml of water, cool and dilute to 300 ml.

or

4.4.1 Perchloric acid, ρ approximately 1,60 g/ml, about 64,5 % (*m/m*) solution.

4.5 Buffer solution, of pH 2,7.

Dissolve 9,45 g of monochloroacetic acid in 50 ml of 1 N sodium hydroxide solution and dilute to 100 ml.

4.6 Thorium nitrate, approximately 0,067 N standard volumetric solution.

1 ml of this standard volumetric solution is equivalent to approximately 1,3 mg of fluorine (F).

4.6.1 *Preparation of the solution*

Dissolve 9,45 g of thorium nitrate tetrahydrate [$Th(NO_3)_4.4H_2O$], or the corresponding mass of thorium nitrate of a different degree of hydration, in water and dilute to 1 000 ml.

4.6.2 *Standardization of the solution*

4.6.2.1 PREPARATION OF THE STANDARD REFERENCE SOLUTION

Weigh, to the nearest 0,000 1 g, about 0,2 g of extra pure anhydrous sodium fluoride previously ignited at 600 °C in a platinum dish and cooled in a desiccator. Transfer, using 20 to 30 ml of water, into the distillation flask (5.4.1) containing several glass balls (diameter 2 to 3 mm).

Stopper the distillation flask and add through the dropping funnel (5.4.5) either 50 ml of the sulphuric acid solution (4.4) or 30 ml of the perchloric acid solution (4.4.1), depending on which has been selected.

Carry out the distillation as specified in 6.3.2.