INTERNATIONAL STANDARD ISO 1620

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Cryolite, natural and artificial — Determination of silica content — Reduced molybdosilicate spectrophotometric method

Cryolithe, naturelle et artificielle — Dosage de la silice — Méthode spectrophotométrique au molybdosilicate réduit

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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 1620 and found it technically suitable for transformation. International Standard ISO 1620 therefore replaces ISO Recommendation R 1620-1970 to which it is technically identical.

ISO Recommendation R 1620 was approved by the Member Bodies of the following countries :

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

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

Canada

No Member Body disapproved the transformation of ISO/R 1620 into an International Standard.

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Cryolite, natural and artificial — Determination of silica content — Reduced molybdosilicate spectrophotometric method

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a reduced molybdosilicate spectrophotometric method for the determination of the silica 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.

The method is applicable to products the phosphorus(V) oxide (P_2O_5) content of which does not exceed 0,02 % (*m/m*).

1.1 Special case (under study)

Phosphorus(V) oxide contents greater than 0,02 % (*m/m*).

2 REFERENCE

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

3 PRINCIPLE

Alkaline fusion of a test portion by means of a mixture of sodium carbonate and boric acid. Dissolution of the fused mass in excess nitric acid so that the final pH of the solution is between 0,7 and 0,9 after dilution to 500 ml, or between 0,3 and 0,5 after dilution to 250 ml.

Formation, in a suitable aliquot, of the (yellow) oxidized molybdosilicate under clearly defined conditions of acidity, concentration of reagents, temperature and time.

Selective reduction of the complex in a high-acidity sulphuric medium and in the presence of tartaric acid.

Spectrophotometric measurement of the reduced coloured complex at a wavelength of about 815 nm.

4 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only redistilled water.

4.1 Sodium carbonate, anhydrous.

4.2 Boric acid (H_3BO_3).

4.3 Nitric acid, approximately 8 N solution.

Dilute 540 ml of nitric acid, ρ approximately 1,40 g/ml, about 68 % (*m/m*) solution, with water to 1 000 ml.

4.4 Sodium molybdate, 195 g/l solution (0,8 M approximately).

In a polytetrafluoroethylene (PTFE) beaker, dissolve 19,5 g of sodium molybdate dihydrate ($Na_2MoO_4.2H_2O$) in hot water and, after cooling, dilute to 100 ml.

Transfer the solution to a bottle made of silica-free material and, if necessary, filter before use.

4.5 Tartaric acid, 100 g/l solution.

4.6 Sulphuric acid, approximately 16 N solution.

Carefully add 450 ml of sulphuric acid, ρ approximately 1,84 g/ml, about 96 % (*m/m*) solution, to about 500 ml of water. Cool, dilute to 1 000 ml and mix.

4.7 Reducing solution.

Use either of the following two solutions :

4.7.1 4-Amino-3-hydroxy-1-naphthalene sulphonic acid, 1,5 g/l solution.

a) Dissolve 7 g of anhydrous sodium sulphite (Na_2SO_3) in 50 ml of water. Add 1,5 g of 4-amino-3-hydroxy-1-naphthalene sulphonic acid ($C_{10}H_9NO_4S$).

b) Dissolve 90 g of anhydrous sodium metabisulphite ($Na_2S_2O_5$) in 900 ml of water.

Mix the two solutions a) and b), dilute to 1 000 ml and mix. Filter if necessary.

Store in a cool place in an amber-coloured bottle made of silica-free material.

4.7.2 Ascorbic acid, 20 g/l solution.

Prepare this solution just before use.