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# INTERNATIONAL STANDARD



# 1694

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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## **Cryolite, natural and artificial — Determination of iron content — 1,10-Phenanthroline photometric method**

*Cryolithe, naturelle et artificielle — Dosage du fer — Méthode photométrique à la phénanthroline-1,10*

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Sample Document

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**Descriptors** : aluminium ores, cryolite, chemical analysis, determination of content, iron, spectrophotometric analysis.

## 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 1664 and found it technically suitable for transformation. International Standard ISO 1694 therefore replaces ISO Recommendation R 1694-1970 to which it is technically identical.

ISO Recommendation R 1694 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	Turkey
Czechoslovakia	Netherlands	United Kingdom
Egypt, Arab Rep. of	New Zealand	U.S.S.R.
France	Norway	Yugoslavia
Germany	Poland	
Greece	Portugal	

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1694 into an International Standard.

# Cryolite, natural and artificial – Determination of iron content – 1,10-Phenanthroline photometric method

## 1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a 1,10-phenanthroline photometric method for the determination of the iron content of natural and artificial cryolite and of natural and synthetic materials having a molar ratio (NaF/AlF<sub>3</sub>) between 3 and 1,7 approximately.

The method is applicable to products having an iron content, expressed as iron(III) oxide, equal to or greater than 0,020 % (*m/m*).

## 2 REFERENCE

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

## 3 PRINCIPLE

Dissolution of a test portion by either alkaline or acid fusion.

Preliminary reduction of iron(III) by means of hydroxylammonium chloride.

Formation of the complex iron(II)-1,10-phenanthroline in a buffered medium (pH value between 3,5 and 4,2).

Photometric measurement of the coloured complex at a wavelength of about 510 nm.

## 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 Boric acid** (H<sub>3</sub>BO<sub>3</sub>).

**4.3 Potassium pyrosulphate** (K<sub>2</sub>S<sub>2</sub>O<sub>7</sub>), finely crushed.

**4.4 Nitric acid**, approximately 8 N solution.

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

**4.5 Hydrochloric acid**, approximately 6 N solution.

Dilute 515 ml of hydrochloric acid,  $\rho$  approximately 1,19 g/ml, about 38 % (*m/m*) solution, with water and dilute to 1 000 ml.

**4.6 Hydroxylammonium chloride** (NH<sub>2</sub>OH.HCl), 10 g/l solution.

**4.7 1,10-Phenanthroline hydrochloride**, monohydrate, (C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>.HCl.H<sub>2</sub>O) 2,5 g/l solution.

This reagent may be replaced by a 2,5 g/l solution of 1,10-phenanthroline monohydrate (C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>.H<sub>2</sub>O).

**4.8 Buffer solution**, of pH 4,9.

Dissolve 272 g of sodium acetate trihydrate (CH<sub>3</sub>COONa.3H<sub>2</sub>O) in approximately 500 ml of water. Add 240 ml of glacial acetic acid,  $\rho$  approximately 1,05 g/ml, about 17,4 N. Dilute to 1 000 ml and mix.

**4.9 Sodium acetate** trihydrate (CH<sub>3</sub>COONa.3H<sub>2</sub>O), 500 g/l solution.

**4.10 Acetic acid**, dilute solution.

Dilute 500 ml of glacial acetic acid,  $\rho$  approximately 1,05 g/ml, about 17,4 N, with water and dilute to 1 000 ml.

**4.11 Iron**, standard solution corresponding to 0,200 g of iron(III) oxide per litre.

Prepare this solution by either of the following two methods :

**4.11.1** Weigh, to the nearest 0,001 g, 0,982 g of ammonium iron(II) sulphate hexahydrate [(NH<sub>4</sub>)<sub>2</sub>Fe(SO<sub>4</sub>)<sub>2</sub>.6H<sub>2</sub>O]. Place in a beaker of suitable capacity (for example 100 ml) and dissolve in water. Add 20 ml of sulphuric acid solution,  $\rho$  approximately 1,84 g/ml, 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 0,200 mg of Fe<sub>2</sub>O<sub>3</sub>.

**4.11.2** Weigh, to the nearest 0,001 g, 0,200 g of iron(III) oxide previously ignited at 600 °C and cooled in desiccator. Transfer to a beaker of suitable capacity (for example 100 ml), add 10 ml of hydrochloric acid solution,  $\rho$  approximately 1,19 g/ml, and heat gently to dissolve. 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 0,200 mg of Fe<sub>2</sub>O<sub>3</sub>.