# INTERNATIONAL STANDARD ISO 3391

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

# Cryolite, natural and artificial — Determination of calcium content — Flame atomic absorption method

*Cryolithe, naturelle et artificielle — Dosage du calcium — Méthode par absorption atomique dans la flamme*

**First edition — 1976-09-01**



**UDC 661.862.369 : 546.41 : 543.422** **Ref. No. ISO 3391-1976 (E)**

**Descriptors** : aluminium ores, cryolite, chemical analysis, determination of content, calcium, spectrophotometric analysis, atomic absorption spectroscopic analysis.

Price based on 4 pages

## 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 3391 was drawn up by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the Member Bodies in March 1974.



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

| | | |
|---|---|---|
| Austria | Hungary | Spain |
| Belgium | India | Sweden |
| Bulgaria | Israel | Switzerland |
| Chile | Italy | Thailand |
| Czechoslovakia | New Zealand | Turkey |
| Egypt, Arab Rep. of | Poland | United Kingdom |
| France | Portugal | U.S.S.R. |
| Germany | South Africa, Rep. of | Yugoslavia |

No Member Body expressed disapproval of the document.

New Zealand



Printed in Switzerland

# Cryolite, natural and artificial — Determination of calcium content — Flame atomic absorption method

## 1 SCOPE AND FIELD OF APPLICATION

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

## 2 REFERENCE

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

## 3 PRINCIPLE

Dissolution of a test portion in concentrated sulphuric acid and treatment with concentrated hydrochloric acid.

Aspiration of the solution into an acetylene dinitrogen monoxide flame.

Determination of calcium content by spectrophotometric measurement of the absorption of the 422,7 nm line emitted by a calcium hollow cathode lamp.

## 4 REAGENTS

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

**4.1 Sulphuric acid,** $\rho$ approximately 1,84 g/ml, about 96 % (*m/m*) solution.

**4.2 Hydrochloric acid,** $\rho$ approximately 1,19 g/ml, about 38 % (*m/m*) solution.

**4.3 Complexant.**

Either :

**4.3.1 Lanthanum nitrate,** 310 g/l solution.

Weigh, to the nearest 0,1 g, 31,0 g of lanthanum nitrate hexahydrate ($La(NO_3)_2.6H_2O$) and place a 100 ml one-mark volumetric flask. Dissolve in water dilute to the mark and mix.

NOTE — It is possible to use lanthanum chloride heptahydrate ($LaCl_3.7H_2O$), 270 g/l solution.

Or :

**4.3.2 Triethanolamine,** dilute solution.

Dilute 100 ml of triethanolamine [$(CH_2OHCH_2)_3N$] to 200 ml with water.

**4.4 Sodium chloride** solution, corresponding to 16,0 g of Na per litre.

Weigh, to the nearest 0,001 g, 4,067 g of sodium chloride, previously dried for 12 h at approximately 120 °C and cooled in a desiccator, into a 100 ml one-mark volumetric flask. Dissolve in water, dilute to the mark and mix.

**4.5 Aluminium,** acid solution corresponding to 6,6 g of Al per litre.

Weigh, to the nearest 0,001 g, 0,66 g of extra pure aluminium in the form of small shavings, and dissolve in 50,0 ml of hydrochloric acid solution, diluted 1 + 1 (*V* + *V*), in a porcelain dish. Transfer the solution quantitatively to a 100 ml one-mark volumetric flask, cool, dilute to the mark and mix.

**4.6 Calcium,** standard solution, corresponding to 1,00 g of Ca per litre.

Weigh, to the nearest 0,000 1 g, 2,497 2 g of calcium carbonate, previously dried for 2 h at approximately 110 °C and cooled in a desiccator. Transfer to a 250 ml beaker and dissolve with care in 12 ml of the hydrochloric acid solution (4.2), previously diluted with 12 ml of water.

Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, cool, dilute to the mark and mix. Transfer to a flask of material free from calcium.

1 ml of this standard solution contains 1,00 mg of Ca.

**4.7 Calcium,** standard solution, corresponding to 0,10 g of Ca per litre.

Place 100,0 ml of the standard calcium solution (4.6) in a 1 000 ml one-mark volumetric flask, dilute to the mark and mix. Transfer to a flask of material free from calcium.

1 ml of this standard solution contains 0,10 mg of Ca.