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**INTERNATIONAL STANDARD**



**3390**

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

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**Aluminium oxide primarily used for the production of aluminium – Determination of manganese content – Flame atomic absorption method**

*Oxyde d'aluminium principalement utilisé pour la production de l'aluminium – Dosage du manganèse – Méthode par absorption atomique dans la flamme*

First edition – 1976-09-01

SEP 10 1976

ANSI Internat Doc Sect

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UDC 661.862.22 : 546.711 : 543.422

Ref. No. ISO 3390-1976 (E)

**Descriptors :** aluminium oxide, chemical analysis, determination of content, manganese, spectrophotometric analysis, atomic absorption spectroscopic analysis.

## FOREWORD

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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 3390 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	Israel	Sweden
Chile	Italy	Switzerland
Czechoslovakia	Netherlands	Thailand
Egypt, Arab Rep. of	New Zealand	Turkey
France	Poland	United Kingdom
Germany	Portugal	Yugoslavia

No Member Body expressed disapproval of the document.

# Aluminium oxide primarily used for the production of aluminium – Determination of manganese 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 manganese content of aluminium oxide primarily used for the production of aluminium.

The method is applicable to products of which the manganese content, expressed as Mn, is between 0,000 2 and 0,005 % (*m/m*) (0,000 258 and 0,006 46 % (*m/m*), expressed as MnO).

NOTE – If the apparatus available does not have a sufficient sensitivity for the lower contents of manganese, it is recommended that there should be prior extraction of the manganese, by the method specified in annex A.

## 2 REFERENCES

ISO 802, *Aluminium oxide primarily used for the production of aluminium – Preparation and storage of test samples.*

ISO 2073, *Aluminium oxide primarily used for the production of aluminium – Preparation of sample solution for analysis – Method by means of attack by hydrochloric acid under pressure.*

## 3 PRINCIPLE

Dissolution of a test portion by heating in a sealed borosilicate tube with hydrochloric acid at a controlled temperature of 250 °C.

Aspiration of the solution in an air-acetylene flame and determination of manganese by spectrophotometric measurement of the absorption of the 279,5 nm line emitted by a manganese hollow cathode lamp.

## 4 REAGENTS

During the analysis, use only reagents of recognized analytical grade and only water with a conductivity less than or equal to 50  $\mu\text{S/m}$ .

**4.1 Hydrochloric acid**,  $\rho$  approximately 1,19 g/ml, about 12 N solution.

**4.2 Aluminium**, acid solution (base solution).

Pickle 60 g of extra pure aluminium (assay 99,999 %), in the form of shavings obtained by turning or drilling, in a little nitric acid,  $\rho$  approximately 1,40 g/ml. Wash the cleaned shavings with water and then dry them by washing with acetone.

Weigh, to the nearest 0,02 g, 53,0 g of the dried shavings, place in a beaker of convenient capacity and add 600 ml of the hydrochloric acid solution (4.1) and 300 ml of water.

Introduce 1 drop of mercury to facilitate the attack. Wait until the reaction subsides and then place the beaker on a sand bath or hot-plate and heat gently until all the aluminium has been dissolved. Add another 120 ml of the hydrochloric acid solution (4.1).

Allow to cool, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

**4.3 Manganese**, standard solution, corresponding to 0,500 g of Mn per litre.

1 ml of this standard solution, prepared according to 4.3.1 or 4.3.2, contains 0,000 5 g of Mn.

**4.3.1** Weigh, to the nearest 0,000 1 g, 1,802 g of manganese(II) chloride tetrahydrate ( $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ ), dissolve in water, transfer quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

**4.3.2** Weigh, to the nearest 0,000 1 g, 0,500 g of manganese, extra pure, place in a beaker of convenient capacity (for example 200 ml) and add 6 ml of water and 6 ml of the hydrochloric acid solution (4.1). Cover the beaker with a clock-glass and warm until the manganese has completely dissolved. Cool to ambient temperature and transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

**4.4 Manganese**, standard solution, corresponding to 0,010 g of Mn per litre.

Place 10,0 ml of the standard manganese solution (4.3) in a 500 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,010 mg of Mn.