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**INTERNATIONAL STANDARD**



**1232**

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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 silica content — Reduced  
molybdosilicate spectrophotometric method**

*Oxyde d'aluminium principalement utilisé pour la production de l'aluminium — Dosage de la silice — Méthode  
spectrophotométrique au molybdosilicate réduit*

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**Descriptors** : aluminium oxide, chemical analysis, determination of content, silicon dioxide, 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 1232 and found it technically suitable for transformation. International Standard ISO 1232 therefore replaces ISO Recommendation R 1232-1970 to which it is technically identical.

ISO Recommendation R 1232 was approved by the Member Bodies of the following countries :

Austria	Greece	Romania
Belgium	Hungary	South Africa, Rep. of
Canada	India	Spain
Chile	Iran	Sweden
Czechoslovakia	Israel	Switzerland
Egypt, Arab Rep. of	Italy	Turkey
France	Netherlands	United Kingdom
Germany	Portugal	U.S.S.R.

No Member Body expressed disapproval of the Recommendation.

No Member Body disapproved the transformation of ISO/R 1232 into an International Standard.

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

The method is applicable to products having an  $\text{SiO}_2$  content between 0,005 and 0,025 % (m/m), provided that the content of  $(\text{P}_2\text{O}_5 + \text{V}_2\text{O}_5)$  does not exceed 0,02 % (m/m).

### 1.1 Special cases

**1.1.1**  $\text{SiO}_2$  content between 0,025 and 0,25 % (m/m) [ $(\text{P}_2\text{O}_5 + \text{V}_2\text{O}_5)$  content not exceeding 0,02 % (m/m)].

**1.1.2**  $(\text{P}_2\text{O}_5 + \text{V}_2\text{O}_5)$  content exceeding 0,02 % (m/m) (under study).

## 2 REFERENCES

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

ISO 804, *Aluminium oxide primarily used for the production of aluminium – Preparation of solution for analysis – Method by alkaline fusion.*

ISO 2927, *Aluminium oxide primarily used for the production of aluminium – Sampling.*

## 3 PRINCIPLE

Formation of the oxidized molybdosilicate (yellow) under well-defined conditions of acidity, concentration of reagents, temperature and time.

Selective reduction of the complex in a highly acidic medium of sulphuric acid 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 analytical grade and only redistilled water.

**4.1 Sodium carbonate**, anhydrous.

**4.2 Boric acid** ( $\text{H}_3\text{BO}_3$ ).

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

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

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

Dissolve in a polytetrafluorethylene (PTFE) beaker 19,5 g of sodium molybdate dihydrate ( $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ ) 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.

Add carefully 450 ml of sulphuric acid,  $\rho$  approximately 1,84 g/ml, about 96 % (m/m), to about 500 ml of water. Cool, dilute to 1 000 ml and mix.

**4.7 Reducing solution.**

Use one 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 ( $\text{Na}_2\text{SO}_3$ ) in 50 ml of water. Add 1,5 g of 4-amino-3-hydroxy-1-naphthalene sulphonic acid ( $\text{C}_{10}\text{H}_9\text{NO}_4\text{S}$ ).

b) Dissolve 90 g of anhydrous sodium metabisulphite ( $\text{Na}_2\text{S}_2\text{O}_5$ ) 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.