

# INTERNATIONAL STANDARD

**ISO  
9285**

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## **Abrasive grains and crude — Chemical analysis of fused aluminium oxide**

**iTeh STANDARD PREVIEW**

*Abrasifs en grains et en roche — Analyse chimique de l'oxyde d'aluminium  
électrofondu*

ISO 9285:1997

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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# Abrasive grains and crude — Chemical analysis of fused aluminium oxide

## 1 Scope

This International Standard deals with the chemical analysis of abrasive grains or crude based on fused aluminium oxide. It applies to commercially available products but not necessarily to products which have been altered by use. It includes the following determinations:

- Loss on ignition
- Silicon dioxide
- Iron oxide
- Titanium dioxide
- Calcium oxide
- Magnesium oxide
- Zirconium oxide
- Aluminium oxide
- Analysis by atomic absorption spectrometry

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## 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 6353-1:1982, *Reagents for chemical analysis — Part 1: General test methods.*

ISO 6353-2:1983, *Reagents for chemical analysis — Part 2: Specifications — First series.*

ISO 6353-2/Add.2:1986, *Addendum 2 to ISO 6353-2:1983.*

ISO 6353-3:1987, *Reagents for chemical analysis — Part 3: Specifications — Second series.*

ISO 9138:1993, *Abrasive grains — Sampling and splitting.*

### 3 Reagents

#### 3.1 General

Unless otherwise indicated, it is intended that all reagents shall conform to the specifications given in ISO 6353-1, ISO 6353-2 and ISO 6353-3. Other grades may be used, provided it be first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. Unless otherwise indicated, references to water shall be understood to mean distilled water.

Paragraphs 3.2 to 3.21 include those reagents common to two or more analytical procedures. Other reagents will be found in the section which deals with the particular method in which they are used.

#### 3.2 Concentrated acids and ammonium hydroxide.

Concentrated acids and ammonium hydroxide having the following approximate volumetric masses  $\rho$ , in grams per millilitre or concentrations, in percentage by mass will be required:

Hydrochloric acid (HCl):	$\rho = 1,18$ g/ml
Nitric acid (HNO <sub>3</sub> ):	$\rho = 1,42$ g/ml
Sulphuric acid (H <sub>2</sub> SO <sub>4</sub> ):	$\rho = 1,84$ g/ml
Hydrofluoric acid (HF):	40 % (m/m)
Phosphoric acid (H <sub>3</sub> PO <sub>4</sub> ):	85 % (m/m)
Ammonium hydroxide (NH <sub>4</sub> OH):	$\rho = 0,90$ g/ml

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#### 3.3 Dilute acids and ammonium hydroxide.

The dilute acids and ammonium hydroxide referred to have varying concentrations. They shall be made up by mixing proportional volumes of the concentrated reagent and water. The dilute sulphuric acid mixtures shall be made up by slowly adding the acid to the water and stirring continuously. These dilutions are designated in the test procedures as (1 + 5), (1 + 8), etc., except for the very dilute solutions which are characterized by the percentage of reagent added. The designations in parentheses indicate the volume of the reagent added to the volume of water; for example H<sub>2</sub>SO<sub>4</sub> (1 + 9) contains 10 % (V/V) of H<sub>2</sub>SO<sub>4</sub> ( $\rho = 1,84$  g/ml).

#### 3.4 Ammonium acetate, 300 g/l solution.

Dissolve 300 g of NH<sub>4</sub>C<sub>2</sub>H<sub>3</sub>O<sub>2</sub> in water. Fill to the 1 l mark with water.

#### 3.5 Ammonium chloride (NH<sub>4</sub>Cl).

#### 3.6 Ammonium oxalate [(NH<sub>4</sub>)<sub>2</sub>C<sub>2</sub>O<sub>4</sub>], saturated solution.

#### 3.7 Diammonium hydrophosphate, 100 g/l solution.

Dissolve 10 g of (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> in 100 ml of water.

#### 3.8 Barium diphenylamine sulphonate, 2 g/l solution.

Dissolve 0,5 g of the salt in 250 ml of H<sub>2</sub>SO<sub>4</sub>.

#### 3.9 Hydrogen peroxide, 30 g/l solution.

**3.10 Hydrogen sulfide gas (H<sub>2</sub>S).****3.11 Hydroxylamine hydrochloride or hydroxylammonium chloride**, 100 g/l solution.

Dissolve 50 g of CINH<sub>3</sub>OH in 500 ml of water. Filter if necessary.

**3.12 Mercuric chloride (HgCl<sub>2</sub>)**, saturated solution.**3.13 Methyl red indicator**, 1 g/l solution.

Dissolve 0,1 g methyl red in 100 ml of methanol.

**3.14 o-Phenanthroline (1,10-phenanthroline)**, 1 g/l solution.

Dissolve 1 g o-phenanthroline monohydrate in 15 ml of ethanol (95 %). Dilute to 1 l with water.

**3.15 Potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>)**, 0,01 mol/l solution.

Dissolve 2,942 4 g of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in water. Make up the solution to the 1 l mark of a volumetric flask. Standardize the solution against a sample with a certified iron content.

**3.16 Potassium permanganate (KMnO<sub>4</sub>)**, standardized solution,  $c(1/5 \text{ KMnO}_4) = 0,01 \text{ mol/l}$ .

Dissolve 1,580 0 g of KMnO<sub>4</sub> in 500 ml of water. Allow to stand for 1 d-2 d. Filter through an asbestos mat and dilute to the 1 l mark of a volumetric flask with water. Standardize against sodium oxalate.

**3.17 Potassium permanganate (KMnO<sub>4</sub>)**, 50 g/l solution.

Dissolve 5 g KMnO<sub>4</sub> in 100 ml of water.

**3.18 Potassium pyrosulfate (K<sub>2</sub>S<sub>2</sub>O<sub>7</sub>)**.**3.19 Anhydrous sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>)**.**3.20 Sodium tetraborate (Na<sub>2</sub>B<sub>4</sub>O<sub>7</sub>)**.**3.21 Stannous chloride (SnCl<sub>2</sub>)**, 50 g/l solution.

Dissolve 50 g of SnCl<sub>2</sub> in 100 ml of HCl. Make up to the 1 l mark of a volumetric flask with water. Store the solution with metallic tin in the bottle.

## 4 Apparatus

**4.1 Small jaw or roll crusher with hardened steel faces.****4.2 Tool steel mortar.**

In situations where it is vital that iron contamination be eliminated, the crushing should be carried out in a tungsten carbide mortar.

**4.3 Sieve**, with mesh of 150 μm aperture size.

**4.4 Sieve**, with 2 mm aperture size.

**4.5 Platinum crucible**, weighed, 30 ml, with lid.

## 5 Procedure

### 5.1 Preparation of samples for analysis

#### 5.1.1 Abrasive crude

The sample, carefully obtained by means of an approved sampling method, is crushed in a small jaw or roll type crusher with hardened steel faces until it can be passed through a sieve having a mesh size of 2 mm; it is then mixed and divided by coning and quartering until approximately 500 g are obtained. The 500 g sample is in turn mixed and divided by coning and quartering until a sample weighing between 10 g and 20 g is obtained. This 20 g sample is then pulverized in a tool steel mortar until, unless otherwise specified, it completely passes through a sieve having a mesh size of 150 µm; it is then mixed thoroughly and placed in a container which will ensure freedom from contamination.

Precautions shall be taken in order to prevent the sample being contaminated by steel particles from the sampling and crushing equipment. Magnetic separation of the sample shall be carried out in order to remove this iron contamination.

#### 5.1.2 Abrasive grain

The sample, carefully obtained by one of the approved methods, is thoroughly mixed and divided by coning and quartering until 10 g to 20 g are obtained. It is then crushed until, unless otherwise specified, it completely passes through a sieve having a mesh size of 150 µm; it is then carefully mixed and placed in a container which will ensure freedom from contamination (see ISO 9138).

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### 5.2 Blank determination

Blank determination on the reagents shall be made for each determination and the necessary corrections applied in each case.

## 6 Gain or loss on ignition

### 6.1 Procedure

Weigh a 1 g sample to within 0,000 1 g and place it in a weighed platinum crucible which has been previously heated to 1 100 °C. Record the mass of the sample and the crucible. Heat the crucible plus sample at 1 100 °C until a constant mass is obtained. Cool the crucible in a desiccator and weigh the crucible plus sample.

### 6.2 Expression of results

Loss on ignition, expressed as a percentage, is given by the formula

$$\frac{(m_1 - m_2)}{m_0} \times 100$$

where

$m_1$  is the mass of the crucible plus sample before ignition, in grams;



$m_2$  is the mass of crucible plus sample after ignition, in grams;

$m_0$  is the mass of sample, in grams.

NOTE — If the sample gains in mass, report as a gain, and use the formula:

$$\frac{(m_2 - m_1)}{m_0} \times 100$$

## 7 Determination of silicon dioxide

### 7.1 Gravimetric method

#### 7.1.1 Apparatus

7.1.1.1 **Platinum crucible**, with lid.

7.1.1.2 **Beaker**, 400 ml.

7.1.1.3 **Filter paper**, medium grade.

7.1.1.4 **Desiccator**.

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#### 7.1.2 Procedure

Weigh a 1 g sample to within 0,000 1 g and place in a platinum crucible containing 3 g of  $\text{Na}_2\text{CO}_3$  and 3 g of  $\text{Na}_2\text{B}_4\text{O}_7$ . Mix the sample and fuse mixture thoroughly. Cover the crucible with a platinum lid and fuse the mixture at 1 000 °C until the entire sample is in solution. Rotate the crucible during cooling to deposit the melt in a thin layer on the side of the crucible. Place crucible and lid in a 400 ml beaker.

Dissolve the fusion in 100 ml of  $\text{H}_2\text{SO}_4$  (1 + 4). When fully dissolved, thoroughly wash crucible and lid, adding washes to main solution.

Carefully evaporate the solution to fumes of  $\text{SO}_3$  and leave to cool. Add 100 ml of water and boil to dissolve salts. Filter using a medium grade ashless filter paper. Wash the paper and precipitate with hot  $\text{H}_2\text{SO}_4$  (2 + 98). Retain the filtrate for the  $\text{Fe}_2\text{O}_3$ ,  $\text{TiO}_2$ ,  $\text{CaO}$  and  $\text{MgO}$  determinations. Transfer the filter paper to a platinum crucible and char at low heat. When fully charred ignite at 1 000 °C. Leave the crucible to cool in a desiccator and weigh to constant mass.

Moisten the solid mass with 1 or 2 drops of water, add 10 ml of HF acid and 2 or 3 drops of  $\text{H}_2\text{SO}_4$  (1 + 1). Slowly evaporate to dryness then ignite at 1 000 °C. Cool the crucible in a desiccator and weigh to constant mass. Evaluate the loss in mass due to HF,  $\text{H}_2\text{SO}_4$  treatment.

#### 7.1.3 Expression of results

Silicon dioxide content, expressed as a percentage by mass, is given by the equation

$$w(\text{SiO}_2) = \frac{m_1}{m_0} \times 100$$

where

$m_1$  is the mass loss due to the HF,  $\text{H}_2\text{SO}_4$  treatment, in grams;

$m_0$  is the mass of the sample, in grams.

## 7.2 Colorimetric method

### 7.2.1 Reagents

#### 7.2.1.1 Molybdic acid solution.

Dissolve 100 g of ammonium molybdate [82 % (*m/m*) of  $\text{MoO}_3$ ] in approximately 600 ml of water. Add 70 ml of  $\text{H}_2\text{SO}_4$  ( $\rho = 1,84$  g/ml). Filter the solution. Dilute to 1 l with water in a volumetric flask.

#### 7.2.1.2 Reducing solution.

Dissolve 25 g of sodium hydrogen sulphite ( $\text{NaHSO}_3$ ) in 25 ml of water. Dissolve 2 g of sodium sulphite ( $\text{Na}_2\text{SO}_3$ ) and 0,4 g of 1-amino-2-naphthol-4-sulphonic acid in 25 ml of water.

Mix the two solutions and dilute to 250 ml with water in a volumetric flask. Note that this solution has a maximum shelf life of two weeks.

#### 7.2.1.3 Silicon dioxide, standard solution corresponding to 0,1 mg $\text{SiO}_2$ per millilitre.

Melt 0,100 g of anhydrous  $\text{SiO}_2$  and 1 g of  $\text{Na}_2\text{CO}_3$  in a platinum crucible. Cool the molten mass. Dissolve in water and make up to 1 000 ml in a volumetric flask. Transfer to a polyethylene bottle.

#### 7.2.1.4 Tartaric acid, 100 g/l solution.

Dissolve 100 g of tartaric acid in water. Dilute to 1 000 ml in a volumetric flask.

**7.2.1.5 Fusion mixture**, comprising 3 parts  $\text{Na}_2\text{CO}_3$  or 1 part  $\text{B}_2\text{O}_3$ . (Boric oxide is the preferred reagent. Salts containing boron have varying amounts of  $\text{SiO}_2$  giving high and frequently inconsistent blank values.)

#### 7.2.1.6 Hydrofluoric acid, 2 % (V/V) solution (for cleaning PTFE).

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### 7.2.2 Apparatus

#### 7.2.2.1 Platinum crucible.

#### 7.2.2.2 PTFE beaker, 250 ml.

#### 7.2.2.3 Magnetic stirrer.

#### 7.2.2.4 PTFE stirring bar

#### 7.2.2.5 pH-meter.

#### 7.2.2.6 Volumetric flask, 100 ml.

#### 7.2.2.7 Spectrophotometer.

### 7.2.3 Procedure

#### 7.2.3.1 Sample size and dilutions

Use the table 1 to determine appropriate sample test portions and dilution.

#### 7.2.3.2 Preparation of test solution

Weigh 5 g of fusion mixture into a 30 ml platinum crucible then weigh the appropriate size sample (see 7.2.3.1) into the crucible and cover with an additional 5 g of fusion mixture. Melt over a burner using platinum or nichrome triangles. Cool the melt to room temperature. Wash the outside of the crucible with water. Place the crucible in a

250 ml PTFE beaker and add enough water to cover the crucible. Bring to the boil until the melt is dissolved. Cool to room temperature and dilute to approximately 175 ml to 200 ml. Place the PTFE stirring bar in the beaker and place on a magnetic stirrer.

Using a pH-meter, adjust very carefully the pH of the solution to 1,8 using H<sub>2</sub>SO<sub>4</sub> (1 + 1). **(Do not allow pH to go below 1,8.)**

If pH goes below 1,8 polymerization of the silicic acid starts to take place and polymerized silicic acid will not react to form silicomolybdic acid capable of being reduced to form the proper colour.

Transfer the solution to a volumetric flask (see 7.2.3.1 for flask size), dilute to the mark with water and mix.

Pipette an appropriate aliquot (see 7.2.3.1) into a 100 ml volumetric flask to which is added in the following order:

- 1) 2 ml of freshly prepared molybdic acid solution (see 7.2.1.1). Mix and allow to stand for 7 min;
- 2) 10 ml of tartaric acid solution (see 7.2.1.4);
- 3) 2 ml of reducing solution (see 7.2.1.2). Mix and allow to stand for 30 min.

Read absorbance at a wavelength of 700 nm, using 1 cm cells (for best accuracy, absorbance shall be in the range of 0,2 to 0,6).

Table 1

Assumed SiO <sub>2</sub> content %	Mass of test portion g	Dilution	Dilution factor <i>d</i>
0,1 to 0,5	1	250 ml – 25 ml	10
0,5 to 2,0	0,5	250 ml – 10 ml	25
1,0 to 5,0	0,2	250 ml – 10 ml	25
3,0 to 10,0	0,1	250 ml – 10 ml	25
10 to 20	0,1	500 ml – 10 ml	50
15 to 40	0,1	250 ml – 25 ml	10
		250 ml – 2,5 ml	100

### 7.2.3.3 Preparation of calibration curve

Prepare a series of standard solutions to cover the expected range of silicon dioxide concentrations. Determine the absorbance of the solutions as described for the sample in 7.2.3.2. Prepare a calibration curve by plotting the absorbance values for the standard solutions against the concentration of SiO<sub>2</sub>, in grams, per 100 ml of solution.

### 7.2.4 Expression of results

Silica content, expressed as a percentage by mass is given by the equation

$$w(\text{SiO}_2) = \frac{m_1 \times d}{m_0} \times 100$$

where

*d* is the dilution factor (see 7.2.3.1);

*m*<sub>1</sub> is the mass, in grams, of silicon dioxide per 100 ml of sample solution as found by interpolation from the calibration curve;

*m*<sub>0</sub> is the mass of the sample, in grams.