
**Chemical analysis of refractories —
General requirements for wet chemical
analysis, atomic absorption spectrometry
(AAS) and inductively coupled plasma
atomic emission spectrometry (ICP-AES)
methods**

*Analyse chimique des matériaux réfractaires — Exigences générales
pour les méthodes d'analyse chimique par voie humide, par
spectrométrie d'absorption atomique (AAS) et par spectrométrie
d'émission atomique avec plasma induit par haute fréquence (ICP-AES)*



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Contents

Page

Foreword.....	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	2
4 Apparatus	2
5 Reagents	6
6 Sampling	11
7 Sample mass	12
8 Basic procedure	12
9 Determination of loss on ignition (gravimetric)	12
10 Expression of test results	13
11 Examination and adoption of test results	13
12 Test report	14

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 26845 was prepared by Technical Committee ISO/TC 33, *Refractories*.

It is to be used in conjunction with ISO 10058-1, ISO 10058-2 and ISO 10058-3, ISO 20565-1, ISO 20565-2 and ISO 20565-3, ISO 21079-1, ISO 21079-2 and ISO 21079-3, and ISO 21587-1, ISO 21587-2 and ISO 21587-3.

Introduction

This International Standard gives the general requirements common to the standards used for the chemical analysis of refractories and refractory products, i.e.:

ISO 10058, *Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method)*:

- Part 1: Apparatus, reagents, dissolution and gravimetric silica
- Part 2: Wet chemical analysis
- Part 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)

ISO 20565 *Chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials (alternative to the X-ray fluorescence method)* —

- Part 1: Apparatus, reagents, dissolution and gravimetric silica
- Part 2: Wet chemical analysis
- Part 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)

ISO 21079 *Chemical analysis of refractories containing alumina, zirconia and silica — Refractories containing 5 % to 45 % of ZrO₂ (alternative to the X-ray fluorescence method)* —

- Part 1: Apparatus, reagents and dissolution
- Part 2: Wet chemical analysis
- Part 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma emission spectrometry (ICP-AES)

ISO 21587 *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method)* —

- Part 1: Apparatus, reagents, dissolution and gravimetric silica
- Part 2: Wet chemical analysis
- Part 3: Inductively coupled plasma and atomic absorption spectrometry methods

These International Standards give wet chemical, AAS and ICP methods for the analysis of refractory materials and products. They are to be used as an alternative to ISO 12677, when the laboratory does not have an XRF instrument or its instrument does not meet the requirements of ISO 12677: *Chemical analysis of refractory products by XRF — Fused cast bead method*.

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Chemical analysis of refractories — General requirements for wet chemical analysis, atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods

1 Scope

This International Standard specifies apparatus, reagents, sampling, sample preparation, terms and definitions, basic procedures, loss on ignition and reporting of results applicable to the following standards, which are used for the chemical analysis of refractory products and raw materials by wet chemical, AAS and ICP-AES:

ISO 10058-1, ISO 10058-2 and ISO 10058-3;

ISO 20565-1, ISO 20565-2 and ISO 20565-3;

ISO 21079-1, ISO 21079-2 and ISO 21079-3;

ISO 21587-1, ISO 21587-2 and ISO 21587-3.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 31-0, *Quantities and units — Part 0: General principles*

ISO 836, *Terminology for refractories*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 5022, *Shaped refractory products — Sampling and acceptance testing*

ISO 6286, *Molecular absorption spectrometry — Vocabulary — General — Apparatus*

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

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

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

ISO 6955, *Analytical spectroscopic methods — Flame emission, atomic absorption, and atomic fluorescence — Vocabulary*

ISO 8656-1, *Refractory products — Sampling of raw materials and unshaped products — Part 1: Sampling scheme*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 836 and the following apply.

3.1

dry unshaped refractories

particles and/or powder of unshaped refractories without liquid (mortar and mixture of refractory aggregate and pitch or resin)

3.2

wet unshaped refractories

particles and/or powder of unshaped refractories with liquid (mortar and mixture of refractory aggregate and pitch or resin)

3.3

alumina-zirconia-silica refractories

refractories in which aluminium oxide (1 % to 80 % by mass), zirconium oxide (including hafnium oxide) (5 % to 50 % by mass) and silicon(IV) oxide (0,1 % to 45 % by mass) are used as chemical components

4 Apparatus

Standard laboratory apparatus and the following.

4.1 Spectrometers

4.1.1 Atomic absorption (AA) spectrometer, conforming to the requirements of ISO 6955. An instrument which can be operated using a dinitrogen oxide/acetylene flame for the determinations of aluminium oxide and calcium oxide is appropriate.

4.1.2 Flame emission spectrophotometer, comprising a stand-alone flame emission instrument or an AA spectrometer used in emission mode.

4.1.3 Inductively coupled plasma atomic emission (ICP-AE) spectrometer.

4.1.4 Molecular absorption spectrometer, conforming to the requirements of ISO 6286.

4.1.5 Photometer, with a 1 cm cell.

4.2 Heaters

4.2.1 Burner, such as a Mecker burner or a Bunsen burner.

4.2.2 Electric muffle furnace, suitable for use above 1 150 °C.

4.2.3 Sand bath, comprising an electrically heated or burner-heated iron plate with sand, which can be heated to the temperature at which white smoke of sulfuric acid occurs.

4.2.4 Steam bath, electric- or burner-type, which can be controlled at the appropriate temperature.

4.3 Devices

4.3.1 Analytical electronic balance, readable to the nearest 0,1 mg.

4.3.2 Magnetic stirrer, comprising a stirring system using a magnetic rotating bar coated with ethylene-4-fluoride resin.

4.3.3 Filter paper, ashless, medium pore.

4.3.4 Cation-exchange resin column, consisting of plastic wool (10 mm) packed into a plastic tube (diameter 12 mm × 200 mm) with a funnel attached to the top and an eluent outlet sharp-pointed to the bottom.

Pour a slurry (18 ml) of the water-expanded strongly acidic cation-exchange resin [divinylbenzene 8 % (DVB), 75 µm to 150 µm] into the column. Adjust the flow rate to 1,0 ml to 1,5 ml per minute by the packing condition of the plastic wool. Wash the column with hydrochloric acid (1+2, 120 ml) and water (70 ml). An example of a normal column is shown in Figure 1.

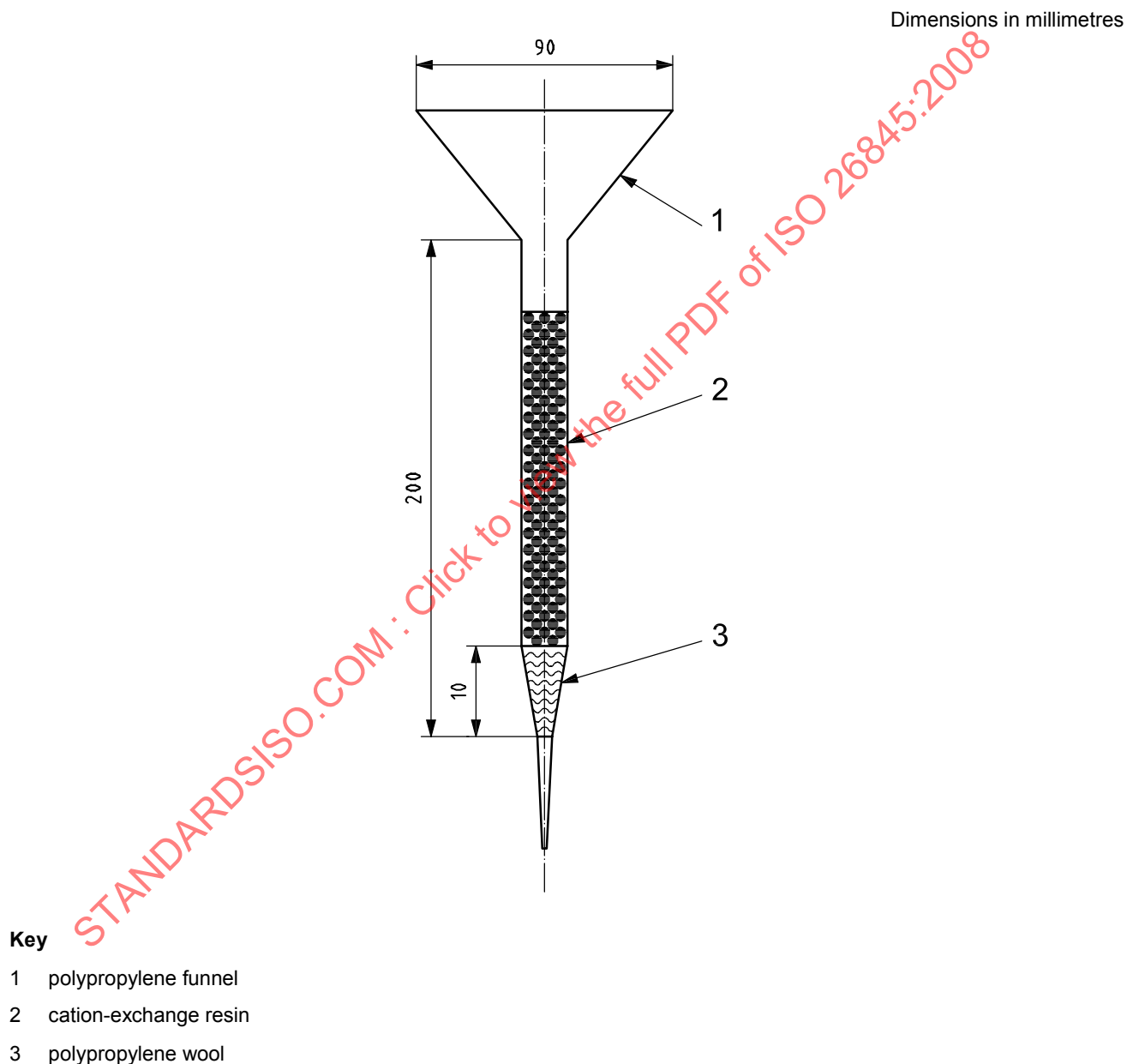


Figure 1 — Example of a cation-exchange resin column

4.4 Platinum ware

The platinum apparatus may be of platinum or platinum alloy.

4.4.1 Platinum crucible, with a volume of 20 ml or 30 ml.

4.4.2 Platinum dish, with the diameters of the base and wall almost the same, and a height of approximately half the diameter. 75 ml or 150 ml volume are commonly used sizes.

4.5 Glassware

4.5.1 Burette, with a 0,1 ml scale and a maximum volume of 50 ml.

4.5.2 Conical flask, capable of holding a volume of 500 ml and 1 l.

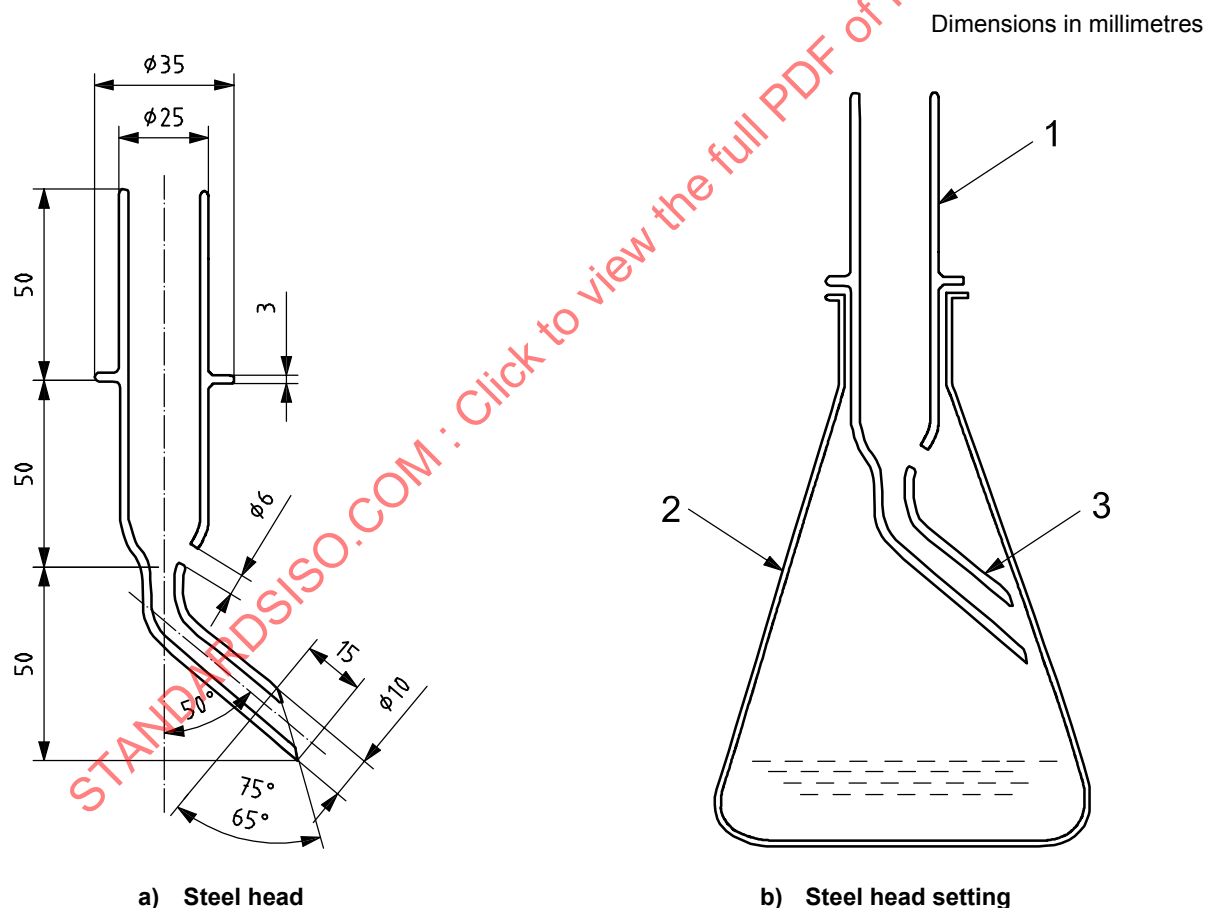
4.5.3 Desiccator, containing dried silica gel as the drying agent.

4.5.4 Erlenmeyer flask, capable of holding a volume of 500 ml.

4.5.5 Glass beakers, with a range of appropriate volumes (100 ml, 200 ml, 300 ml, 400 ml, 500 ml, etc.).

4.5.6 One-mark pipette, suitable for the transfer of each sample solution or standard solution.

4.5.7 Reflux steel head, comprising a funnel, with a long leg capable of being bent so that the end touches the inside wall of a flask (see Figure 2).



Key

- 1 steel head
- 2 erlenmeyer flask
- 3 head that contacts internal wall

Figure 2 — Steel head for reflux

4.5.8 Separating funnels, 250 ml and 500 ml, with stoppers.

4.5.9 Volumetric flasks, made of glassware (100 ml, 250 ml, 500 ml, 1 000 ml, etc.) as appropriate for each solution.

4.5.10 Volumetric pipette, of capacity 5 ml.

4.5.11 Watch glass, of diameter 75 mm.

4.6 Porcelain ware

4.6.1 Porcelain basin, with a diameter of 125 mm.

4.6.2 Porcelain crucible, capable of holding a volume of 15 ml.

4.7 Plastic ware

4.7.1 Plastic beaker, of capacity 100 ml.

4.7.2 Polyethylene tetrafluoride beaker, of capacity 200 ml. Heat in nitric acid for at least 2 h and wash in water.

NOTE Instead of a polyethylene tetrafluoride beaker, a 150 ml platinum dish can be used.

4.7.3 Volumetric flasks, made of plastic, as appropriate for each solution, and calibrated as follows. Either

- wash a plastic flask thoroughly, invert it and allow it to stand to dry naturally, or
- wash a plastic flask with water, ethanol and diethylether and dry it by blowing air into it.

Cut 20 graduations from a sheet of section paper (1 mm squares) into a strip and attach it to the marked line of the plastic flask, so that the line coincides with the central line of the paper. Weigh the flask to the nearest milligram.

Add water at approximately room temperature up to the lower end (B) of the strip, and weigh the flask. Then add more water up to the upper end (A) of the strip, and weigh the flask. Separately, measure the water temperature, in degrees Celsius, the room temperature, in degrees Celsius and the atmospheric pressure, in kilopascals, and obtain the correct marked line, S , the number of graduations counted from the bottom edge (B) of the graduation paper, using the following equation:

$$S = \frac{\left[\frac{1\,000\,000 - (m + m')}{f} \right] - m_B}{\frac{m_A - m_B}{20}}$$

where

m_A is the mass of water up to the top edge (A) of the graduated paper, in milligrams, [i.e. (mass obtained by second weighing) – (mass of Erlenmeyer flask)]; $m_A = m_B + [\text{mass of water from (A) to (B)}]$;

m_B is the mass of water up to to bottom edge (B) of the graduated paper, in milligrams, [i.e. (mass obtained by first weighing) – (mass of Erlenmeyer flask)] ;

m is the correction value, in milligrams, at 20 °C room temperature, and 101,325 kPa {760 mmHg} atmospheric pressure;

m' is the correction value, in milligrams, due to deviation from a room temperature of 20 °C, and an atmospheric pressure of 101,325 kPa [760 mmHg]; it is $\pm 4,0$ mg per ± 1 °C room temperature, and $\pm 1,3$ mg per $\pm 0,133$ kPa (1 mmHg) atmospheric pressure;

f is a constant given by
$$\frac{100}{\text{normal capacity of plastic flask to be calibrated}}$$

Mark the new line on the strip.

NOTE When the correct line is shown on the flask, the strip can be removed.

4.8 Miscellaneous

4.8.1 Electric muffle furnace, capable of being controlled at temperatures up to at least 1 100 °C.

4.8.2 Desiccator.

4.8.3 pH meter, with a glass electrode.

5 Reagents

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

Reagents should conform to the requirements of ISO 6353-1, ISO 6353-2 and ISO 6353-3, as appropriate. Specific requirements for reagents are given in the appropriate clause.

5.1 Stock reagents

5.1.1 Acetic acid, concentrated, (ISO 6353-2, R 1) minimum mass fraction 99,7 %.

5.1.2 Aluminium chloride solution, Al (25 mg/ml). Dissolve 123,5 g of aluminium chloride in water and dilute to 1 l with water.

5.1.3 Aluminium oxide solution, Al₂O₃, 10 mg/ml. Weigh 5,3 g of aluminium (minimum mass fraction 99,9 %) in a 300 ml beaker and cover with a watch glass. Then add 250 ml of hydrochloric acid (1+1) and heat on a steam bath until dissolved. Remove the watch glass and evaporate to 30 ml to remove excess hydrochloric acid. After cooling, add water and dilute to 1 000 ml in a volumetric flask with water.

5.1.4 Ammonia solution, concentrated (ISO 6353-2, R 3), minimum mass fraction 25 %.

5.1.5 Ammonia solution (1+1). Add 1 volume of ammonia solution (concentrated) to 1 volume of water.

5.1.6 Ammonia solution (1+9). Add 1 volume of ammonia water (concentrated) to 9 volumes of water.

5.1.7 Ammonium acetate buffer solution. Add 120 ml of acetic acid to 500 ml of water, followed by 74 ml of ammonia solution (concentrated) (5.1.4). Dilute to 1 l.

5.1.8 Ammonium acetate solution, 100 g/l. Dissolve 100 g of ammonium acetate in water and dilute to 1 l.

5.1.9 Ammonium acetate solution, 200 g/l. Dissolve 200 g of ammonium acetate in water and dilute to 1 l.

5.1.10 Ammonium chloride.

5.1.11 Ammonium chloride solution, 20 g/l. Dissolve 10 g of ammonium chloride in 500 ml of water, add a drop of methyl red solution and drop in ammonia solution (1+9) (5.1.6) until the colour turns yellow. If the colour becomes red again due to heating, drop in ammonia solution (1+9).

5.1.12 Ammonium ceric nitrate solution, 10 g/l. Dissolve 5 g of ammonium ceric nitrate in about 200 ml of water. Cautiously add 7 ml of sulfuric acid, cool, dilute to 500 ml with water and mix.

5.1.13 Ammonium molybdate solution, 100 g/l. Dissolve 10 g of ammonium molybdate tetrahydrate in 100 ml of water, and filter if necessary. Store in a plastic bottle. Discard after 4 weeks.

5.1.14 Ammonium molybdate solution, 80 g/l. Dissolve 40 g of ammonium molybdate tetrahydrate in 500 ml of water, and filter if necessary. Store in a plastic bottle. Discard after 4 weeks or earlier if any visible deposit of molybdic acid is observed.

5.1.15 Ammonium molybdate solution, 20 g/l. Dissolve 2 g of ammonium molybdate tetrahydrate in 20 ml of hot water, filter if necessary and dilute to 100 ml with the addition of 60 ml of sulfuric acid (1+1) and water.

5.1.16 L(+)-ascorbic acid solution, 100 g/l. Dissolve 10 g of L (+)-ascorbic acid in water and dilute to 100 ml. This solution shall be kept in the dark and at a low temperature. Discard after 2 weeks.

5.1.17 Boric acid, H_3BO_3 .

5.1.18 Boric acid solution, 40 g/l. Dissolve 40 g of boric acid in water and dilute to 1 l.

5.1.19 Buffer solution, (pH 10). Dissolve 70 g of ammonium chloride in 570 ml of ammonia solution (concentrated) (5.1.4) and dilute to 1 000 ml with water.

5.1.20 Chloroform, (ISO 6353-2, R7) minimum mass fraction 99 %.

5.1.21 Chromium(III) oxide solution, Cr_2O_3 10 mg/ml. Weigh 6,8 g of chromium (minimum mass fraction more than 99,9 %) in a 300 ml beaker and cover with a watch glass. Add 250 ml of hydrochloric acid (1+1) and heat until dissolved on a steam bath. Remove the watch glass and evaporate until 30 ml remains, to remove excess hydrochloric acid. Cool and transfer to a 1 000 ml volumetric flask and dilute to the mark with water.

5.1.22 Cupferron, 60 g/l. Dissolve 6 g of cupferron in 100 ml of water. Filter if necessary. Prepare this solution freshly when required. Store the solid reagent in a tightly stoppered bottle, in the presence of a piece of ammonium carbonate to prevent decomposition.

5.1.23 CyDTA solution, 8 g/l. Add 4,6 ml of sodium hydroxide solution, 100 g/l, to 2 g of cyclohexanediamine tetraacetate hydrate and dissolve by heating. After cooling, dilute to 250 ml with water.

5.1.24 Diantiprylmethane (DAM) solution, 10 g/l. Dissolve 1 g of diantiprylmethane monohydrate in 30 ml of hydrochloric acid (1+50), and dilute to 100 ml with water.

5.1.25 Diethyl ether.

5.1.26 Diphenylcarbazide solution, 10 g/l. Dissolve 0,1 g of diphenylcarbazide in 10 ml of acetone. It is essential to prepare this solution immediately before use.

5.1.27 Eluent A, 0,8 mol/l hydrofluoric acid, 0,65 mol/l boric acid. Dissolve 40 g of boric acid in 960 ml of water, pour the solution into a plastic vessel (1 l) and add 35 ml of hydrofluoric acid (minimum mass fraction 46 %). Mix by shaking.

5.1.28 Eluent B. Combine 995 ml of water and 5 ml of hydrofluoric acid (minimum mass fraction 46 %) in a suitably sized plastic vessel and mix by shaking.

5.1.29 Filter-paper pulp, powdery.

5.1.30 Ethanol, minimum mass fraction 95 %.

5.1.31 Fusion mixture. Mix anhydrous sodium and potassium carbonates in equimolar proportions.

5.1.32 Hexamethylenetetramine (hexamine).

5.1.33 Hydrazinium dichloride solution, 150 g/l. Dissolve 15 g of hydrazinium dichloride in water and dilute to 100 ml.

5.1.34 Hydrochloric acid, concentrated, (ISO 6353-3, R 13), minimum mass fraction 35 %.

5.1.35 Hydrochloric acid (1+1). Add 1 volume of hydrochloric acid, concentrated (5.1.32) to 1 volume of water.

5.1.36 Hydrochloric acid (1+2). Add 1 volume of hydrochloric acid, concentrated (5.1.32) to 2 volumes of water.

5.1.37 Hydrochloric acid (1+3). Add 1 volume of hydrochloric acid, concentrated (5.1.32) to 3 volumes of water.

5.1.38 Hydrochloric acid (1+4). Add 1 volume of hydrochloric acid, concentrated (5.1.32) to 4 volumes of water.

5.1.39 Hydrochloric acid (1+9). Add 1 volume of hydrochloric acid, concentrated (5.1.32) to 9 volumes of water.

5.1.40 Hydrochloric acid (1+20). Add 1 volume of hydrochloric acid, concentrated (5.1.32) to 20 volumes of water.

5.1.41 Hydrochloric acid (1+50). Add 1 volume of hydrochloric acid, concentrated (5.1.32) to 50 volumes of water.

5.1.42 Hydrochloric acid (1+150). Add 1 volume of hydrochloric acid, concentrated (5.1.32) to 150 volumes of water.

5.1.43 Hydrofluoric acid, concentrated, (ISO 6353-3, R 67), minimum mass fraction 40 % to 48 %.

5.1.44 Hydrofluoric acid (1+9). Add 1 volume of hydrofluoric acid, concentrated (5.1.41) to 9 volumes of water. Store in a plastic container.

5.1.45 Hydrogen peroxide solution, minimum mass fraction 30 %.

5.1.46 Hydrogen peroxide solution, minimum mass fraction 6 %.

5.1.47 Hydroxyammonium chloride solution, 100 g/l. Dissolve 10 g of hydroxyammonium chloride in warm water, filter if necessary, cool and dilute to 100 ml.

5.1.48 Iron(III) oxide solution, Fe_2O_3 , 10 mg/ml. Weigh 7,0 g of iron (minimum mass fraction 99,9 %) in a 300 ml beaker and cover with a watch glass. Then add 250 ml of hydrochloric acid (1+1) and heat until dissolved on a steam bath. Remove the watch glass and evaporate until 30 ml remains, to remove excess hydrochloric acid. After cooling, transfer to a 1 000 ml volumetric flask, and dilute to the mark with water.

5.1.49 Iron(III) chloride solution. Dissolve 1 g of iron(III) chloride hexahydrate in 100 ml of hydrochloric acid (1+50) (5.1.39).

5.1.50 Lanthanum solution, 50 g/l. Weigh 50 g of lanthanum(III) oxide into a 1 000 ml beaker. Carefully add 200 ml of hydrochloric acid (1+1) and dissolve while heating. Dilute to 1 000 ml with water. If the reaction is too violent on addition of hydrochloric acid (1+1), move the beaker into flowing water to cool.

5.1.51 Magnesium oxide solution, MgO , 10 mg/ml. Weigh 6 g of magnesium metal (minimum mass fraction 99,9 % and a mass fraction of Si less than 0,001 %), transfer to a 500 ml beaker and cover with a watch glass. Add 100 ml of hydrochloric acid (1+1), and heat on a steam bath until dissolved. After cooling, transfer to a 1 000 ml volumetric flask, and dilute to the mark with water.

5.1.52 DL-mandelic acid.

5.1.53 Mixed acid. Mix sulfuric acid (1+1) and phosphoric acid with a 1:1 ratio.

5.1.54 Nitric acid, concentrated, minimum mass fraction 65 %.

5.1.55 Nitric acid (1+1). Add one volume of nitric acid, concentrated (5.1.52) to one volume of water.

5.1.56 Nitric acid (1+3). Add one volume of nitric acid, concentrated (5.1.52) to three volumes of water.

5.1.57 Nitric acid (1+20). Add one volume of nitric acid, concentrated (5.1.52) to twenty volumes of water.

5.1.58 2,2', 2''-Nitrilotriethanol (1+1). Add 1 volume of 2,2', 2''-nitrilotriethanol to one volume of water.

5.1.59 Perchloric acid, concentrated, minimum mass fraction 70 %.

5.1.60 1,10-Phenanthroline chloride solution, 1 g/l. Dissolve 0,1 g of 1,10-phenanthroline chloride monohydrate in water, dilute to 100 ml, and keep in a dark and cool place. Discard the solution if it becomes coloured during storage.

5.1.61 1,10-Phenanthroline chloride solution, 10 g/l. Dissolve 1 g of 1,10-phenanthroline chloride monohydrate in 100 ml of water, and keep in a dark and cool place. Discard the solution if it becomes coloured during storage.

5.1.62 Ortho-phosphoric acid, minimum mass fraction 98 %.

5.1.63 Phosphoric acid, concentrated, minimum mass fraction 85 %.

5.1.64 Phosphoric acid (2+3). Add 2 volumes of phosphoric acid, concentrated (5.1.61) to 3 volumes of water, then mix and leave to cool.

5.1.65 Phosphoric acid (1+9). Add 1 volume of phosphoric acid, concentrated (5.1.61) to 9 volumes of water, then mix and leave to cool.

5.1.66 Partially hydrolysed polyacrylamide, aqueous solution, 20 g/l.

5.1.67 Polyethylene oxide solution, 2,5 g/l. Add 0,5 g of polyethylene oxide to 200 ml of water slowly while stirring, preferably on a mechanical stirrer, until dissolved. Discard after two weeks.

5.1.68 Polyethylene oxide solution, 0,5 g/l. Add 0,1 g of polyethylene oxide to 200 ml of water slowly while stirring, preferably on a mechanical stirrer, until dissolved. Discard after two weeks.

5.1.69 Potassium hydroxide solution, 250 g/l. Dissolve 250 g of potassium hydroxide in water, cool and dilute to 1 l. Store in a polyethylene bottle.

5.1.70 Potassium chloride.

5.1.71 Potassium disulfate, pure.

5.1.72 Potassium periodate.

5.1.73 Potassium permanganate solution, 3 g/l. Dissolve 0,6 g of potassium permanganate in 200 ml of water. Store in the dark.

5.1.74 Silver nitrite solution, 10 g/l. Store the reagent in a brown bottle.

5.1.75 Sodium azide solution, 20 g/l. Dissolve 2 g of sodium azide in 100 ml of water and mix.

5.1.76 Sodium carbonate, anhydrous.

In some brands of this agent, a minute quantity of calcium oxide is included. When determining calcium oxide by inductively coupled plasma atomic emission spectrometry (ICP-AES), a pure high-quality agent should be used.

5.1.77 Sodium diethyldithiocarbamate solution, 100 g/l. Dissolve 5 g of sodium diethyldithiocarbamate cupferron in 50 ml of water. Filter if necessary. Prepare this solution freshly when required.

5.1.78 Sodium diphenylamine-4-sulfonate solution, 2 g/l.

5.1.79 Sodium hydroxide solution, 100 g/l. Cautiously, to avoid a vigorous reaction, dissolve 20 g of sodium hydroxide in 150 ml of water, while stirring and cooling. After cooling, dilute to approximately 200 ml.

5.1.80 Sodium sulfide solution, 100 g/l. Dissolve 1 g of sodium sulfide nonahydrate in 10 ml of water. Prepare this solution freshly when required.

5.1.81 Sulfuric acid, concentrated, (ISO 6353-2, R 37), minimum mass fraction 95 %.

5.1.82 Sulfuric acid solution (1+1). Cautiously add, while stirring, 200 ml of sulfuric acid, concentrated (5.1.79) to 200 ml of water, while cooling the solution.

5.1.83 Sulfuric acid solution (1+3). Cautiously add, while stirring, 200 ml of sulfuric acid, concentrated (5.1.79) to 600 ml of water, while cooling the solution.

5.1.84 Sulfuric acid solution (1+4). Cautiously add, while stirring, 50 ml of sulfuric acid, concentrated (5.1.79) to 200 ml of water, while cooling the solution.

5.1.85 Sulfuric acid solution (1+5). Cautiously add, while stirring, 50 ml of sulfuric acid, concentrated (5.1.79) to 250 ml of water, while cooling the solution.

5.1.86 Sulfuric acid solution (1+9). Cautiously add, while stirring, 20 ml of sulfuric acid, concentrated (5.1.79) to 180 ml of water, while cooling the solution.

5.1.87 Sulfuric acid solution (1+15). Cautiously add, while stirring, 20 ml of sulfuric acid, concentrated (5.1.79) to 300 ml of water, while cooling the solution.

5.1.88 Sulfuric acid solution (1+150). Cautiously add, while stirring, 5 ml of sulfuric acid, concentrated (5.1.79) to 750 ml of water, while cooling the solution.

5.1.89 L(+)-tartaric acid solution, 100 g/l. Dissolve 100 g of L(+)-tartaric acid with water and dilute to 1 l.

5.2 Indicators

5.2.1 Barium diphenylamine sulfonate.

5.2.2 Bromophenol blue, 1 g/l solution. Dissolve 0,4 g of sodium hydroxide in 100 ml of water to make NaOH 4 g/l. Grind 0,1 g of bromophenol blue with 1,5 ml of sodium hydroxide solution (4 g/l), dilute to 100 ml with water and mix.

5.2.3 Calcein indicator (screened), (3,3'-bis[N,N'-bis(carboxymethyl) aminomethyl fluorescein]). Mix, by grinding together, 0,2 g of calcein, 0,12 g of thymolphthalein and 20,0 g of potassium chloride.

5.2.4 2,4-Dinitrophenol, saturated solution. Dissolve 1 g of 2,4-dinitrophenol in 100 ml of hot water, cool and filter.

5.2.5 Dithizone indicator, 0,25 g/l. Dissolve 0,012 5 g of dithizone in 50 ml of ethanol (mass fraction 95 %). The solution will keep for at least a week.

5.2.6 Eriochrome Black T solution, 2 g/l. Dissolve 0,20 g of Eriochrome Black T in 100 ml of ethanol and add 0,5 g of hydroxylammonium chloride. Store in a black glass bottle with a stopper.

5.2.7 Methyl orange solution, 0,5 g/l. Dissolve 0,05 g of methyl orange in 100 ml of hot water, cool and filter.

5.2.8 Methyl thymol blue complexone indicator. Mix, by grinding together, 0,2 g of methyl thymol blue complexone and 20 g of potassium nitrate.

5.2.9 Methyl orange solution, 0,4 g/l. Dissolve 0,10 g of methyl orange C.I. 13025 in 50 ml of ethanol (minimum volume fraction 95 %) and dilute to 250 ml with water in accordance with ISO 6353-1:1982, 4.3.5. Keep this solution in a dark glass bottle.

5.2.10 Methyl red solution, 0,1 g/l. Warm 25 mg of finely powdered methyl red C.I. 13020 with 0,95 ml of sodium hydroxide solution (2 g/l) and 5 ml of ethanol (mass fraction 95 %), and dilute to 250 ml with water in accordance with ISO 6353-1:1982, 4.3.6. Keep this solution in a dark glass bottle.

5.2.11 *p*-Nitrophenol solution, 2 g/l. Dissolve 0,20 g of *p*-nitrophenol in 100 ml of water.

5.2.12 Solochrome Black 6B indicator, (as ElioChrome Blue-Black B). Mix, by grinding together, 0,5 g of Solochrome Black 6B and 20 g of sodium chloride.

5.2.13 Xylenol orange solution, 1 g/l. Dissolve 0,10 g of the xylenol orange with water and dilute to 100 ml. Keep this solution in the dark in a refrigerator. Discard after 4 weeks.

6 Sampling

6.1 General

Take a laboratory sample in accordance with ISO 5022 for shaped, fired refractory products, and with ISO 8656-1 for unshaped refractory products and raw materials, or by a procedure agreed between the parties concerned.

6.2 Refractory brick or its raw material

Crush the laboratory sample obtained in 6.1 to pass through a 6,7 mm sieve, conforming to the requirements of ISO 3310-1, and reduce to 100 g by riffing or quartering. Grind the reduced sample to pass through a 300 µm sieve conforming to the requirements of ISO 3310-1.

6.3 Unshaped refractory products

6.3.1 Dry unshaped refractory products

Carry out the procedures specified in 6.1 and 6.2.

6.3.2 Wet unshaped refractory products

Spread the laboratory sample obtained in 6.1 on a heat-resistant plate that will not react with the sample (e.g. polyethylene tetrafluoride plate) to form a charge of 10 mm thickness. Dry in an air-bath at $110\text{ °C} \pm 5\text{ °C}$ for at least 10 h. Crush the sample as described in 6.2.

6.4 Preparation of the test sample

Reduce the sample prepared using the procedure specified in 6.1 to about 10 g by quartering, and grind the sample to pass through a 106 µm sieve conforming to the requirements of ISO 3310-1. This is the test sample for analysis.