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**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  
determination of gravimetric silica**

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*Analyse chimique des produits réfractaires contenant du chrome et des  
matières premières contenant du chrome (méthode alternative à la  
méthode par fluorescence de rayons X) —*

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*Partie 1: Appareillage, réactifs, mise en solution et détermination de la  
teneur en silice par gravimétrie*



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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.

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 20565-1 was prepared by Technical Committee ISO/TC 33, *Refractories*, in collaboration with Technical Committee CEN/TC 187, *Refractory products and materials*.

ISO 20565 consists of the following parts, under the general title *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 determination of gravimetric silica*
- *Part 2: Wet chemical analysis*
- *Part 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES)*

# 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 determination of gravimetric silica

### 1 Scope

This part of ISO 20565 specifies methods for the chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials, using traditional (“wet”) methods, ICP-AES spectrometry and FAAS spectrometry. It covers apparatus, reagents, dissolution and determination of gravimetric silica.

It is applicable in the ranges of determination given in Table 1.

ISO 20565 provides alternatives to the X-ray fluorescence (XRF) method given in ISO 12677.

**Table 1 — Range of determination (% by mass)**

Component	Range
SiO <sub>2</sub>	0,5 to 10
Al <sub>2</sub> O <sub>3</sub>	2 to 30
Fe <sub>2</sub> O <sub>3</sub>	0,5 to 25
TiO <sub>2</sub>	0,01 to 1
MnO	0,01 to 1
CaO	0,01 to 3
MgO	15 to 85
Na <sub>2</sub> O	0,01 to 1
K <sub>2</sub> O	0,01 to 1
Cr <sub>2</sub> O <sub>3</sub>	2 to 60
ZrO <sub>2</sub>	0,01 to 0,5
P <sub>2</sub> O <sub>5</sub>	0,01 to 5
LOI	−0,5 to 5
NOTE These values are after the loss on ignition (LOI) has been taken into account.	

## 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 6353-1, *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 26845:2008, *Chemical analysis of refractories — General requirements for wet chemical analysis, atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 26845 apply.

## 4 Apparatus

Use normal laboratory apparatus and the following.

NOTE Other apparatus is defined in ISO 26845.

### 4.1 Polyethylene tetrafluoride beaker, 200 ml.

Heat in nitric acid for at least 2 h and wash in water.

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

### 4.2 Volumetric flasks, 100 ml each, made of plastics material as appropriate for each solution, calibrated as follows.

Wash the plastic flask and stand it to dry naturally, or wash it with water, ethanol and diethylether and dry it by sending air into it. Cut the 20 graduations off a sheet of section paper (1 mm<sup>2</sup>) into a strip and attach it on the marked line of the plastic flask with the central line of the paper. Weigh the flask to the nearest milligram.

Pour water (at a temperature approximately equal to the room temperature) up to the lower end (B) of the strip and weigh the flask. Then add water up to the upper end (A) of the strip and weigh the flask. Separately, measure the water temperature (°C), the room temperature (°C) and the atmospheric pressure (kPa). Obtain the correct marked line [i.e. the number of graduations counted from bottom edge (B) of graduation paper],  $S$ , by using Equation (1).

$$S = \frac{\left[ \frac{1000\,000 - (m + m')}{f} - m_B \right]}{\frac{m_A - m_B}{20}} \quad (1)$$

where

$m_A$  is the mass of water up to the top edge (A) of the graduation paper, in milligrams (mg), i.e. [(mass obtained by second weighing) — (mass of Erlenmeyer flask)]:

$$m_A = m_A + (\text{mass of water from A to B});$$

- $m_B$  is the mass of water up to bottom edge (B) of the graduation paper, in milligrams (mg), i.e. [(mass obtained by first weighing) — (mass of Erlenmeyer flask)];
- $m$  is the correction value, in milligrams (mg), at 20 °C room temperature, and 101,325 kPa atmospheric pressure;
- $m'$  is the correction value, in milligrams (mg), due to the deviation from room temperature of 20 °C, and an atmospheric pressure 101,325 kPa; it is  $\pm 4,0$  mg per  $\pm 1$  °C room temperature, and  $\pm 1,3$  mg per  $\pm 0,133$  kPa atmospheric pressure;
- $f$  is 100 ml (nominal capacity of plastic flask to be calibrated).

Change the paper strip for a fresh paper strip marked with the correct marked line made on the plastic flask. When using this flask, employ the marked line newly obtained above.

## 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 shall 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.

Reagents that are listed in ISO 26845 and the following.

NOTE Where solutions are referred to as (1+1), etc., this implies v/v unless otherwise stated.

### 5.1 Stock reagents

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

**5.1.2 Aluminum chloride solution**, Al (25 mg/ml).

Dissolve 123,5 g of aluminum chloride into water and dilute to 1 l with water.

**5.1.3 Ammonium chloride.**

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

**5.1.5 Ammonia solution** (1+1).

Add 1 volume of ammonia solution (concentrated) (5.1.4) to 1 volume of water.

**5.1.6 Ammonia solution** (1+9).

Add 1 volume of ammonia solution (concentrated) (5.1.4) to 9 volumes of water.

**5.1.7 Ammonium acetate solution**, 200 g/l.

Dissolve 200 g of ammonium acetate with water and dilute to 1 l.

**5.1.8 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.

**5.1.9 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 low temperature. Discard after 2 weeks.

**5.1.10 Boric acid solution, 40 g/l.**

Dissolve 40 g of boric acid with water and dilute to 1 l.

**5.1.11 Chloroform.**

**5.1.12 Cupferron solution, 60 g/l.**

Dissolve 6 g of cupferron in 100 ml of water; filter if necessary. Prepare this solution freshly, as required. Store the solid reagent in a tightly stoppered bottle in the presence of a piece of ammonium carbonate to prevent decomposition.

**5.1.13 Diantipyrylmethane (DAM) solution, 10 g/l.**

Dissolve 1 g of diantipyrylmethane monohydrate in 30 ml of hydrochloric acid (1+50), and dilute to 100 ml with water.

**5.1.14 Fusion mixture, an equimolecular mixture of sodium and potassium carbonates.**

**5.1.15 Hydrazinium dichloride solution, 150 g/l.**

Dissolve 15 g of hydrazinium dichloride in water and dilute to 100 ml.

**5.1.16 Hydrochloric acid (concentrated), (ISO 6353-2, R 13), 35 % by mass.**

**5.1.17 Hydrochloric acid (1+1),** <https://standards.iteh.ai/catalog/standards/sist/4be2bca5-a779-40cc-b403-91743c4014dc/iso-20565-1-2008>

Add 1 volume of hydrochloric acid (concentrated) (5.1.16) to 1 volume of water.

**5.1.18 Hydrochloric acid (1+50).**

Add 1 volume of hydrochloric acid (concentrated) (5.1.16) to 50 volumes of water.

**5.1.19 Hydrofluoric acid (concentrated), (ISO 6353-3, R 67), 40 % to 42 % by mass.**

**5.1.20 Hydrofluoric acid (1+9).**

Add 1 volume of hydrofluoric acid (concentrated) (5.1.19) to 9 volumes of water. Store in a plastic container.

**5.1.21 Hydroxyammonium chloride solution, 100 g/l.**

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

**5.1.22 Liquid cation-exchange resin, for example, Amberlite LA-2 resin (1+4), made by adding 50 ml of the resin to 200 ml of chloroform and mixing.**

Transfer the mixture to a 500 ml separating funnel and shake gently with 25 ml of H<sub>2</sub>SO<sub>4</sub> (1+9) for a few seconds, releasing the pressure frequently. Run off the organic layer into a 250 ml measuring cylinder.

**5.1.23 Nitric acid (concentrated), 70 % by mass.**

**5.1.24 Nitric acid (1+1).**

Add 1 volume of nitric acid (concentrated) (5.1.23) to one volume of water.



**5.1.25 2,2', 2''-Nitrilotriethanol (1+1).**

Add 1 volume of 2,2', 2''-nitrilotriethanol to one volume of water.

**5.1.26 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. However, discard the solution if it colours during storage.

**5.1.27 Phosphoric acid (concentrated), 85 % by mass.****5.1.28 Phosphoric acid, (2+3).**

Add 2 volumes of phosphoric acid (concentrated) (5.1.27) to 3 volumes of water, then mix and allow to cool.

**5.1.29 Phosphoric acid (1+9).**

Add 1 volume of phosphoric acid (concentrated) (5.1.27) to 9 volumes of water, then mix and allow to cool.

**5.1.30 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.

**5.1.31 Potassium periodate.****5.1.32 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 needs to be used.

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**5.1.33 Sodium hydroxide solution, 100 g/l.**

Cautiously dissolve 20 g of sodium hydroxide in 150 ml of water, while stirring and cooling. After cooling, dilute to approximately 200 ml.

**5.1.34 Sulfuric acid (concentrated), (ISO 6353-2, R 37), minimum 95 % by mass.****5.1.35 Sulfuric acid (1+1).**

Cautiously add, while stirring, 200 ml of sulfuric acid (concentrated) (5.1.34) to 200 ml of water, cooling the solution.

**5.1.36 Sulfuric acid (1+9).**

Cautiously add, while stirring, 20 ml of sulfuric acid (concentrated) (5.1.34) to 180 ml of water, cooling the solution.

**5.1.37 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 Bromophenol blue solution, 1 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.

**5.2.2 Calcein indicator (screened)**, (3,3'-bis[*N,N'*-bis(carboxymethyl) aminomethyl] fluorescein).

Mix, by grinding together, 0,2 g calcein, 0,12 g of thymolphthalein and 20,0 g of potassium chloride.

**5.2.3 Dithizone solution**, 0,25 g/l.

Dissolve 0,012 5 g of dithizone in 50 ml of ethanol (95 %). The solution will keep for a week.

**5.2.4 Xylenol orange solution**, 1 g/l.

Dissolve 0,10 g of the xylenol orange with water and dilute to 100 ml. This solution shall be kept in the dark and at low temperature. Discard after 4 weeks.

**5.3 Standard solutions**

**5.3.1 0,1 mol/l ammonium iron(II) sulfate solution.**

Measure 300 ml of water, add gradually 30 ml of sulfuric acid (concentrated) (5.1.34) while agitating and allow it to cool. Then add 40 g of ammonium iron(II) sulfate hexahydrate and 700 ml of water to dissolve it, and store the solution in an airtight container. Prepare this when it is needed.

Dry the required amount (about 0,3 g) of potassium dichromate, reference material for volumetric analysis, at 150 °C for about 60 min, and allow it to cool in a desiccator. Weigh 0,12 g to the nearest 0,1 mg in a 200 ml conical flask and add 100 ml of water to dissolve it. Then add gradually 30 ml of sulfuric acid (concentrated) (5.1.34) while agitating and allow it to cool. As an indicator, add several drops of ferroin solution, and titrate it with the 0,1 mol/l ammonium iron(II) sulfate solution. Make it the end-point when the solution turns from bluish green to reddish brown.

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Calculate the factor,  $F$ , of the 0,1 mol/l ammonium iron(II) sulfate solution using Equation (2).

$$F = \frac{m}{0,004\ 903 \times V} \times \frac{A}{100} \quad (2)$$

where

- $m$  is the mass of the weighed potassium dichromate, in grams (g);
- $A$  is the purity of the potassium dichromate, in percentage (%) by mass;
- 0,004 903 is the mass of potassium dichromate equivalent to 1 ml of 0,1 mol/l ammonium iron(II) sulfate solution, in grams (g);
- $V$  is the volume of the 0,1 mol/l ammonium iron(II) sulfate solution needed for titration, in millilitres (ml).

**5.3.2 Aluminium oxide standard solution**,  $\text{Al}_2\text{O}_3$  1 mg/ml.

Wash the surface of a sufficient amount (about 1 g) of aluminium metal (purity greater than 99,9 % by mass) with hydrochloric acid (1+4) to dissolve the oxidized layer. Then wash with water, ethanol, and diethyl ether in succession, and dry in a desiccator. Weigh 0,529 2 g of the aluminium and transfer into a 250 ml beaker. Cover with watch glass, add 20 ml hydrochloric acid (1+1), and heat to dissolve. After cooling, dilute to 1 000 ml in a volumetric flask with water.

**5.3.3 Calcium oxide standard solution**, CaO 1 mg/ml.

Dissolve 1,785 g of pure calcium carbonate, previously dried at 150 °C, in a slight excess of diluted hydrochloric acid (1+4) in a 250 ml beaker covered with a watch glass. Boil to expel carbon dioxide, cool and dilute to 1 000 ml in a volumetric flask.

**5.3.4 Diluted calcium oxide standard solution, CaO 0,05 mg/ml.**

Dilute precisely the calcium oxide standard solution (5.3.3) with water to a concentration of one twentieth.

**5.3.5 Chromium (III) oxide standard solution.**

Dry about 2 g to 3 g of potassium dichromate at 110 °C for at least 2 h. Weigh 1,935 g of this and dissolve in water, diluting to 1 000 ml in a volumetric flask.

**5.3.6 0,01 mol/l CyDTA solution.**

Add 8 ml of sodium hydroxide solution (100 g/l) and about 150 ml of water to 3,65 g of 1,2-cyclohexanediamine-*N,N,N',N'*-tetraacetic acid monohydrate (CyDTA) and dissolve by heating. After cooling, dilute to 1 000 ml with water.

**5.3.7 0,05 mol/l CyDTA solution.**

Add 40 ml of sodium hydroxide solution (100 g/l) and about 500 ml of water to 18,25 g of 1,2-cyclohexanediamine-*N,N,N',N'*-tetraacetic acid monohydrate (CyDTA) and dissolve by heating. After cooling, dilute to 1 000 ml with water.

Transfer 250 ml of magnesium oxide standard solution (MgO 1 mg/ml) (5.3.14) to a 500 ml conical flask. Add 100,0 ml of the CyDTA solution. Then add 2 g of ammonium chloride and 25 ml of ammonia solution, concentrated, 25 % by mass (5.1.4). Titrate with the CyDTA solution, using Solochrome Black 6B as the indicator, from red through purple to the last change to a clear ice blue.

Calculate the factor,  $F$ , of the 0,05 mol/l CyDTA standard solution using Equation (3).

$$F = \frac{f \times \frac{1 \times 250}{40,30}}{0,05 \times V} \quad (3)$$

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where

$f$  is the factor of the magnesium oxide standard solution (MgO 1 mg/ml) (see ISO 26845);

$V$  is the volume of the 0,05 mol/l CyDTA solution, in millilitres (ml).

**5.3.8 0,02 mol/l EDTA standard solution.**

Dissolve 7,5 g of EDTA2Na (ethylenediamine-tetraacetic acid disodium salt dihydrate) with 1 l of water. Store in a plastic bottle. Transfer 50 ml of 0,01 mol/l zinc solution, obtained in 5.3.31, to a 300 ml beaker and add 50 ml of water. Drop in sodium hydroxide solution (100 g/l) of up to pH 6 to pH 8 and add 2 ml of buffer solution (pH 10) (see 4.1.18 of ISO 26845:2008) and, while stirring, add 3 to 4 drops of Eriochrome Black T solution as an indicator and titrate with 0,02 mol/l EDTA solution. Observe the end point when the reddish purple colour of the solution is altered to blue.

Calculate the factor,  $F$ , of the 0,02 mol/l EDTA standard solution using Equation 4.

$$F = \frac{f \times 50}{2 \times V} \quad (4)$$

where

$f$  is the factor of the 0,01 mol/l zinc solution;

$V$  is the volume of the 0,02 mol/l EDTA solution, in millilitres (ml).