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

Part 3:

**Flame atomic absorption spectrometry
(FAAS) and inductively coupled plasma
atomic emission spectrometry (ICP-AES)**

*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) —*

*Partie 3: Méthodes par spectrométrie d'absorption atomique dans la
flamme (FAAS) et spectrométrie d'émission atomique avec plasma
induit par haute fréquence (ICP-AES)*



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Foreword

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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 20565-3 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 3: Flame atomic absorption spectrometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES)

1 Scope

This part of ISO 20565 specifies flame atomic absorption spectrometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods for the chemical analysis of chrome-bearing refractory products and chrome-bearing raw materials.

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

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

Table 1 — Range of determination (% by mass)

Component	Range
SiO ₂	0,5 to 10
Al ₂ O ₃	2 to 30
Fe ₂ O ₃	0,5 to 25
TiO ₂	0,01 to 1
MnO	0,01 to 1
CaO	0,01 to 3
MgO	15 to 85
Na ₂ O	0,01 to 1
K ₂ O	0,01 to 1
Cr ₂ O ₃	2 to 60
ZrO ₂	0,01 to 0,5
P ₂ O ₅	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 20565-1:2008, *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*

ISO 26845, *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 Instrumental methods using inductively coupled plasma emission spectrometry (ICP-AES)

3.1 Determination of residual silica in stock solutions (S1) by ICP-AES

3.1.1 Principle

The residual silica remaining in solution in solutions (S1) is determined using ICP-AES.

3.1.2 Reagents

Prepare the following reagents in addition to any reagents described in ISO 20565-1:2008, Clause 5, that are necessary.

3.1.2.1 Diluted silicon(IV) oxide standard solution, SiO₂ 0,08 mg/ml.

Transfer 20 ml of the silicon(IV) oxide standard solution (SiO₂ 1 mg/ml) into a 250 ml volumetric flask and dilute to the mark with water.

3.1.2.2 Matrix solution 2.

Carry out the procedure given in ISO 20565-1:2008, 9.2.2.3, without the sample, but omit heating the fusion mixture or anhydrous sodium carbonate. The equivalent solution to stock solution (S1) is referred to as matrix solution 2.

3.1.2.3 Series 2 solution for calibration.

Transfer appropriate aliquot portions of diluted silicon(IV) oxide standard solution (0,08 mg/ml) precisely into several 100 ml volumetric flasks in accordance with the composition of the samples. Add 10 ml each of matrix solution 2 (3.1.2.2) and dilute to the mark with water.

In Table 2, a typical example of the preparation of solutions is shown. In accordance with the compositions of the samples, and the type and capabilities of the instrument used, an appropriate series of solutions for calibration is prepared.

Table 2 — Example of series 2 solution for calibration

Solution No.	Matrix solution 2	Diluted silicon(IV) oxide standard solution	Concentration of solution
	ml	ml	SiO ₂ mg/100 ml
1	10	0	0
2	10	5	0,4
3	10	10	0,8
4	10	15	1,2
5	10	20	1,6
6	10	25	2,0

3.1.3 Procedure

Determine the silicon(IV) oxide remaining in solution (S1) (see ISO 20565-1) as follows.

Transfer 10 ml of stock solution (S1) to a 100 ml volumetric flask and dilute to the mark with water. This solution, for the determination of dissolved silicon(IV) oxide, is referred to as diluted stock solution (S1d).

Spray a portion of diluted stock solution (S1d) into the argon plasma flame of the ICP-AE spectrometer, and measure the emission intensity at, for example, the wavelength of 251,611 nm.

3.1.4 Blank test

Carry out the procedure in 3.1.3 with blank solution (B1) (see ISO 20565-1). The equivalent diluted blank solution to diluted stock solution (S1d) is referred to as diluted blank solution (B1d).

3.1.5 Plotting of calibration graph

Calibrate the ICP-AE spectrometer using the series 2 solution (3.1.3.3) and the emission procedure described in 3.1.3. Plot the relation between the emission intensity and mass of oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

A new calibration should be carried out using the range of calibration and blank solution solutions for each set of determinations.

3.1.6 Calculation

Calculate the mass fraction of silicon(IV) oxide, w_{SiO_2} , as a percentage, using Equation (1). Use the mass of silicon(IV) oxide derived from the figures obtained from 3.1.3 and 3.1.4 and the calibration prepared in 3.1.5.

$$w_{\text{SiO}_2} = \frac{(m_1 - m_2) + (m_s - m_b) \times \frac{250}{10}}{m} \times 100 \quad (1)$$

where

m is the mass of the test portion (see ISO 20565-1), in grams (g);

m_1 is the first mass from ISO 20565-1:2008, 9.2.2.3.3, in grams (g);

m_2 is the second mass from ISO 20565-1:2008, 9.2.2.3.3, in grams (g);

m_s is the mass of silicon(IV) oxide in diluted stock solution (S1d) as described in 3.1.3, in grams (g);

m_b is the mass of silicon(IV) oxide in diluted blank solution (B1d) as described in 3.1.4, in grams (g).

3.2 Determination of silicon(IV) oxide, aluminium oxide, iron(III) oxide, titanium(IV) oxide, manganese(II) oxide, calcium oxide, magnesium oxide, chromium(III) oxide and zirconium oxide using stock solutions (S1) or (S'1) by ICP-AES

3.2.1 Principle

The emission intensities of silicon(IV) oxide, aluminium oxide, iron(III) oxide, titanium(IV) oxide, manganese(II) oxide, calcium oxide, magnesium oxide, chromium(III) oxide and zirconium oxide are measured by an ICP-AE spectrometer for stock solutions (S1) (see ISO 20565-1) or (S'1) (see ISO 20565-1). This method should be applied to components in (S1) or (S'1) in accordance with Table 3.

Table 3 — Application range (% by mass)

Component	Range
SiO ₂	0,1 to 10
Al ₂ O ₃	0,05 to 10
Fe ₂ O ₃	0,01 to 10
TiO ₂	0,01 to 1
MnO	0,01 to 1
CaO	0,01 to 10
Cr ₂ O ₃	0,01 to 10
ZrO ₂	0,01 to 0,5

NOTE 1 When solution (S1) is used, the SiO₂ is residual silica. When solution (S'1) is used, the SiO₂ is all of the silicon(IV) oxide.

NOTE 2 Determination of calcium oxide by this method cannot be applied to calcium oxide contents of more than 10 % by mass.

3.2.2 Reagents

Prepare the following reagents in addition to any reagents described in ISO 20565-1:2008, Clause 5, that are necessary.

3.2.2.1 Mixed standard solution 3, SiO₂ 0,04 mg/ml, Al₂O₃ 0,04 mg/ml, Fe₂O₃ 0,04 mg/ml, TiO₂ 0,005 mg/ml, MnO 0,005 mg/ml, CaO 0,04 mg/ml, Cr₂O₃ 0,04 mg/ml, ZrO₂ 0,005 mg/ml.

Transfer aliquot portions of standard silicon(IV) oxide (40 ml), aluminium oxide (40 ml), iron(III) oxide (40 ml), titanium(IV) oxide (5 ml), manganese(II) oxide (5 ml), calcium oxide (40 ml), chromium(III) oxide (40 ml) and zirconium oxide (5 ml) solutions to a 1 000 ml volumetric flask and dilute to the mark with water.

3.2.2.2 Matrix solution 3 or 3'.

Carry out the procedure given in ISO 20565-1:2008, 9.2.2.3 or 9.2.3.3, without the sample, but omit heating the fusion mixture or anhydrous sodium carbonate. The equivalent solution to stock solution (S1) or (S'1) is referred to as matrix solution 3 or 3' as applicable.

3.2.2.3 Internal standard solution.

Transfer 10 ml of standard scandium oxide solution (1 mg/ml) and standard yttrium oxide solution (1 mg/ml) to a 100 ml volumetric flask and dilute to the mark with water. Prepare when necessary.

3.2.2.4 Series 3 solutions for calibration.

Transfer appropriate aliquot portions of mixed standard solution 3 to each of several 100 ml volumetric flasks. Add 20 ml of matrix solution 3 or matrix solution 3' and 5 ml of internal standard solution, respectively, and dilute to the mark with water.

In Table 4, an example of the preparation of solutions is shown. Prepare an appropriate solution series for calibration in accordance with the composition of the sample, the type and capabilities of instrument used. When using this approach to calibration, it is essential to check for line interferences of any of these oxides on each other. If any are present, appropriate corrections should be applied.

Table 4 — Example of series 3 solution for calibration

Solution for calibration No.	Matrix solution 3 ml	Internal standard solution ml	Mixed standard solution 3 ml	Concentration of solution mg/100 ml							
				SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	MnO	CaO	Cr ₂ O ₃	ZrO ₂
1	10	5	0	0,00	0,00	0,00	0,000	0,000	0,00	0,00	0,000
2	10	5	2	0,08	0,08	0,08	0,010	0,010	0,08	0,08	0,010
3	10	5	5	0,20	0,20	0,20	0,025	0,025	0,20	0,20	0,025
4	10	5	10	0,40	0,40	0,40	0,050	0,050	0,40	0,40	0,050
5	10	5	20	0,80	0,80	0,80	0,100	0,100	0,80	0,80	0,100
6	10	5	30	1,20	1,20	1,20	0,150	0,150	1,20	1,20	0,150
7	10	5	40	1,60	1,60	1,60	0,200	0,200	1,60	1,60	0,200
8	10	5	50	2,00	2,00	2,00	0,250	0,250	2,00	2,00	0,250

3.2.3 Procedure

Transfer a 10 ml aliquot portion of stock solution (S1) or (S'1) as prepared in ISO 20565-1:2008, 9.2.2.3.4 or 9.2.3.3, to a 100 ml volumetric flask. Add 5 ml of internal standard solution, and dilute to the mark with water. This solution is designated as stock solution (S1dScY) or (S1'dScY).

Spray a portion of stock solution (S1dScY) or (S1'dScY) into the argon plasma flame of an ICP-AE spectrometer, and measure the emission intensity of each element and internal standard elements at the appropriate wavelength given in Table 5.

Table 5 — Example of wavelength

Component	Element	Wavelength nm
SiO ₂	Si	251,611
Al ₂ O ₃	Al	396,152
Fe ₂ O ₃	Fe	259,940
TiO ₂	Ti	334,941
MnO	Mn	257,610
CaO	Ca	393,366
Cr ₂ O ₃	Cr	267,716
ZrO ₂	Zr	343,823
Internal standard element	Y	371,030
	Sc	361,383

3.2.4 Blank test

Using a 10 ml aliquot portion of blank solution (B1) (see ISO 20565-1) or (B'1) (see ISO 20565-1), carry out the procedure given in 3.2.3. The solution corresponding to stock solution (S1) or (S'1) is designated as blank solution (B1dScY) or (B'1dScY).

3.2.5 Plotting the calibration graph

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Use series 3 solutions for calibration. Carry out the procedure described in 3.2.3 and plot the relationship between the emission intensity and mass of each component (SiO₂, Al₂O₃, Fe₂O₃, TiO₂, MnO, CaO, Cr₂O₃ and ZrO₂). Prepare a calibration graph for each component.

3.2.6 Calculation

Calculate the mass fraction $w_{M_nO_m}$ of each component w_{SiO_2} , $w_{Al_2O_3}$, $w_{Fe_2O_3}$, w_{TiO_2} , w_{MnO} , w_{CaO} , $w_{Cr_2O_3}$ and w_{ZrO_2} , expressed as a percentage, using Equation (2). Use the mass of each component derived from the emission intensity in 3.2.3 and 3.2.4 and the calibration prepared in 3.2.5.

$$w_{M_nO_m} = \frac{m_s - m_b}{m} \times \frac{100}{10} \times 100 \tag{2}$$

where

m_s is the mass of each component, indicated as M_nO_m , in stock solution (S1dScY) or (S'1dScY), in grams (g);

m_b is the mass of each component, indicated as M_nO_m , in blank solution (B1dScY) or (B'1dScY), in grams (g);

m is the mass of the test portion (see ISO 20565-1), in grams (g).

3.3 Determination of sodium oxide and potassium oxide using stock solutions (S4) by ICP-AES

3.3.1 Principle

The emission intensity of sodium and potassium in stock solution (S4) (see ISO 29565-1) is measured by ICP-AE spectrometer.

3.3.2 Reagents

Prepare the following reagents in addition to any reagents described in ISO 20565-1:2008, Clause 5, that are necessary.

3.3.2.1 Matrix solution 4.

Transfer amounts of calcium oxide and magnesium oxide solutions, corresponding to the contents of calcium oxide and magnesium oxide in the sample, and dilute to 250 ml with water.

NOTE The added volumes of the matrix solution are approximate values ± 5 ml. For example, when the mass fractions of calcium oxide and magnesium oxide are 24 % and 71 %, respectively, the added volumes of calcium oxide and magnesium oxide solutions are 25 ml and 75 ml, respectively.

3.3.2.2 Mixed standard solution 4, Na₂O 0,05 mg/ml, K₂O 0,05 mg/ml.

Transfer 25 ml of the standard sodium oxide and potassium oxide solutions to a 500 ml volumetric flask and dilute to the mark with water.

3.3.2.3 Series 4 solution for calibration.

Transfer aliquot portions of mixed standard solution 4 to each of several 100 ml volumetric flasks. To each, add 20 ml of matrix solution 4 and 5 ml of hydrochloric acid (1+1) (see ISO 20565-1:2008, 5.1.17) and dilute to the mark with water. Typical examples of preparation are shown in Table 6.

Table 6 — Example of series 4 solution for calibration

Solution No.	Matrix solution 4 ml	Hydrochloric acid (1+1) ml	Mixed standard solution 4 ml	Concentration of solution mg/100 ml	
				Na ₂ O	K ₂ O
1	20	5	0	0	0
2	20	5	2	0,10	0,10
3	20	5	5	0,25	0,25
4	20	5	10	0,50	0,50
5	20	5	20	1,00	1,00
6	20	5	30	1,50	1,50
7	20	5	40	2,00	2,00

3.3.3 Procedure

Spray a portion of stock solution (S4) (see ISO 20565-1) into the argon plasma flame of an ICP-AE spectrometer, and measure the emission intensity of each element at the appropriate wavelength given in Table 7.