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ISO 10058-2

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Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) —

Part 2:
Wet chemical analysis

Analyse chimique des produits de magnésie et de dolomie (méthode alternative à la méthode par fluorescence de rayons X) —

Partie 2: Méthodes d'analyse chimique par voie humide

Reference number
ISO 10058-2:2008(E)

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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.

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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 10058-2 was prepared by Technical Committee ISO/TC 33, *Refractories*.

This first edition of ISO 10058-2, together with ISO 10058-1 and ISO 10058-3, cancels and replaces ISO 10058:1992 which has been technically revised to include the increasing use of flame atomic absorption spectrophotometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods as well some improvements in the wet chemical analyses procedures developed in Japan.

ISO 10058 consists of the following parts, under the general title *Chemical analysis of magnesite and dolomite refractory products (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 spectrophotometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES)*

Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) —

Part 2:
Wet chemical analysis

1 Scope

This part of ISO 10058 specifies traditional (“wet process”) methods for the chemical analysis of magnesite and dolomite refractory products and raw materials.

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

Table 1 — Range of determination (percentage by mass)

Component	Range	Component	Range
SiO_2	0,1 to 10	MgO	30 to 99,9
Al_2O_3	0,05 to 10	Na_2O	0,01 to 1
Fe_2O_3	0,01 to 10	K_2O	0,01 to 1
TiO_2	0,01 to 1	Cr_2O_3	0,01 to 3
MnO	0,01 to 1	ZrO_2	0,01 to 1
CaO	0,01 to 60	P_2O_5	0,01 to 5
LOI	0,01 to 60	—	—

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 10058-1:2008, *Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) — Part 1: Apparatus, reagents, dissolution and determination of gravimetric silica*

ISO 10058-3:2008, *Chemical analysis of magnesite and dolomite refractory products (alternative to the X-ray fluorescence method) — Part 3: Flame atomic absorption spectrophotometry (FAAS) and inductively coupled atomic plasma emission spectrometry (ICP-AES)*

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 Determination of silicon(IV) oxide

3.1 General

The determination of silicon(IV) oxide is carried out using one of the following methods.

a) Combined use of the dehydration or the coagulation and molybdenum blue methods

This method is applied to samples consisting of more than 4 % by mass of silicon(IV) oxide (see 3.2).

b) Molybdenum blue method

This method is applied to samples consisting of less than 10 % by mass of silicon(IV) oxide (see 3.3).

3.2 Combined use of the coagulation and molybdenum blue methods

3.2.1 Principle

An aliquot portion of the stock solution (S1) (see ISO 10058-1:2008, Annex A), after pH adjustment, is treated with ammonium molybdate and the silicomolybdate is reduced to yield molybdenum blue, the absorbance of which is measured.

The sum of this residual silicon(IV) oxide in solution plus the mass of silicon (IV) oxide $(m_1 - m_2)$ calculated in accordance with ISO 10058-1:2008, 8.2.2.3.3, gives the total silicon(IV) oxide content.

3.2.2 Procedure

This determination should be commenced with little delay after the stock solution (S1) is prepared, as prolonged standing may allow polymerization of silica to occur leading to low results.

Transfer 10 ml of stock solution (S1) (see ISO 10058-1:2008, Annex A) to a 100 ml plastic beaker, add 2 ml of hydrofluoric acid (1+9) and mix with a plastic rod. Allow to stand for 10 min and add 50 ml of boric acid solution. Add 2 ml of ammonium molybdate solution while mixing at a temperature of 25 °C and allow to stand for 10 min. Add 5 ml of L (+)-tartaric acid solution while stirring and, after 1 min, add 2 ml of L (+)-ascorbic acid solution. Transfer the solution to a 100 ml volumetric flask, dilute to the mark with water, mix and allow to stand for 60 min.

Measure the absorbance of the solution in a 10 mm cell at a wavelength of 650 nm using water as reference.

3.2.3 Plotting the calibration graph

Transfer 0 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml aliquot portions [0 mg to 0,4 mg as silicon(IV) oxide] of dilute standard silicon(IV) oxide solution (SiO_2 0,04 mg/ml) to separate 100 ml plastic beakers and add to each 10 ml of blank solution (B1) (see ISO 10058-1:2008, Annex A). Treat these solutions and measure the absorbance as given in 3.2.2, and plot the absorbances against the amounts of silicon(IV) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

3.2.4 Blank test

Using the blank solution (B1) (see ISO 10058-1:2008, Annex A), carry out the procedure given in 3.2.2.

3.2.5 Calculation

Calculate the mass fraction of silicon(IV) oxide, $w_{\mathrm{SiO_2}}$, expressed as a percentage, using Equation (1), with the absorbances obtained by the procedures given in 3.2.2 and 3.2.4 and the calibration graph plotted in 3.2.3.

$$w_{\mathrm{SiO_2}} = \frac{(m_1 - m_2) + (m_{\mathrm{s}} - m_{\mathrm{b}}) \times \frac{250}{10}}{m} \times 100 \qquad (1)$$

where

m_1 is the mass from ISO 10058-1:2008, 8.2.2.3.3, in grams (g);

m_2 is the mass from ISO 10058-12008, 8.2.2.3.3, in grams (g);

m_{s} is the mass of silicon(IV) oxide in the aliquot portion of stock solution (S1) as applicable, in grams (g);

m_{b} is the mass of silicon(IV) oxide in the aliquot portion of blank solution (B1) as applicable, in grams (g);

m is the mass of the test portion calculated in accordance with ISO 10058-1:2008, 8.2.2.3.1, in grams (g).

3.3 Molybdenum blue method

3.3.1 Principle

An aliquot portion of the stock solution (S′1) (see ISO 10058-1:2008, Annex A), after pH adjustment, is treated with ammonium molybdate and the silicomolybdate is reduced to yield molybdenum blue, the absorbance of which is measured.

3.3.2 Procedure

Transfer precisely an aliquot portion of stock solution (S′1) (see ISO 10058-1:2008, Annex A) to two 100 ml plastic beakers and add to each an aliquot portion of blank solution (B′1) (see ISO 10058-1:2008, Annex A). Add to each 2 ml of hydrofluoric acid (1+9), mix with a plastics rod and allow to stand for 10 min. Add 50 ml of boric acid solution, dilute to 80 ml with water. Add 5 ml of ammonium molybdate solution while mixing at a temperature of 25 °C and allow to stand for 10 min. Add 5 ml of L (+)-tartaric acid solution while stirring and, after 1 min, add to 10 ml of L (+)-ascorbic acid solution. Transfer each solution to a 200 ml volumetric flask, dilute to the mark with water and mix. Allow to stand for 60 min and measure the absorbance of the solutions in a 10 mm cell at a wavelength of 650 nm against water as a reference. Average the two measurements.

NOTE Aliquot volumes of stock solution (S′1) and blank solution (B′1) corresponding to the mass fraction of silicon(IV) oxide in the sample are shown in Table 2.

If the difference of the two absorbance measurements is greater than 0,005, repeat the procedure given in 3.3.2 and ISO 10058-1:2008, 8.2.3.3. When measurements of the same sample with around 1,0 absorbance are repeated, it is necessary for the spectrophotometer to show the differences within 0,002.

Table 2 — Aliquot volumes of stock solution (S′1) and blank solution (B′1)

Mass fraction of silicon(IV) oxide %	Aliquot volume of stock solution (S′1) ml	Aliquot volume of blank solution (B′1) ml
< 2	20	0
⩾ 2, < 4	10	10
⩾ 4, ⩽ 10	5	15

3.3.3 Blank test

Carry out a blank determination by treating the blank solution (B1) (see ISO 10058-1:2008, Annex A) and following the procedure described into 3.3.2. The volume of the aliquot portion of blank solution is the same as those for the corresponding “S” solution.

3.3.4 Plotting of calibration graph

Transfer 0 ml, 5 ml, 10 ml, 15 ml, 20 ml and 25 ml aliquot portions [0 mg to 1 mg as silicon(IV) oxide] of dilute standard silicon(IV) oxide solution (SiO_2 0,04 mg/ml) to separate 100 ml plastic beakers and add to each 20 ml of blank solution (B′1) (see ISO 10058-1:2008, Annex A). Treat these solutions and measure the absorbance in accordance with the procedure from the addition of hydrofluoric acid (1+9) in 3.3.2. Plot the absorbance against the amounts of silicon(IV) oxide and prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

3.3.5 Calculation

Calculate the mass fraction of silicon(IV) oxide, $w_{\mathrm{SiO_2}}$, expressed as a percentage, using Equation (2), with the amount of silicon(IV) oxide derived from the absorbance measurements obtained in 3.3.2 and 3.3.3 and the calibration in 3.3.4.

$$w_{\mathrm{SiO_2}} = \frac{m_{\mathrm{s}} - m_{\mathrm{b}}}{m} \times \frac{250}{V} \times 100 \tag{2}$$

where

m_{s} is the mass of silicon(IV) oxide in the aliquot portion of stock solution (S′1), in grams (g);

m_{b} is the mass of silicon(IV) oxide in the aliquot portion of blank solution (B′1), in grams (g);

V is the aliquot portion volume of stock solution (S′1), in millilitres (ml);

m is the mass of the test portion in ISO 10058-1:2008, 8.2.3.3, in grams (g).

4 Determination of aluminium oxide

4.1 Principle

An aliquot portion of stock solution (S1) or (S′1) (see ISO 10058-1:2008, Annex A) is transferred. Excess standard volumetric CyDTA solution is added to an aliquot portion of stock solution. A chelate compound of aluminium CyDTA is formed by adjusting the pH with ammonia water. The pH is further adjusted by addition of hexamethylenetetramine. The amount of remaining standard volumetric CyDTA is determined by back-titration with standard volumetric zinc solution using xylenol orange as an indicator. The content of aluminium oxide is calculated by allowing for the content of iron(III) oxide determined by the method given in Clause 5.

4.2 Procedure

4.2.1 Transfer precisely 50 ml of stock solution (S1), or stock solution (S′1), to a 300 ml beaker. Precisely add an adequate amount of standard volumetric CyDTA solution [c(CyDTA) = 0,01 mol/l] and dilute to 100 ml with water.

The volume of standard volumetric CyDTA solution to be added depends on the percentage of aluminium oxide and iron(III) oxide as shown in Table 3.

Table 3 — Volumes of standard volumetric CyDTA solution

Mass fraction of aluminium oxide and iron(III) oxide %	Volume of standard volumetric CyDTA solution ml
< 1	5
⩾ 1, < 2	10
⩾ 2, < 4	20
⩾ 4, < 7	30
⩾ 7	40

4.2.2 Add 1 g of hexamethylenetetramine and a drop of methyl orange solution as an indicator. Using drops, add ammonia water (1+1) and ammonia water (1+9) of up to pH 3 until the solution indicates a slightly orange colour. Allow to stand for 5 min.

If ammonia water (1+9) is added excessively, the pH can be adjusted to less than 3, showing a red colour, by adding hydrochloric acid (1+1), before repeating the procedure in above paragraph.

4.2.3 Add 5 g of hexamethylenetetramine of up to pH 5,5 to pH 5,8, add 4 or 5 drops of xylenol orange solution as an indicator and titrate with standard volumetric zinc solution [$c(\text{Zn}) = 0{,}01$ mol/l]. Carry out the titration gently while mixing. Record the end point of the titration when the colour changes from yellow to the first appearance of a permanent reddish colour.

4.3 Blank test

Using a 50 ml aliquot portion of blank solution (B1), or blank solution (B′1) (see ISO 10058-1:2008, Annex A), carry out the procedure in accordance with 4.2. Ensure that CyDTA standard volumetric solution is the same as that used for the corresponding stock solution.

4.4 Calculation

Calculate the mass fraction of aluminium oxide, $w_{\text{Al}_2\text{O}_3}$, expressed as a percentage, using Equation (3).

$$\begin{aligned} w_{\text{Al}_2\text{O}_3} &= \frac{(V_2 - V_1)\times F \times 0{,}001\,019\,6}{m}\times\frac{250}{50}\times 100 \\ &-[\left(w_{\text{Fe}_2\text{O}_3} + w_{\text{TiO}_2}\right)\times 0{,}638 + w_{\text{MnO}}\times 0{,}719 + w_{\text{ZrO}_2}\times 0{,}414] \end{aligned} \quad (3)$$

where

V_1 is the volume of standard volumetric zinc solution in 4.2.3, in millilitres (ml);

V_2 is the volume of standard volumetric zinc solution in 4.3, in millilitres (ml);

F is the factor of standard volumetric zinc solution;

m is the mass of the weighed sample in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.3.3, in grams (g);

$w_{\text{Fe}_2\text{O}_3}$ is the mass fraction of iron(III) oxide determined in 5.5 or in ISO 10058-3:2008, 3.2.6, expressed as a percentage;

w_{TiO_2} is the mass fraction of titanium(IV) oxide determined in 6.5 or in ISO 10058-3:2008, 3.2.6, expressed as a percentage;

w_{MnO} is the mass fraction of manganese oxide determined in 7.5 or in ISO 10058-3:2008, 3.2.6 or 4.1.6, expressed as a percentage;

w_{ZrO_2} is the mass fraction of zirconium oxide determined in 13.5 or in ISO 10058-3:2008, 3.2.6, expressed as a percentage.

5 Determination of iron(III) oxide

5.1 Principle

Stock solution (S1) or (S′1) (see ISO 10058-1:2008, Annex A) is transferred and iron is reduced with L (+)-ascorbic acid. 1,10-Phenanthrolinium chloride is added, the pH is adjusted by adding ammonium acetate and the iron coloured complex with phenanthrolinium is developed. The absorbance is measured.

5.2 Procedure

5.2.1 Transfer an aliquot portion, determined in accordance with Table 4, of either stock solution (S1) or (S′1), to a 100 ml volumetric flask.

NOTE The aliquot portion volumes of stock solutions (S1) or (S′1) shown in Table 4 correspond to the content of iron(III) oxide in the sample.

Table 4 — Aliquot volumes of stock solution (S1) or (S′1)

Mass fraction of iron(III) oxide %	Aliquot volume of stock solution (S1) or (S′1) ml
< 0,5	25
⩾ 0,5, < 1,5	10
⩾ 1,5, < 5	5
⩾ 5	2 [a]

[a] Alternatively, transfer precisely 20 ml of the stock solution to a 100 ml volumetric flask and dilute to the mark with water. An aliquot portion of 10 ml may be used instead of the 2 ml of the stock solution.

5.2.2 Dilute to about 60 ml with water and, while mixing, add 5 ml of L (+)-tartaric acid solution and 2 ml of L (+)-ascorbic acid solution. Add 10 ml of 1,10-phenanthrolinium chloride solution and 10 ml of ammonium acetate solution. Dilute to the mark with water and allow to stand for 30 min. Measure the absorbance of the solution in a 10 mm cell at a wavelength of 510 nm against water.

5.3 Blank test

Using an aliquot portion of blank solution (B1) or blank solution (B′1) (see ISO 10058-1:2008, Annex A), carry out the procedure in accordance with 5.2. Use the same volume of aliquot portion for the blank solution as used for the corresponding stock solution.

5.4 Plotting of calibration graph

Transfer 0 ml (as reference), 5 ml, 10 ml and 15 ml aliquot portions [0 mg to 0,6 mg as iron(III) oxide] of the dilute standard iron(III) oxide solution (Fe_2O_3 0,04 mg/ml) to separate 100 ml volumetric flasks. Treat these solutions in accordance with 5.2.2 and measure the absorbance against the reference solution. Plot the relation between the absorbance measurements and mass of iron(III) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

5.5 Calculation

Calculate the mass fraction of iron(III), $w_{\mathrm{Fe_2O_3}}$, expressed as a percentage, using Equation (4). Use the amount of iron(III) oxide derived from the absorbance in 5.2 and 5.3, and the calibration in 5.4.

$$w_{\mathrm{Fe_2O_3}} = \frac{m_{\mathrm{s}} - m_{\mathrm{b}}}{m} \times \frac{250}{V} \times 100 \qquad (4)$$

where

m_{s} is the mass of iron(III) oxide in the aliquot portion of stock solution (S1) or (S′1), in grams (g);

m_{b} is the mass of iron(III) oxide in the aliquot portion of blank solution (B1) or (B′1), in grams (g);

m is the mass of the test portion in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.3.3, in grams (g);

V is the volume of the aliquot portion taken from stock solution (S1) or (S′1) in 5.2.1, in millilitres (ml).

6 Determination of titanium(IV) oxide

6.1 Principle

Sample solution (S1) or (S′1) (see ISO 10058-1:2008, Annex A) is transferred. After the adjustment of acidity, iron is reduced with the addition of L (+)-ascorbic acid. The titanium is coloured by the di-antipyrylmethane (DAM) and the absorbance is measured.

6.2 Procedure

6.2.1 Transfer an aliquot portion, determined using Table 5, of either stock solution (S1) or (S′1) (see ISO 10058-1:2008, Annex A), to a 50 ml volumetric flask.

NOTE The aliquot portions taken from stock solution (S1) or (S′1) shown in Table 5 correspond to the content of titanium(IV) oxide in the sample.

Table 5 — Aliquot volumes taken from stock solution (S1) or (S′1)

Mass fraction of titanium(IV) oxide %	Aliquot volume taken of (S1) or (S′1) ml
< 0,5	25
⩾ 0,5, < 1,5	10
⩾ 1,5	5

6.2.2 Add 5 ml of hydrochloric acid (1+1), 2 ml of L (+)-ascorbic acid and allow to stand for 1 min. Add 15 ml of DAM solution, dilute to the mark with water and allow to stand for 90 min. Measure the absorbance of the solution in a 10 mm cell at a wavelength of 390 nm against water.

6.3 Blank test

Using the same aliquot portion of stock solution used in blank test solution (B1) or (B′1) (see ISO 10058-1:2008, Annex A), carry out the procedure in 6.2.

6.4 Plotting of calibration graph

Transfer 0 ml (as reference), 5 ml, 10 ml, 15 ml and 20 ml aliquot portions [0 mg to 0,20 mg as titanium(IV) oxide] of the dilute standard titanium(IV) oxide solution (TiO_2 0,01 mg/ml) to separate 50 ml volumetric flasks and treat these solutions in accordance with 6.2. Measure the absorbance against the reference solution. Plot the relation between the absorbance and the amount of titanium(IV) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

6.5 Calculation

Calculate the mass fraction of titanium(IV) oxide, $w_{\mathrm{TiO_2}}$, expressed as a percentage, using Equation (5), with the amount of titanium(IV) oxide derived from the absorbance in 6.2.2 and 6.3, and the calibration in 6.4.

$$w_{\mathrm{TiO_2}} = \frac{m_\mathrm{s} - m_\mathrm{b}}{m} \times \frac{250}{V} \times 100 \qquad (5)$$

where

m_s is the mass of titanium(IV) oxide in the aliquot portion of stock solution (S1) or (S′1), in grams (g);

m_b is the mass of titanium(IV) oxide in the aliquot portion of the blank test solution (B1) or (B′1), in grams (g);

V is the volume of the aliquot portion taken for stock solution (S1) or (S′1) in 6.2.1, in millilitres (ml);

m is the mass of the test portion specified in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.3.3, in grams (g).

7 Determination of manganese(II) oxide

7.1 Principle

Stock solution (S1) or (S′1) is transferred. To remove chloride ions, fume with sulfuric acid. The manganese(II) is coloured by oxidation of manganese with potassium periodate and the absorbance is measured.

7.2 Procedure

7.2.1 Transfer 20 ml of stock solution (S1) or (S′1) (see ISO 10058-1:2008, Annex A) to a 200 ml beaker.

7.2.2 Add 10 ml of sulfuric acid (1+1) and evaporate until the sulfuric acid begins to fume intensively, to remove chloride ions and to decompose the polyethylene oxide. After cooling, add 20 ml of concentrated nitric acid, 10 ml of phosphoric acid (1+9) and approximately 50 ml of water. Boil to dissolve any salts present and to remove nitrous gases, allow to cool slightly and add 0,2 g of potassium periodate. Boil again for 2 min and place the beaker on a steam bath for 40 min. Allow to cool, transfer the solution quantitatively to a 100 ml volumetric flask and dilute to the mark with water and stir vigorously. Measure the absorbance of the solution in a 10 mm cell at a wavelength of 525 nm (or 545 nm) against water.

7.3 Blank test

Using the same aliquot portion of stock solution as that of the blank solution (B1) or (B′1) (see ISO 10058-1:2008, Annex A), carry out the procedure described in 7.2.

7.4 Plotting of calibration graph

Transfer 0 ml (as reference), 5 ml, 10 ml, 15 ml, 20 ml and 25 ml aliquot portions [0 mg to 1,00 mg as manganese(II) oxide] of the dilute standard manganese(II) oxide solution (MnO 0,04 mg/ml) to six 200 ml beakers and treat these solutions in accordance with 7.2.2. Measure the absorbance against the reference solution. Plot the relation between the absorbance and the amount of manganese(II) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

7.5 Calculation

Calculate the mass fraction of manganese(II) oxide, w_{MnO}, expressed as a percentage, using Equation (6), with the amount of manganese(II) oxide derived from the absorbance in 7.2.2 and 7.3, and the calibration in 7.4.

$$w_{\mathrm{MnO}} = \frac{m_{\mathrm{s}} - m_{\mathrm{b}}}{m} \times \frac{250}{20} \times 100 \qquad (6)$$

where

m_{s} is the mass of manganese(II) oxide in the aliquot portion of stock solution (S1) or (S′1), in grams (g);

m_{b} is the mass of manganese(II) oxide in the aliquot portion of blank solution (B1) or (B′1), in grams (g);

m is the mass of the test portion in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.2.4, in grams (g).

8 Determination of calcium oxide

8.1 Principle

Stock solution (S1) or (S′1) (see ISO 10058-1:2008, Annex A) is transferred and 2,2′,2″-nitrilotriethanol and sodium sulfide are added in order to mask interference ions. After adjusting pH to about 13, the preparatory standard volumetric EDTA titration is carried out using calcein as an indicator. Stock solution (S1) or (S′1) is transferred and interference ions are masked. The standard volumetric EDTA solution, which is 1 ml to 2 ml less than that obtained during preparatory titration, is added and the solution is diluted with water. After adjusting pH to about 13 with potassium hydroxide, the standard volumetric EDTA titration is carried out using calcein (see ISO 26845) as an indicator.

8.2 Procedure

8.2.1 Transfer precisely 20 ml of stock solution (S1) or (S′1) to a 500 ml beaker and dilute to 300 ml with water. Add 5 ml of 2,2′,2″-nitrilotriethanol (1+2) and 1 ml of sodium sulfide solution and adjust the pH to 12,7 to 13,2 by adding potassium hydroxide solution. Stir for 2 min to 3 min. Add 0,05 g of calcein indicator and titrate with standard volumetric EDTA [c(EDTA) = 0,02 mol/l] solution until the fluorescent green colour of the solution has changed to orange.

8.2.2 Transfer precisely 20 ml of stock solution (S1) or (S′1) to a 500 ml beaker and dilute to 300 ml with water. Add 5 ml of 2,2′,2″-nitrilotriethanol (1+2), 1 ml of sodium sulfide solution and a volume of standard volumetric EDTA solution 1 ml to 2 ml less than that obtained in the preparatory titration performed (8.2.1). Adjust the pH to 12,7 to 13,2 by adding potassium hydroxide solution. Stir for 2 min to 3 min. After adding 0,05 g of calcein indicator, stir and titrate gradually with standard volumetric EDTA solution until the fluorescent green colour of the solution has changed to orange.

NOTE If the titration is carried out on black paper or chalkboard, it will make the end point clearer.

8.3 Blank test

Using the same aliquot volume of stock solution as that of the blank solution (B1) or (B′1) (see ISO 10058-1:2008, Annex A), carry out the procedure described in 8.2.

8.4 Calculation

Calculate the mass fraction of calcium oxide, w_{CaO}, expressed as a percentage, using Equation (7).

$$w_{\mathrm{CaO}} = \frac{(V_1 - V_2) \times F \times 0{,}001\,121\,6}{m} \times \frac{250}{20} \times 100 \qquad (7)$$

where

V_1 is the volume of standard volumetric EDTA solution on titration in 8.2.2, in millilitres (ml);

V_2 is the volume of standard volumetric EDTA solution on titration in 8.3, in millilitres (ml);

F is the factor of standard volumetric EDTA solution;

m is the mass of the test portion in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.2.4, in grams (g).

9 Determination of magnesium oxide

9.1 Principle

Stock solution (S1) or (S′1) (see ISO 10058-1:2008, Annex A) is transferred and hydroxyammonium chloride, 2,2′,2″-nitrilotriethanol and sodium sulfide are added to an aliquot portion of stock solution in order to mask interfering ions. Buffer is added to adjust the pH to about 10. The sum of the content of calcium oxide and magnesium oxide is determined by titration using standard volumetric EDTA solution. The content of magnesium oxide is calculated by using the sum and the content of calcium oxide obtained in Clause 8, or in ISO 10058-3:2008, Clause 3 or Clause 4.

9.2 Procedure

Transfer precisely 20 ml of stock solution (S1) or (S′1) to a 500 ml beaker and dilute to 200 ml with water. Add 5 ml of hydroxyammonium chloride solution, 20 ml of 2,2′,2″-nitrilotriethanol (1+1), 10 ml of buffer (pH 10) and 1 ml of sodium sulfide solution. Add 3 to 4 drops of Eriochrome Black T solution as an indicator while stirring and titrate with standard volumetric EDTA solution [$c(\mathrm{EDTA}) = 0{,}02$ mol/l]. Determine and record the end point when the reddish purple colour of the solution is altered to blue.

NOTE Titration carried out on an opal glass or an opal plastic board being permeated with the light of a tungsten lamp will make the end point clearer.

9.3 Blank test

Carry out the procedure described in 9.2 with blank solution (B1) or (B′1) (see ISO 10058-1:2008, Annex A).

9.4 Calculation

Calculate the mass fraction of magnesium oxide, w_{MgO}, expressed as a percentage, using Equation (8), with the volumes of standard volumetric EDTA solution used for the titrations in 9.2 and 9.3.

$$w_{\mathrm{MgO}} = \frac{(V_1 - V_2) \times F \times 0{,}000\ 806\ 4}{m} \times \frac{250}{20} \times 100 - (w_{\mathrm{CaO}} \times 0{,}719) \tag{8}$$

where

V_1 is the volume of standard volumetric EDTA solution used in 9.2, in millilitres (ml);

V_2 is the volume of standard volumetric EDTA solution used in 9.3, in millilitres (ml);

F is the factor of standard volumetric EDTA solution;

m is the mass of the test portion in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.2.4, in grams (g);

w_{CaO} is the mass fraction of calcium oxide determined in 8.4 or ISO 10058-3:2008, Clause 3 or Clause 4, expressed as a percentage.

10 Determination of sodium oxide by flame photometry

10.1 Principle

A portion of a stock solution (S2) (see ISO 10058-1:2008, Annex A) is sprayed into the flame of a flame emission spectrophotometer and the emission intensity of sodium is measured.

10.2 Reagents

Prepare the following reagents in addition to any in ISO 26845 that are required.

10.2.1 Mixed standard solution 1, Na_2O 0,05 mg/ml, K_2O 0,05 mg/ml.

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

10.2.2 Series 1 solution for calibration.

Transfer precisely adequate aliquot portions of the mixed standard solution 1 to separate 100 ml volumetric flasks stepwise. Add a specified amount of magnesium oxide solution (MgO 10 mg/ml) corresponding to magnesium oxide content in the samples (e.g. 16 ml of magnesium oxide solution in the case of the 82 % by mass of magnesium oxide). Add 5 ml of hydrochloric acid (1+1) and dilute to the mark with water. Table 6 provides an example of the series 1 solution for calibration. Prepare calibration standards to cover the range of concentrations being analysed. For the initial calibration, there shall be a zero and at least five other standards. Subsequently, for re-standardization three standards including a zero should be sufficient. Table 6 gives examples of standards that could cover the entire magnesite and dolomite range.

NOTE The amount of the magnesium oxide solution is approximately 5 ml per 10 % of the mass of the magnesium oxide content.

Table 6 — Example of series 1 solution for calibration

Series 1 solution No.	Magnesium oxide solution (MgO 10 mg/ml) ml	Hydrochloric acid (1+1) ml	Mixed standard solution 1 ml	Concentration of solution mg/100 ml	
				Na_2O	K_2O
1	16	5	0	0	0
2	16	5	1	0,05	0,05
3	16	5	2	0,10	0,10
4	16	5	3	0,15	0,15
5	16	5	4	0,20	0,20
6	16	5	5	0,25	0,25
7	16	5	6	0,30	0,30
8	16	5	8	0,40	0,40
9	16	5	10	0,50	0,50
10	16	5	15	0,75	0,75
11	16	5	20	1,00	1,00
12	16	5	25	1,25	1,25
13	16	5	30	1,50	1,50
14	16	5	40	2,00	2,00

NOTE 1 ml of magnesium oxide solution corresponds to 5 % by mass. In this case, the content of magnesium oxide is 80 % by mass.

10.3 Procedure

Spray a portion of stock solution (S2) into the flame of a flame emission spectrophotometer and measure the emission intensity at a wavelength of 589,0 nm.

An optical filter for sodium may be used.

10.4 Blank test

Carry out the procedure described in 10.3 using blank solution (B2).

10.5 Plotting of calibration graph

Prepare a calibration by using the series 1 solutions described in 10.2.2. Carry out the procedure described in 10.3, and plot the relation between the emission intensity and mass of sodium oxide.

NOTE The measurement of the solution for calibration is carried out simultaneously with the measurements for the stock and blank solutions. The calibration line is newly prepared for each measurement.

10.6 Calculation

Calculate the mass fraction of sodium oxide, $w_{\mathrm{Na_2O}}$, expressed as a percentage, using Equation (9), with the mass of sodium oxide derived from the emission intensity obtained in 10.3 and 10.4, and the calibration prepared in 10.5.

$$w_{\mathrm{Na_2O}} = \frac{m_{\mathrm{s}} - m_{\mathrm{b}}}{m} \times 100 \qquad (9)$$

where

m_{s} is the mass of sodium oxide in stock solution (S2), in grams (g);

m_{b} is the mass of sodium oxide in blank solution (B2), in grams (g);

m is the mass of the test portion in ISO 10058-1:2008, 8.3.2.3.1, in grams (g).

11 Determination of potassium oxide by flame spectrophotometry

11.1 Principle

A portion of a stock solution (S2) (see ISO 10058-1:2008, Annex A) is sprayed into the flame of a flame emission spectrophotometer and the emission intensity of potassium is measured.

11.2 Procedure

Spray a portion of stock solution (S2) into the flame of a flame emission spectrophotometer and measure the emission intensity at a wavelength of 766,5 nm.

An optical filter for potassium may be used.

11.3 Blank test

Using blank solution (B2) (see ISO 10058-1:2008, Annex A), carry out the procedure in 11.2.

11.4 Plotting of calibration graph

Transfer the series 1 solutions for calibration as described in 10.2.2, carry out the procedure described in 11.2 and plot the relation between the emission intensity and mass of potassium oxide.

NOTE See Note to 10.5.

11.5 Calculation

Calculate the mass fraction of potassium oxide, $w_{\mathrm{K_2O}}$, expressed as a percentage, using Equation (10), with the mass of potassium oxide derived from the emission intensity obtained in 11.2 and 11.3, and the calibration prepared in 11.4.

$$w_{\mathrm{K_2O}} = \frac{m_{\mathrm{s}} - m_{\mathrm{b}}}{m} \times 100 \qquad (10)$$

where

m_{s} is the mass of potassium oxide in stock solution (S2), in grams (g);

m_{b} is the mass of potassium oxide in blank solution (B2), in grams (g);

m is the mass of the test portion in ISO 10058-1:2008, 8.3.2.3.1, in grams (g).

12 Determination of chromium(III) oxide

12.1 General

The determination of chromium(III) oxide is carried out using one of the following two methods:

a) diphenylcarbazide method (see 12.2);

b) EDTA method (see 12.3).

12.2 Diphenylcarbazide method

12.2.1 Principle

Ammonium ceric nitrate solution is added to an aliquot portion of stock solution (S1) or (S′1) (see ISO 10058-1:2008, Annex A) and previously evaporated with sulfuric acid to remove any chlorides. Sodium azide solution is added in order to destroy excess ceric ions, followed by diphenylcarbazide solution. The absorbance of the solution is measured at 540 nm.

12.2.2 Procedure

12.2.2.1 Transfer an aliquot portion, determined using Table 7, of stock solution (S1) or (S′2) to a 100 ml beaker, add 5 ml of sulfuric acid (1+9) and evaporate until dry. To the dry residue, add 2 ml of sulfuric acid and 15 ml of water. Warm to dissolve as much of the residue as possible. Filter, if necessary, through a fine filter paper and wash the residue with warm water. Evaporate to 20 ml, add 2 ml of the ammonium ceric nitrate solution and allow to stand on a steam bath for 25 min. Cool to 10 °C and add the sodium azide solution, drop by drop, to destroy the colour of the excess ceric ion.

NOTE The aliquot volumes of stock solution (S1) or (S′1) depend on the content of chromium(III) oxide in the sample as shown in Table 7.

Table 7 — Aliquot volumes of stock solution (S1) or (S′1)

Mass fraction of chromium(III) oxide %	Volume of stock solution (S1) or (S′1) ml
< 0,6	10
⩾ 0,6, < 1,2	5
⩾ 1,2	2 [a]

[a] Alternatively, transfer precisely 20 ml of the stock solution to a 100 ml volumetric flask and dilute to the mark with water. An aliquot portion of 10 ml may be used instead of the 2 ml of the stock solution.

12.2.2.2 Transfer the solution to a 100 ml volumetric flask containing 3 ml of the sulfuric acid (1+9) and dilute to 90 ml with water. Add 2 ml of the diphenylcarbazide solution, dilute to 100 ml with water and mix. Allow to stand for 5 min. Measure the absorbance of the solution against water in 10 mm cells at a wavelength of 540 nm.

12.2.3 Blank test

Using an aliquot portion of blank solution (B1) or blank solution (B′1) (see ISO 10058-1:2008, Annex A), carry out the procedure in accordance with 12.2.2. The volumes of the aliquot portion of blank solution are the same as those for the corresponding stock solution.

12.2.4 Plotting of calibration graph

Transfer 0 ml (as reference), 1 ml, 2 ml, 3 ml, 4 ml and 5 ml aliquot portions [0 mg to 0,125 mg as chromium(III) oxide] of the dilute standard chromium(III) oxide solution (Cr_2O_3 0,025 mg/ml) to separate 100 ml volumetric flasks. Treat these solutions in accordance with 12.2.2.1, add 5 ml of sulfuric acid (1+9), and measure the absorbance against the reference solution. Plot the relation between the absorbance measurements and mass of chromium(III) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

12.2.5 Calculation

Calculate the mass fraction of chromium(III) oxide, $w_{\mathrm{Cr_2O_3}}$, expressed as a percentage, using Equation (11). Use the amount of chromium(III) oxide derived from the absorbance in 12.2.2.1 and 12.2.3, and the calibration in 12.2.4.

$$w_{\mathrm{Cr_2O_3}} = \frac{m_\mathrm{s} - m_\mathrm{b}}{m} \times \frac{250}{V} \times 100 \qquad (11)$$

where

m_s is the mass of chromium(III) oxide in the aliquot portion of stock solution (S1) or (S′1), in grams (g);

m_b is the mass of chromium(III) oxide in the aliquot portion of blank solution (B1) or (B′1), in grams (g);

m is the mass of the test portion in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.3.3, in grams (g);

V is the volume of the aliquot portion taken from stock solution (S1) or (S′1) in 12.2.2.1, in millilitres (ml).

12.3 EDTA method

NOTE This method is suitable for determining chromium(III) oxide contents from 0,1 % to 1 %. Larger amounts may be determined using smaller aliquot portions if their concentrations do not exceed the usual limits for these materials. However, the EDTA complex, which is also formed with iron(III) oxide, has only a slight absorbing effect (1 % Fe_2O_3 corresponding with 0,1 % Cr_2O_3) so that a correction can be made by calculation when the ferric oxide content is known. For Cr_2O_3 up to approximately 0,1 %, the diphenylcarbazide method (see 12.2) is to be used.

12.3.1 Principle

A solution of ethylenediaminotetraacetic acid is added to an aliquot portion of stock solution (S1) which is boiled and then buffered with acetate. The absorbance of the stable violet complex formed is measured, the absorbance maximum of which lies at a wavelength of 540 nm.

12.3.2 Procedure

Pipette 10 ml of stock solution (S1) or (S′1) (see ISO 10058-1:2008, Annex A) into a 250 ml beaker. Add one drop of hydrogen peroxide solution and neutralize by adding ammonium hydroxide solution (concentrated) drop by drop until a precipitate forms. Dissolve the precipitate in a quantity of hydrochloric acid (1+1), and add in excess 5 drops of hydrochloric acid. Add 10 ml of EDTA solution, cover with a watch glass and heat to boiling. Boil gentry for 15 min. Add 10 ml of buffer solution and allow to cool. Transfer the solution to a 100 ml volumetric flask, fill up to the volume and stir vigorously. Measure, in 4 cm cells, the absorbance of the solution brought to 20 °C ± 2 °C against water at a wavelength of 540 nm.

12.3.3 Plotting the calibration graph

Pipette 0 ml, 1 ml, 3 ml, 5 ml, 10 ml, 15 ml, 20 ml and 30 ml volumes [0 mg to 0,75 mg as chromium(III) oxide] of dilute standard chromium(III) oxide solution (Cr_2O_3 0,025 mg/ml) into eight 250 ml beakers. Carry out the procedure in accordance with 12.3.2. Use these results to plot a calibration graph making the necessary corrections for the blank solutions.

12.3.4 Determination of the correction factor for the influence of iron

Pipette 0 ml, 5 ml, 10 ml, 15 ml and 20 ml volumes [0 mg to 4 mg as chromium(**III**) oxide] of dilute standard iron(**III**) oxide solution (Fe_2O_3 0,2 g/ml) into five 250 ml beakers. Carry out the procedure in accordance with 12.3.2. Use these results to plot a calibration graph making the necessary corrections for the blank solution.

Calculate the correction factor, C, for the influence of iron using Equation (12):

$$C = K \frac{A_{\text{corr}}}{m_3} \tag{12}$$

where

A_{corr} is the absorbance of iron(**III**) oxide calibration solutions corrected for the absorbance of the blank;

m_3 is the corresponding mass of iron(**III**) oxide per 100 ml of the calibration solution, in milligrams (mg);

K is the factor of the method, calculated using Equation (13):

$$K = \frac{m_4}{A_{\text{(calib)corr}}} \tag{13}$$

where

m_4 is the mass of chromium(**III**) oxide per 100 ml of the calibration solution, in milligrams (mg);

$A_{\text{(calib)corr}}$ is the absorbance of these calibration solutions corrected for the absorbance of the blank solutions.

NOTE For the conditions described here (measurement at 546 nm, 4 cm cells), the value of K is about 9,5 and the value of the correction factor, C, for the influence of iron is about 0,01.

12.3.5 Calculation

Calculate the mass fraction of chromium(**III**) oxide, $w_{Cr_2O_3}$, expressed as a percentage, using Equation (14):

$$w_{Cr_2O_3} = \frac{m_1 - m_2}{m_0} \times 0{,}5 \tag{14}$$

where

m_0 is the mass of the test portion, in grams (g);

m_1 is the mass of chromium(**III**) oxide in the sample solution, in grams (g);

m_2 is the mass of chromium(**III**) oxide in the blank solution, in grams (g).

Correct the mass fraction of chromium(**III**) oxide for the influence of iron(**III**) oxide, $w_{(Cr_2O_3)\,\text{corr}}$, using Equation (15):

$$w_{(Cr_2O_3)\,\text{corr}} = w_{(Cr_2O_3)\,\text{det}} \left(C \times D \right) \tag{15}$$

where

$w_{(\mathrm{Cr_2O_3})\ \mathrm{det}}$ is the mass fraction of chromium(III) oxide taken from the calibration graph, expressed as a percentage;

C is the correction factor for the influence of iron, determined in 12.3.4;

D is the mass fraction of iron(III) oxide of the sample determined in accordance with Clause 5, expressed as a percentage.

13 Determination of zirconium oxide by xylenol orange absorption spectroscopy

13.1 Principle

Transfer an aliquot portion of stock solution (S1) or (S′1) (see ISO 10058-1:2008, Annex A), add aluminium chloride and hydrazinium chloride as masking agents for interfering ions, and adjust the concentration of hydrochloric acid. Add xylenol orange to colour the solution and measure the absorbance.

13.2 Procedure

13.2.1 Transfer an aliquot portion, determined using Table 8, of stock solution (S1) or (S′1) to a 50 ml volumetric flask (a) and add 8 ml of aluminium chloride solution.

NOTE The aliquot volumes of stock solution (S1) or (S′1) depend on the content of zirconium oxide in the sample as shown in Table 8.

Table 8 — Aliquot volumes of stock solution (S1) or (S′1)

Mass fraction of zirconium oxide %	Aliquot volume of stock solution ml
< 0,20	25
⩾ 0,2, < 0,50	10
⩾ 0,50	5

13.2.2 Transfer the same volume of stock solution in 13.2.1 to a 100 ml beaker, add a drop of the methyl orange solution as an indicator and add ammonia solution (1+1) dropwise until the colour of the solution becomes yellow. Record the volume, x, in millilitres of ammonia added.

13.2.3 Add $\left[4{,}5-(7/x)\right]$ ml of hydrochloric acid (1+1), 8 ml of aluminium chloride solution and 5 ml of hydrazinium chloride into a 50 ml volumetric flask (a). Dilute to 40 ml with water and shake lightly. Add 4,5 ml of hydrochloric acid (1+1), 8 ml of aluminium chloride solution, and 5 ml of hydrazinium dichloride solution into another 50 ml volumetric flask (b). Dilute to 40 ml with water and shake lightly. Put volumetric flasks (a) and (b) into a boiling water bath for 15 min and cool in flowing water.

13.2.4 Add precisely 5 ml of xylenol orange solution into volumetric flasks (a) and (b), dilute to the mark with water and allow to stand for 10 min. Measure the absorbance of solution (a) in a 10 mm cell at the wavelength of 535 nm against solution (b).

13.3 Blank test

Carry out the procedure described in 13.2 with blank test solution (B1) or (B′1) (see ISO 10058-1:2008, Annex A) corresponding to stock solution (S1) or (S′1). Ensure that the volumes of blank test solution (B1) or (B′1) are the same as that of stock solutions (S1) or (S′1).

13.4 Plotting of calibration graph

Transfer 0 ml, 2 ml, 4 ml, 6 ml, 8 ml and 10 ml aliquot portions [0 mg to 0,05 mg as zirconium oxide] of the dilute standard zirconium oxide solution (ZrO_2 0,005 mg/ml) to separate 50 ml volumetric flasks. Add to each 4,5 ml of hydrochloric acid (1+1), 8 ml of aluminium chloride solution and 5 ml of hydrazinium dichloride solution. Dilute to 40 ml with water and carry out the procedure described in 13.2.3 and 13.2.4. Plot the relation between the absorbance and mass of zirconium oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

NOTE See Note to 10.5.

13.5 Calculation

Calculate the mass fraction of zirconium oxide, $w_{\mathrm{ZrO_2}}$, expressed as a percentage, using Equation (16). Use the amount of zirconium oxide derived from the absorbance in 13.2.4 and 13.3, and the calibration graph in 13.4.

$$w_{\mathrm{ZrO_2}} = \frac{m_{\mathrm{s}} - m_{\mathrm{b}}}{m} \times \frac{250}{V} \times 100 \tag{16}$$

where

m_{s} is the mass of stock solution (S1) or (S′1) of zirconium oxide, in grams (g);

m_{b} is the mass of the blank test solution (B1) or (B′1) of zirconium oxide, in grams (g);

V is the volume of stock solution (S1) or (S′1), in millilitres (ml);

m is the mass of the test portion in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.3.3, in grams (g).

NOTE See Note to 10.5.

14 Determination of phosphorus(V) oxide by molybdenum blue method

14.1 Principle

An aliquot portion of stock solution (S1) or (S′1) (see ISO 10058-1:2008, Annex A) is transferred. After adjusting the acid concentration, ammonium molybdate and L (+)-ascorbic acid are added. The absorbance is measured when molybdenum blue appears by heating.

14.2 Procedure

14.2.1 Pipette an aliquot volume, determined using Table 9, of stock solution (S1) or (S′1) precisely into a 100 ml volumetric flask.

NOTE The aliquot volumes of stock solution (S1) or (S′1) depend on the content of phosphorus(V) oxide in the sample as shown in Table 9.

Table 9 — Aliquot volumes of stock solution (S1) or (S′1)

Mass fraction of phosphorus (V) oxide %	Volume of stock solution (S1) or (S′1) ml
$< 0{,}4$	25
$\geqslant 0{,}4$, $< 1{,}0$	10
$\geqslant 1{,}0$, $< 2{,}0$	5
$\geqslant 2{,}0$	2 [a]

[a] Transfer precisely 20 ml to a 100 ml volumetric flask and dilute to the mark with water. An aliquot portion of 10 ml can be used instead of the 2 ml of the stock solution.

14.2.2 Add 2 to 3 drops of p-nitrophenol solution as an indicator and drop sodium hydroxide solution until the colour changes to yellow. Drip sulfuric acid (1+1) to decolourize and add an excess of 2 to 3 drops. Add 10 ml of ammonium molybdate solution and 2 ml of L (+)-ascorbic acid solution and dilute to the mark with water. Heat for 15 min on a steam bath and then cool under running water. Measure the absorbance of the solution against water in 10 mm cells at a wavelength of 830 nm.

14.3 Blank test

Using an aliquot portion of blank solution (B1) or (B′1) (see ISO 10058-1:2008, Annex A), carry out the procedure described in 14.2.

14.4 Plotting of calibration graph

Transfer 0 ml (as reference), 5 ml, 10 ml, 15 ml, 20 ml and 25 ml aliquot portions [0 mg to 0,25 mg as phosphorus(V) oxide] of the dilute standard phosphorus(V) oxide solution (P_2O_5 0,01 mg/ml) to separate 100 ml volumetric flasks and carry out the procedure described in 14.2. Plot the relation between the absorbance and mass of phosphorus(V) oxide and prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

14.5 Calculation

Calculate the mass fraction of phosphorus (V) oxide, $w_{\mathrm{P_2O_5}}$, expressed as a percentage, using Equation (17) from the absorbance obtained in 14.2.2 and 14.3, and the calibration line prepared in 14.4.

$$w_{\mathrm{P_2O_5}} = \frac{m_{\mathrm{s}} - m_{\mathrm{b}}}{m} \times \frac{250}{V} \times 100 \qquad (17)$$

where

m_{s} is the mass of phosphorus(V) oxide in the aliquot portion of stock solution (S1) or (S′1), in grams (g);

m_{b} is the mass of phosphorus(V) oxide in the aliquot portion of blank solution (B1) or (B′1), in grams (g);

V is the aliquot volume of stock solution (S1) or (S′1), in millilitres (ml);

m is the mass of the test portion in ISO 10058-1:2008, 8.2.2.3.1 or 8.2.3.3, in grams (g).

15 Test report

Prepare a test report as described in ISO 26845.

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