# INTERNATIONAL STANDARD

# ISO 21587-2

First edition
2007-03-01

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

## Part 2:
## Wet chemical analysis

*Analyse chimique des produits réfractaires d'aluminosilicates (méthode alternative à la méthode par fluorescence de rayons X) —*

*Partie 2: Méthodes d'analyse chimique par voie humide*

Reference number
ISO 21587-2:2007(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.

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 21587-2 was prepared by Technical Committee ISO/TC 33, *Refractories*.

ISO 21587 consists of the following parts, under the general title *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*

**INTERNATIONAL STANDARD** **ISO 21587-2:2007(E)**

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

## Part 2:
## Wet chemical analysis

## 1 Scope

This part of ISO 21587 specifies traditional ("wet") methods for the chemical analysis of aluminosilicate refractory products and raw materials.

The methods are applicable to the determination of the following:

- silicon(IV) oxide ($SiO_2$)
- aluminium oxide ($Al_2O_3$)
- iron(III) oxide (total iron oxide calculated as $Fe_2O_3$)
- titanium(IV) oxide ($TiO_2$)
- manganese(II) oxide (MnO)
- calcium oxide (CaO)
- magnesium oxide (MgO)
- sodium oxide ($Na_2O$)
- potassium oxide ($K_2O$)
- chromium(III) oxide ($Cr_2O_3$)
- zirconium oxide ($ZrO_2$)
- phosphorous(V) oxide ($P_2O_5$)

This part of ISO 21587 gives alternatives to the X-ray fluorescence (XRF) method given in ISO 12677:2003, *Chemical analysis of refractory products by XRF — Fused cast bead method.*

## 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 21587-1:2007, *Chemical analysis of aluminosilicate refractory products (alternative to the X-ray fluorescence method) — Part 1: Apparatus, reagents, dissolution and gravimetric silica*

ISO 26845, *Chemical analysis of refractories — General requirements for wet chemical analysis, atomic absorption spectrometry and inductively coupled plasma methods*

# 3 Determination of residual silicon(IV) oxide in solution

## 3.1 Principle

An aliquot portion of the stock solution (S1) or (S′1), 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 ($m_1 - m_2$) derived in ISO 21587-1:2007, 4.2.2.3 or 4.2.3.3, gives the total silicon(IV) oxide content.

## 3.2 Procedure

This determination should be commenced with little delay after the stock solution (S1) or (S′1) is prepared, as prolonged standing can allow polymerization of silica to occur, leading to low results.

Transfer 10 ml of stock solution (S1) or (S′1) obtained in 4.2.2.3 or 4.2.3.3 of ISO 21587-1:2007 to a 100 ml plastic beaker, and add 2 ml of hydrofluoric acid (1+9), mix with a plastic rod and allow to stand for 10 min. Then 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 to it 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 the reference.

## 3.3 Plotting the calibration graph

Transfer 0, 2, 4, 6, 8 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) into several 100 ml plastic beakers, and to each add 10 ml of blank solution (B1) or (B′1) obtained in 4.2.2.4 or 4.2.3.4 of ISO 21587-1:2007. Treat these solutions and measure the absorbance as given in 3.2, 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.

## 3.4 Calculation

Calculate the mass fraction of silicon(IV) oxide, $w_{\mathrm{SiO_2}}$, as a percentage, using the following equation, with the absorbances obtained by the procedure in 3.2 and the plotting of the calibration graph by the procedure in 3.3.

$$w_{\mathrm{SiO_2}} = \frac{(m_1 - m_2) + (m_3 - m_4) \times \frac{500}{10}}{m} \times 100 \qquad (1)$$

where

$m_1$ is the mass, in g, from 4.2.2.3 or 4.2.3.3 of ISO 21587-1:2007;

$m_2$ is the mass, in g, from 4.2.2.3 or 4.2.3.3 of ISO 21587-1:2007;

$m_3$ is the mass, in g, of silicon(IV) oxide in the aliquot portion of stock solution (S1) or (S′1) as applicable;

$m_4$ is the mass, in g, of silicon(IV) oxide in the aliquot portion of blank solution (B1) or (B′1) as applicable;

$m$ is the mass, in g, of the test portion from 4.2.2.2 or 4.2.3.2 of ISO 21587-1:2007.

# 4 Determination of aluminium oxide

## 4.1 General

The determination of titanium(IV) oxide is carried out using one of the following methods:

a) separation by cupferron-CyDTA-zinc back titration method;

b) CyDTA-zinc back titration method (without separation method).

## 4.2 Separation by cupferron-CyDTA-zinc back titration method

### 4.2.1 Principle

Hydrochloric acid is added to an aliquot portion of stock solution (S1) or (S′1), obtained in 4.2.2.3 or 4.2.3.3 of ISO 21587-1:2007, to adjust the acidity. Iron, titanium, manganese and zirconium are separated from the solution by solvent extraction with cupferron solution and the precipitate removed by dissolution in chloroform. The organic phase is discarded. Excess standard volumetric CyDTA solution is added to the aqueous solution after adjusting the pH with ammonia solution, and a chelate compound of aluminium CyDTA is formed. The pH is further adjusted by the addition of ammonium acetate buffer solution and an equivalent volume of ethanol is added to the solution. The amount of excess CyDTA is determined by back-titration with standard volumetric zinc solution using dithizone as an indicator and the content of aluminium oxide calculated.

### 4.2.2 Procedure

Transfer a 100 ml aliquot portion of the stock solution (S1) or (S′1), obtained in 4.2.2.3 or 4.2.3.3 of ISO 21587-1:2007, to a 500 ml separating funnel and add 20 ml of hydrochloric acid (concentrated) To the solution, add 20 ml of chloroform and 10 ml of cupferron solution. Stopper the funnel and shake vigorously. Release the pressure in the funnel by carefully removing the stopper and rinse the stopper and neck of the funnel with a little water. Allow the layers to separate and withdraw the chloroform layer. Confirm that extraction is complete by checking that the addition of a few drops of the cupferron solution to the aqueous solution does not produce a permanent coloured precipitate. Add further 10 ml portions of the chloroform and repeat the extraction until the chloroform layer is colourless. Wash the stem of the funnel, inside and out, with chloroform, using a polyethylene wash bottle. Discard the chloroform extracts; do not allow them to dry as there can be an explosion risk. Transfer the aqueous solution and washings from the funnel into a 1 l conical flask. Add a few drops of bromophenol blue indicator and ammonia solution (concentrated) until the solution is just alkaline. Re-acidify quickly with hydrochloric acid (concentrated) and add 5 to 6 drops in excess. Cool to room temperature. Add sufficient standard volumetric CyDTA solution [$c$(CyDTA) = 0,05 mol/l] to combine with the aluminium oxide present, and a few millilitres in excess.

NOTE 1 1 ml of standard volumetric CyDTA solution [$c$(CyDTA) = 0,05 mol/l] is equivalent to 1,275 % $Al_20_3$ for a 100 ml aliquot portion.

Add ammonium acetate buffer solution until the indicator turns blue, followed by 10 ml in excess. Add a volume of the ethanol equal to the total volume of the solution. If sulfates are precipitated by the alcohol, add just enough water to re-dissolve them. Add 20 ml of hydroxyammonium chloride and 1 ml to 2 ml of dithizone indicator, and titrate with standard volumetric zinc solution [$c$(Zn) = 0,05 mol/l] from green to the first appearance of a permanent pink colour.

NOTE 2 The end point is often improved by the addition of a little naphthol green B solution (1 g/l) to eliminate any pink colour which can be formed in the solution on addition of the indicator.

### 4.2.3 Blank test

Transfer an aliquot portion of blank solution (B1) or (B′1) obtained in 4.2.2.4 or 4.2.3.4 of ISO 21587-1:2007 and carry out the procedure given in 4.2.2. Use the same volumes of the aliquot portion of blank solution (B1) or (B′1) and standard volumetric CyDTA solution [$c$(CyDTA) = 0,05 mol/l] as those for the corresponding stock solution (S1) or (S′1).

### 4.2.4 Calculation

Calculate the mass fraction of aluminium oxide in the sample as follows.

If the standard volumetric CyDTA solution [$c$(CyDTA) = 0,05 mol/l] is not exactly 0,05 mol/l, calculate the equivalent volume of standard volumetric CyDTA solution.

Calculate the mass fraction of aluminium oxide, $w_{Al_2O_3}$, as a percentage, using the equation

$$w_{Al_2O_3} = 1{,}275\left(V - V_1\right) \qquad (2)$$

where

$V$ is the volume of standard volumetric CyDTA solution [$c$(CyDTA) = 0,05 mol/l] added, in ml;

$V_1$ is the volume of standard volumetric zinc solution [$c$(Zn) = 0,05 mol/l] used in the back-titration, in ml, for a 1 g test sample.

## 4.3 CyDTA-Zinc back titration method (without separation method)

### 4.3.1 Principle

Excess standard volumetric CyDTA solution is added to an aliquot of stock solution (S1) or (S′1). A chelate compound of aluminium CyDTA is formed by adjusting the pH with ammonia water. The pH is further adjusted by the addition of hexamethylenetetramine. The amount of remaining 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 adjusting the content of iron(III) oxide, titanium(IV) oxide, manganese(II) oxide and zirconium oxide by other methods.

### 4.3.2 Procedure

Determination is carried out in accordance with the following procedure.

Transfer precisely 50 ml of stock solution (S1) or (S′1), obtained in 4.2.2.3 or 4.2.3.3 of ISO 21587-1:2007, to a beaker (300 ml), and add 2 ml of hydrochloric acid (1+1). Then add a precisely known amount of standard volumetric CyDTA solution [$c$(CyDTA) = 0,02 mol/l], and dilute to 100 ml with water.

NOTE The relation between the volume of the aliquot of standard volumetric CyDTA solution [$c$(CyDTA) = 0,02 mol/l] and the mass fraction of aluminum oxide, iron(III) oxide, and titanium(IV) oxide are shown in Table 1.

**Table 1 — Aliquots of stock solution (S1) or (S′1) and volume of standard volumetric CyDTA solution**

| Sum of mass fractions of aluminum oxide, iron(III) oxide and titanium(IV) oxide<br>% | Volume of standard volumetric CyDTA solution<br>[$c$(CyDTA) = 0,02 mol/l]<br>ml |
|---|---|
| less than 10 | 20 |
| 10 to 20 | 30 |
| 20 to 30 | 40 |
| 30 to 50 | 50 |

Add 1 g of hexamethylenetetramine and a drop of methyl orange solution as an indicator. Then, drop in ammonia water (1+1), and ammonia water (1+9) of up to pH 3 indicating a slightly orange colour. If ammonia water is added excessively, the pH is adjusted to less than 3 (showing a red colour) by adding hydrochloric acid (1+1). Then, subsequently, carry out the identical adjustment. Add 5 g of hexamethylenetetramine of up to pH 5,5 to 5,8, add 4 or 5 drops of xylenol orange solution as an indicator, and titrate with standard volumetric zinc solution [$c$(Zn) = 0,02 mol/l]. In the vicinity of the end point, titration is carried out gently while mixing, and the end point is decided when the colour changes from yellow to the first appearance of a permanent reddish colour.

### 4.3.3 Blank test

Transfer an aliquot of blank solution (B1) or (B′1), obtained in 4.2.2.4 or 4.2.3.4 of ISO 21587-1:2007, and carry out the procedure in accordance with 4.3.2. The volumes of the aliquot of blank solution (B1) or (B′1) and standard volumetric CyDTA solution [$c$(CyDTA) = 0,02 mol/l] are the same as those for the corresponding stock solution (S1) or (S′1).

### 4.3.4 Calculation

Calculate the mass fraction of aluminum oxide in the sample, $w_{\mathrm{Al_2O_3}}$, as a percentage, using the following equation:

$$w_{\mathrm{Al_2O_3}} = \frac{(V_2 - V_1) \times F \times 0{,}001\,019\,6}{m} \times \frac{500}{50} \times 100 - \left[0{,}638 \times \left(w_{\mathrm{Fe_2O_3}} + w_{\mathrm{TiO_2}}\right) + 0{,}729 \times w_{\mathrm{MnO}} + 0{,}414 \times w_{\mathrm{ZrO_2}}\right] \qquad (3)$$

where

$V_1$ is the used quantity, in ml, of standard volumetric zinc solution [$c$(Zn) = 0,02 mol/l] in 4.3.2;

$V_2$ is the used quantity, in ml, of standard volumetric zinc solution [$c$(Zn) = 0,02 mol/l] in 4.3.3;

$F$ is the factor of standard volumetric zinc solution [$c$(Zn) = 0,02 mol/l];

$m$ is the mass, in g, of sample in 4.2.2.2 or 4.2.3.2 in ISO 21587-1:2007;

$w_{\mathrm{Fe_2O_3}}$ is the mass fraction, in %, of iron(III) oxide determined in Clause 5 or ISO 21587-3:2007 (Clause 4);

$w_{\mathrm{TiO_2}}$ is the mass fraction, in %, of titanium(IV) oxide determined in Clause 6 or ISO 21587-3:2007 (Clause 5);

$w_{\mathrm{MnO}}$ is the mass fraction, in %, of manganese(II) oxide determined in Clause 7 or ISO 21587-3:2007 (Clause 6 or 18);

$w_{\mathrm{ZrO_2}}$ is the mass fraction, in %, of zirconium oxide determined in Clause 13 or ISO 21587-3:2007 (Clause 12).

## 5 Determination of iron(III) oxide

### 5.1 Principle

Two methods are given, using alternative reducing agents for the ferric oxide in solution. The iron in stock solution (S1) or (S′1) is reduced with L(+)-ascorbic acid or hydroxyammonium chloride, 1,10-phenanthroline is added, and the pH is adjusted by adding ammonium acetate. The absorbance is measured.

## 5.2 Procedure

Transfer 5 ml of stock solution (S1) or (S′1) obtained in 4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007 into a 100 ml volumetric flask.

5 ml of aliquot volume is a typical amount. An aliquot volume of stock solution (S1) or (S′1) should be adjusted according to the mass fraction of iron(III) oxide in the sample as shown in Table 2.

**Table 2 — Aliquot volume of stock solution (S1) or (S′1)**

| Mass fraction of iron(III) oxide<br>% | Aliquot volume of stock solution (S1) or (S′1)<br>ml |
|---|---|
| Less than 0,5 | 25 |
| 0,5 to 1,5 | 10 |
| More than 1,5 | 5 |

Reduce the solution by one of the following methods.

a) Reduce with ascorbic acid by diluting to about 60 ml with water, and adding 5 ml of L(+)-tartaric acid solution and 2 ml of L(+)-ascorbic acid solution while shaking. Add 10 ml of 1,10-phenanthrolinium chloride solution (1 g/l) and 10 ml of ammonium acetate solution (20 %). Dilute to the mark with water, then allow to stand for 30 min.

b) Reduce with hydroxyammonium chloride by adding 2 ml of hydroxyammonium chloride solution or L(+) ascorbic acid solution (100 g/l), 5 ml of 1,10-phenanthroline solution (10 g/l) and 5 ml of ammonium acetate solution (100 g/l). Allow the solution to stand for 15 min, dilute to the mark and mix. The colour is stable between 15 min and 75 min after the addition of the ammonium acetate solution.

Measure the absorbance of the solution in a 10 mm cell at a wavelength of 510 nm or a colour filter against water in a suitable instrument.

## 5.3 Blank test

Transfer the same volume of the blank solution (B1) or (B′1), obtained in 4.2.2.4 or 4.2.3.4 of ISO 21587-1:2007, as that of stock solution (S1) or (S′1), and carry out the appropriate procedure given in 5.2 a) or b), as applicable.

## 5.4 Plotting the calibration graph

Transfer 0 (as reference), 5,0, 10,0 and 15,0 ml 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) into several 100 ml volumetric flasks. Treat these solutions in accordance with 5.2 a) or b) and measure the absorbance against the reference solution. Then plot the relation between the absorbances 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) oxide in the sample, $w_{\mathrm{Fe_2O_3}}$, as a percentage, using the following equation. Use the amount of iron(III) oxide that is derived from the absorbance in 5.2 and the calibration in 5.4.

$$w_{\mathrm{Fe_2O_3}} = \frac{m_1 - m_2}{m} \times \frac{500}{V} \times 100 \qquad (4)$$

where

$m_1$ is the mass, in g, of iron(III) oxide in the aliquot portion of stock solution (S1) or (S′1);

$m_2$ is the mass, in g, of iron(III) oxide in the aliquot portion of blank solution (B1) or (B′1);

$m$ is the mass, in g, of the test portion in 4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007;

$V$ is the volume, in ml, of the aliquot portion taken from stock solution (S1) or (S′1) in 5.2, i.e. 5 ml.

# 6 Determination of titanium(IV) oxide

## 6.1 General

The determination of titanium(IV) oxide is carried out using one of the following methods:

a) di-antipyrylmethane (DAM) absorption spectrophotometric method;

b) hydrogen peroxide absorption spectrophotometric method.

## 6.2 DAM absorption spectrophotometric method

### 6.2.1 Principle

An aliquot portion of stock solution (S1) or (S′1) is adjusted for acidity. The iron is reduced with an addition of L(+)-ascorbic acid and diantipyrylmethane. The absorbance of the colour produced is then measured.

### 6.2.2 Procedure

Carry out the determination using the following procedure.

Transfer 5 ml of stock solution (S1) or (S′1), obtained in 4.2.2.3 or 4.2.3.3 of ISO 21587-1:2007, into a 50 ml volumetric flask.

5 ml of aliquot volume is a typical amount. An aliquot volume of stock solution (S1) or (S′1) should be adjusted according to the mass fraction of titanium(IV) oxide in the sample as shown in Table 3.

**Table 3 — Aliquot taken from stock solution (S1) or (S′1)**

| Mass fraction of titanium(IV) oxide<br>% | Aliquot taken<br>ml |
|---|---|
| Less than 0,5 | 25 |
| 0,5 to 1,5 | 10 |
| More than 1,5 | 5 |

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

### 6.2.3 Blank test

Using the same volume of blank solution (B1) or (B′1), obtained in 4.2.2.4 or 4.2.3.4 in ISO 21587-1:2007, as that of stock solution (S1) or (S′1), carry out the procedure described in 6.2.2.

### 6.2.4 Plotting the calibration graph

Transfer 0 (as reference), 5, 10, 15, 20 and 25 ml aliquot portions [0 mg to 0,25 mg as titanium(IV) oxide] of the dilute standard titanium(IV) oxide solution ($TiO_2$ 0,01 mg/ml) into each of several 50 ml volumetric flasks. Treat these solutions as in 6.2.3 and 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.

### 6.2.5 Calculation

Calculate the mass fraction of titanium(IV) oxide, $w_{\mathrm{TiO_2}}$, in the sample as a percentage, using the following equation, with the amount of titanium(IV) oxide that is derived from the absorbance in 6.2.2 and 6.2.3, and the calibration in 6.2.4.

$$w_{\mathrm{TiO_2}} = \frac{m_1 - m_2}{m} \times \frac{500}{V} \times 100 \qquad (5)$$

where

$m_1$ is the mass, in g, of titanium(IV) oxide in the aliquot portion of stock solution (S1) or (S′1),

$m_2$ is the mass, in g, of titanium(IV) oxide in the aliquot portion of blank solution (B1) or (B′1);

$V$ is the volume, in ml, of the aliquot portion taken from the stock solution in 6.2.2;

$m$ is the mass, in g, of the test portion in 4.2.2.2 or 4.2.3.2 in ISO 21587-1:2007.

## 6.3 Hydrogen peroxide absorption spectrophotometric method

### 6.3.1 Principle

The colour from the iron content of the stock solution (S1) or (S′1) is masked by an addition of phosphoric acid and the titanium is reacted with hydrogen peroxide. The absorbance is measured.

### 6.3.2 Procedure

Carry out the determination using the following procedure.

Transfer 20 ml of stock solution (S1) or (S′1) obtained in 4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007 into each of two 50 ml volumetric flasks, A and B.

To each flask, add 10 ml of phosphoric acid (2+3), and to flask A only add 10 ml of hydrogen peroxide solution (6 % by mass). Allow to stand for 10 min. Dilute each to the mark with water and shake vigorously. Measure the absorbance of the solution in flask A against that in flask B, using a 10 mm cell at a wavelength of 398 nm or using a colour filter in a suitable instrument.

### 6.3.3 Blank test

Using the same volume of blank solution (B1) or (B′1) as that of the aliquot portion of stock solution (S1) or (S′1), obtained in 4.2.2.4 or 4.2.3.4 in ISO 21587-1:2007, carry out the procedure described in 6.3.2.

### 6.3.4 Plotting the calibration graph

Transfer 0 (as reference), 10, 20, 30 and 40 ml aliquot portions [0 mg to 4,0 mg as titanium(IV) oxide] of the dilute standard titanium(IV) oxide solution ($TiO_2$ 0,1 mg/ml) into five 100 ml volumetric flasks, and treat these solutions as in 6.3.2. Then measure the absorbance against the reference solution (0 ml). Plot the relation between the absorbance and the mass of titanium(IV) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

### 6.3.5 Calculation

Calculate the mass fraction of titanium(IV) oxide, $w_{\mathrm{TiO_2}}$, as a percentage, using the following equation, with the amount of titanium(IV) oxide that is derived from the absorbance in 6.3.2 and 6.3.3, and the calibration in 6.3.4.

$$w_{\mathrm{TiO_2}} = \frac{m_1 - m_2}{m} \times \frac{500}{50} \times 100 \qquad (6)$$

where

$m_1$ is the mass, in g, of titanium(IV) oxide in the aliquot portion of stock solution (S1) or (S′1);

$m_2$ is the mass, in g, of titanium(IV) oxide in the aliquot portion of blank solution (B1) or (B′1);

$m$ is the mass, in g, of the test portion in 4.2.2.2 or 4.2.3.2 in ISO 21587-1:2007.

## 7 Determination of manganese(II) oxide by permanganate absorption spectrophotometric method

### 7.1 Principle

Chloride ions are removed from aliquot portions of stock solutions (S1) or (S′1) by evaporating with sulfuric acid until white fumes appear. The manganese is converted to permanganate by oxidation with potassium periodate, and the absorbance is measured. Phosphoric acid is used to prevent the interference from iron(III) salts and the possible precipitation of manganese(IV) oxide.

### 7.2 Procedure

Transfer a 50 ml aliquot portion of stock solution (S1) or (S′1) obtained in 4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007 into a 250 ml beaker.

Add 10 ml of sulfuric acid (1+1), and 5 ml of nitric acid (65 % by mass) and evaporate to remove chloride ions (and in the case of solution (S′1), to decompose the poly(ethylene oxide)), until the sulfuric acid begins to fume intensively. After cooling, add 20 ml of nitric acid (65 % by mass), 10 ml of phosphoric acid (1+9) and approximately 50 ml of water. Boil to dissolve any salts and to remove nitrous gases, then allow to cool slightly. Add 0,2 g of potassium periodate and boil again for 2 min. and then transfer to a steam bath for 10 min. Allow the solution to cool and transfer to a 100 ml volumetric flask. Dilute to the mark of the flask with water and shake vigorously. Measure the absorbance of the solution in a 10 mm cell at a wavelength of 524 nm or using a colour filter against water in a suitable instrument.

### 7.3 Blank test

Use the same volume of blank solution (B1) or (B′1), obtained in 4.2.2.4 or 4.2.3.4 in ISO 21587-1:2007, as the aliquot portion of stock solution (S1) or (S′1) taken, and carry out the procedure described in 7.2.

### 7.4 Plotting the calibration graph

Transfer 0 (as reference), 5, 10, 15, 20 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) into 250 ml beakers. Treat each of these as in 7.2. Then measure the absorbance against the reference solution. Plot the relation between the absorbance and the mass 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, $w_{\text{MnO}}$, as a percentage, using the following equation, with the amount of manganese(II) oxide that is derived from the absorbance in 7.2 and 7.3, and the calibration in 7.4.

$$w_{\text{MnO}} = \frac{m_1 - m_2}{m} \times \frac{500}{20} \times 100 \tag{7}$$

where

$m_1$ is the mass, in g, of manganese(II) oxide in the aliquot portion of stock solution (S1) or (S′1);

$m_2$ is the mass, in g, of manganese(II) oxide in the aliquot portion of blank solution (B1) or (B′1);

$m$ is the mass, in g, of the test portion described in 4.2.2.2 or 4.2.3.2 in ISO 21587-1:2007.

## 8 Determination of calcium oxide

### 8.1 Principle

To an aliquot portion of the solution prepared by hydrofluoric acid attack (S2) a solution of 2,2′,2″-nitrilotriethanol is added to mask interference ions. After adjusting the pH to about 13, an EDTA titration is carried out using screened calcein as indicator.

### 8.2 Procedure

Pipette 100 ml of the solution (S2) from 4.3.2.3 in ISO 21587-1:2007 into a 500 ml conical flask. Add 5 ml of 2,2′, 2″-nitrilotriethanol (1+1), and 10 ml of potassium hydroxide solution (250 g/l), and dilute to about 200 ml with water. Add about 0,015 g of screened Calcein indicator, and titrate with standard volumetric EDTA solution [$c$(EDTA) = 0,012 5 mol/l], from a semi-micro burette, the colour change being from fluorescent green to pink. The end-point is the final change in colour.

NOTE In the event of the sample containing manganese(II) oxide at a level of more than about 0,1 % by mass, the determination is modified as follows.

Transfer precisely 100 ml of stock solution (S2) into a 200 ml beaker and add 5 ml of bromine water. Make the solution slightly alkaline with ammonia solution (1+1), maintaining this alkalinity by the addition of drops of ammonia solution (1+1) as required. After the precipitate has flocculated, filter through a filter paper (coarse) and wash thoroughly with warm water. Collect the filtrate and washings in a 300 ml beaker and acidify the solution with hydrochloric acid (1+1). Boil to completely remove any remaining bromine and evaporate to less than 80 ml. After cooling, carry out the procedure described above.

### 8.3 Blank test

Carry out the procedure in 8.2 using the solution (B2) derived from 4.3.2.4 in ISO 21587-1:2007. Use an equivalent aliquot portion as used for the sample in 8.2.

### 8.4 Calculation

Calculate the mass fraction of calcium oxide, $w_{\text{CaO}}$, as a percentage, using the following equation.

$$w_{\text{CaO}} = \frac{(V - V_1) \times F}{m} \times \frac{250}{100} \times 100 \qquad (8)$$

where

$V$ is the equivalent volume, in ml, of standard volumetric EDTA solution in 8.2;

$V_1$ is the equivalent volume, in ml, of standard volumetric EDTA solution in 8.3;

$F$ is the factor for calcium oxide of standard volumetric EDTA solution [$c$(EDTA) = 0,012 5 mol/l], in 3.1.3 in ISO 21587-1:2007;

$m$ is the mass, in grams, of the test portion in 4.3.2.2 in ISO 21587-1:2007.

## 9 Determination of magnesium oxide

### 9.1 Principle

Hydroxyammonium chloride and 2,2′, 2″-nitrilotriethanol are added to an aliquot portion of stock solution (S2) to mask interfering ions. Then a buffer solution is added to adjust the pH to about 10. The sum of the content of calcium oxide and magnesium oxide is determined by titration with standard volumetric EDTA solution. The content of magnesium oxide is calculated from this volume and that obtained in 8.2.

In the presence of significant amounts of chromium and manganese sodium sulfide is added prior to the titration.

### 9.2 Procedure

Pipette 100 ml of stock solution (S2) obtained in 4.3.2.3 in ISO 21587-1:2007 into a 500 ml conical flask and dilute with water to about 200 ml. Add 10 drops of hydrochloric acid (concentrated), 20 ml of 2,2′, 2″-nitrilotriethanol (1+1), and 25 ml of ammonia solution (concentrated). Add 0,04 g of the methyl thymol blue complexone indicator and titrate with standard volumetric EDTA solution [$c$(EDTA) = 0,012 5 mol/l] solution from a semi-micro burette, until the colour changes from blue to colourless.

NOTE If manganese oxide and/or chromium(III) oxide are present in significant amounts, use the following procedure.

Pipette 100 ml of stock solution (S2) obtained in 4.3.2.3 in ISO 21587-1:2007 into a 500 ml conical flask and dilute with water to about 200 ml. Add 5 ml of hydroxyammonium chloride solution (100 g/l), 20 ml of 2,2′, 2″-nitrilotriethanol (1+1), 2 g of ammonium chloride, 25 ml of ammonia solution (concentrated) and 1 ml of sodium sulfide solution. Add 0,04 g of the methyl thymol blue complexone as an indicator, while stirring, and titrate with standard volumetric EDTA solution [$c$(EDTA) = 0,012 5 mol/l] from a semi-micro burette, until the colour changes from blue to colourless.

### 9.3 Blank test

Carry out the procedure on an equivalent aliquot portion of blank solution (B2) obtained in 4.3.2.4 in ISO 21587-1:2007.

### 9.4 Calculation

Calculate the mass fraction of magnesium oxide in the sample, as a percentage, as follows.

Deduct the volume of standard volumetric EDTA solution [$c$(EDTA) = 0,012 5 mol/l] used in the titration for the determination of calcium oxide from that used for the titration of the sum of calcium and magnesium oxide, after correcting for the volumes in the blank determinations.

Calculate the mass fraction percentage of magnesium oxide, $w_{\mathrm{MgO}}$, as a percentage, using the following equation.

$$w_{\mathrm{MgO}} = \frac{\left(V_2 - V_1 - V_3\right) \times F}{m} \times \frac{250}{100} \times 100 \tag{9}$$

where

$V_1$ is the equivalent volume, in ml, of standard volumetric EDTA solution used in the determination of calcium oxide in 8.2;

$V_2$ is the equivalent volume, in ml, of standard volumetric EDTA solution used in 9.2;

$V_3$ is the equivalent volume, in ml, of standard volumetric EDTA solution used in 9.3;

$F$ is the factor for magnesium oxide of standard volumetric EDTA solution [$c$(EDTA) = 0,012 5 mol/l], in 3.1.3 in ISO 21587-1:2007;

$m$ is the mass, in grams, of the test portion in 4.3.2.2 in ISO 21587-1:2007.

## 10 Determination of sodium oxide by flame photometry

### 10.1 Principle

A portion of stock solution (S2) is sprayed into the flame of a flame photometer, and the emission intensity of sodium is measured.

### 10.2 Reagents

Prepare the following reagents, in addition to any in ISO 26845 and ISO 21587-1 that are required.

**10.2.1 Mixed standard solution 1**, 0,1 mg CaO/ml, 0,1 mg MgO/ml, 0,1 mg $Na_2O$/ml, 0,1 mg $K_2O$/ml.

Transfer 50 ml aliquot portions each of the standard calcium oxide solution (1 mg/ml), standard magnesium oxide solution (1 mg/ml), standard sodium oxide solution (1 mg/ml) and standard potassium oxide solution (1 mg/ml) to a 500 ml volumetric flask, and dilute to the mark with water.

**10.2.2 Series 1 solution for calibration**.

Transfer aliquot portions of mixed standard solution 1 to each of several 100 ml volumetric flasks. To each, add 5 ml of hydrochloric acid (1+1) and the appropriate amount of aluminium oxide solution (10 mg/ml), and dilute to the mark with water. Typical examples of preparation are shown in Table 4.

NOTE The volume of aluminium oxide required is determined by the content of that oxide in the sample.

**Table 4 — Example of series 1 solution for calibration (mass fraction of aluminium oxide is 30 %)**

| Solution for calibration No. | Aluminium oxide solution 1 ml | Hydrochloric acid (1+1) ml | Mixed standard solution 1 ml | Concentration of solution mg/100 ml | | | |
|---|---|---|---|---|---|---|---|
| | | | | CaO | MgO | $Na_2O$ | $K_2O$ |
| 1 | 3 | 5 | 0 | 0 | 0 | 0 | 0 |
| 2 | 3 | 5 | 2 | 0,2 | 0,2 | 0,2 | 0,2 |
| 3 | 3 | 5 | 4 | 0,4 | 0,4 | 0,4 | 0,4 |
| 4 | 3 | 5 | 6 | 0,6 | 0,6 | 0,6 | 0,6 |
| 5 | 3 | 5 | 8 | 0,8 | 0,8 | 0,8 | 0,8 |
| 6 | 3 | 5 | 10 | 1.0 | 1.0 | 1.0 | 1.0 |
| 7 | 3 | 5 | 20 | 2,0 | 2,0 | 2,0 | 2,0 |
| 8 | 3 | 5 | 30 | 3,0 | 3,0 | 3,0 | 3,0 |
| 9 | 3 | 5 | 40 | 4,0 | 4,0 | 4,0 | 4,0 |
| 10 | 3 | 5 | 50 | 5,0 | 5,0 | 5,0 | 5,0 |
| 11 | 3 | 5 | 60 | 6,0 | 6,0 | 6,0 | 6,0 |

## 10.3 Procedure

Spray a portion of stock solution (S2) (4.3.2.3 in ISO 21587-1:2007) into the flame of a flame photometer and measure the emission intensity at a wavelength of 589,0 nm.

NOTE An optical filter for sodium may be used.

## 10.4 Blank test

Carry out the procedure described in 10.3 using blank solution (B2) from 4.3.2.4 in ISO 21587-1:2007.

## 10.5 Plotting the 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.

## 10.6 Calculation

Calculate the mass fraction of sodium oxide, $w_{Na_2O}$, as a percentage, using the following equation, 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.

$$w_{Na_2O} = \frac{m_1 - m_2}{m} \times 100 \qquad (10)$$

where

$m_1$ is the mass, in grams, of sodium oxide in stock solution (S2);

$m_2$ is the mass, in grams, of sodium oxide in blank solution (B2);

$m$ is the mass, in grams, of the test portion in 4.3.2.2 in ISO 21587-1:2007.

## 11 Determination of potassium oxide by flame photometry

### 11.1 Principle

The emission intensity of potassium is measured on stock solution (S2) by spraying into the flame of a flame photometer.

### 11.2 Procedure

Spray a portion of stock solution (S2) obtained in 4.3.2.3 in ISO 21587-1:2007 into the flame of a flame photometer, and measure the emission intensity at a wavelength of 766,5 nm.

NOTE An optical filter for potassium may be used.

### 11.3 Blank test

Carry out the procedure described in 11.2, using blank solution (B2) obtained in 4.3.2.4 in ISO 21587-1:2007.

### 11.4 Plotting the calibration graph

Carry out the procedure described in 11.2, using Series 1 solutions for calibration described in 10.2.2. Plot the relation between the emission intensity and mass of potassium oxide.

### 11.5 Calculation

Calculate the mass fraction of potassium oxide in the sample, $w_{K_2O}$, as a percentage, by using the following equation, 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.

$$w_{K_2O} = \frac{m_1 - m_2}{m} \times 100 \tag{11}$$

where

$m_1$ is the mass, in grams, of potassium oxide in stock solution (S2);

$m_2$ is the mass, in grams, of potassium oxide in blank solution (B2);

$m$ is the mass, in grams, of the test portion in 4.3.2.2 in ISO 21587-1:2007.

## 12 Determination of chromium(III) oxide using diphenylcarbazide

### 12.1 Principle

Ammonium ceric nitrate solution is added to a portion of the stock solution (S1) or (S′1), prepared as in 4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007, previously evaporated with sulfuric acid to remove chlorides. Sodium azide solution is then added in order to destroy excess ceric ions, followed by diphenylcarbazide solution. The absorbance of the complex is measured at 540 nm.

### 12.2 Procedure

Transfer 5 ml of the stock solution (S1) or (S′1), prepared as in 4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007 into a 100 ml beaker, add 5 ml of sulfuric acid (1+9) and evaporate to dryness. To the dry residue, add 2 ml of sulfuric acid (1+9) and about 15 ml of water. Warm to dissolve as much of the residue as possible. Filter, if necessary, through a filter paper (fine) and wash the residue with warm water. Evaporate to 20 ml, add 2 ml of the ammonium ceric nitrate solution and allow the solution 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.

5 ml of aliquot volume is a typical amount. An aliquot volume of stock solution (S1) or (S′1) should be adjusted according to the mass fraction of chromium(VI) oxide in the sample as shown in Table 5.

**Table 5 — Aliquot volumes of stock solution (S1) or (S′1)**

| **Mass fraction of chromium(VI) oxide** % | **Volumes of stock solution (S1) or (S′1)** ml |
|---|---|
| Less than 0,6 | 10 |
| 0,6 – 1,2 | 5 |
| More than 1,2 | 2 |

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

## 12.3 Blank test

Carry out the procedure given in 12.2 using an aliquot portion of blank solution (B1) or (B′1) obtained in 4.2.2.4 or 4.2.3.4 in ISO 21587-1:2007. The volumes of the aliquot portion of blank solution used should be identical with those for the corresponding stock solution.

## 12.4 Plotting the calibration graph

Transfer 0 (as reference), 1, 2, 3, 4 and 5 ml aliquot portions [0 mg to 0,125 mg as chromium(III) oxide] of dilute standard chromium(III) oxide solution ($Cr_2O_3$ 0,025 mg/ml) into each of several 100 ml volumetric flasks. Treat each of these solutions in accordance with 12.2, but add 5 ml of sulfuric acid instead of 3 ml of sulfuric acid, and measure the absorbance against the reference solution. Plot the relation between the absorbances and mass of chromium(III) oxide. Prepare the calibration graph by adjusting the curve so that it passes through the point of origin.

## 12.5 Calculation

Calculate the chromium(III) oxide mass fraction, $w_{Cr_2O_3}$, as a percentage, using the following equation. Use the amount of chromium(III) oxide which is derived from the absorbance in 12.2 and 12.3 and the calibration in 12.3.

$$w_{Cr_2O_3} = \frac{m_1 - m_2}{m} \frac{500}{V} \times 100 \qquad (12)$$

where

$m_1$ is the mass, in grams, of chromium(III) oxide in the aliquot portion of stock solution (S1) or (S′1);

$m_2$ is the mass, in grams, of chromium(III) oxide in the aliquot portion of blank solution (B1) or (B′1);

$m$ is the mass, in grams, of the test portion in 4.2.2.2 or 4.2.3.2 in ISO 21587-1:2007;

$V$ is the volume, in millilitres, of the aliquot portion taken from stock solution (S1) or (S′1) in 12.2.

# 13 Determination of zirconium oxide by Xylenol orange

## 13.1 Principle

Transfer an aliquot portion of stock solution (S1) or (S′1), add aluminium chloride and hydrazinium dichloride as masking agents for interfering ions, adjust the concentration of hydrochloric acid, add xylenol orange, and measure the absorbance.

## 13.2 Procedure

Transfer 10 ml of stock solution (S1) or (S′1), prepared as in 4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007 to a 100 ml beaker (A) and add 8 ml of aluminium chloride solution.

10 ml of aliquot volume is a typical amount. An aliquot volume of stock solution (S1) or (S′1) should be adjusted according to the mass fraction of zirconium oxide in the sample as shown in Table 6.

**Table 6 — Aliquot volume of stock solution (S1) or (S′1)**

| **Mass fraction of zirconium oxide** % | **Aliquot volume of stock solution (S1) or (S′1)** ml |
|---|---|
| less than 0,20 | 25 |
| 0,20 to 0,50 | 10 |
| more than 0,50 | 5 |

In order to determine the amounts of ammonia and hydrochloric acid required, transfer 10 ml of the stock solution into another beaker (100 ml). Add a drop of methyl orange solution as indicator, and add ammonia solution (1+1) dropwise, until the solution just becomes yellow. Note the volume of ammonia solution used ($X$ ml).

NOTE 1 Calculate the volume, $Y$ ml, of hydrochloric acid (1+1) required using the following equation:

$$Y = 4{,}5 - \frac{7}{X} \qquad (13)$$

Add $Y$ ml of hydrochloric acid (1+1), and 5 ml of hydrazinium dichloride solution to the 100 ml beaker (A). Then dilute to approximately 40 ml with water and stir. To another 100 ml beaker (B), add 4,5 ml of hydrochloric acid (1+1), 8 ml of aluminium chloride solution, and 5 ml of hydrazinium dichloride solution. Dilute to approximately 40 ml with water and stir. Place both the beakers (A) and (B) in a boiling water bath for 15 min. Cool in flowing water.

Transfer the contents of the beakers to 100 ml volumetric flasks (A) and (B) respectively, rinsing the beakers into the flasks. Add precisely 5 ml of xylenol orange solution to each of the flasks, dilute to the mark with water, mix and allow to stand for 10 min. Measure the absorbance of solution (A) in a 10 mm cell at a wavelength of 535 nm, against solution (B).

NOTE 2 If stock solution (S′1) is used, turbidity can appear, caused as a result of using poly(ethylene oxide) as a coagulating agent. In this case, treat as follows. Transfer 10 ml of stock solution (S1) or (S′1) to a beaker (100 ml), add 5 ml of nitric acid and 2 ml of sulfuric acid (1+1) and heat on a sand bath until white fumes appear. After cooling, add approximately 30 ml of water, heat, filter with a filter paper (closed-pore), and wash several times with warm water. Reduce the volume, if necessary, by evaporation and transfer to a 100 ml volumetric flask and complete as above.

### 13.3 Blank test

Carry out the procedure described in 13.2 using blank solution (B1) or (B′1), prepared as in 4.2.2.4 or 4.2.3.4 in ISO 21587-1:2007 corresponding to stock solution (S1) or (S′1). Use the same volume of blank solution (B1) or (B′1) as that of stock solution.

### 13.4 Plotting the calibration graph

Transfer 0, 2, 4, 6, 8 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) into several volumetric flasks (50 ml). To each, add 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. 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.

### 13.5 Calculation

Calculate the mass fraction of zirconium oxide, $w_{ZrO_2}$, as a percentage. Use the following equation with zirconium oxide that is derived from the emission intensity in 13.2. and 13.3, and the calibration in 13.4.

$$w_{ZrO_2} = \frac{m_1 - m_2}{m} \times \frac{500}{V} \times 100 \qquad (14)$$

where

$m_1$ is the mass, in g, of zirconium oxide in the aliquot portion of stock solution (S1) or (S′1);

$m_2$ is the mass, in g, of zirconium oxide in the aliquot portion of blank solution (B1) or (B′1);

$m$ is the mass, in g, of the test portion in 4.2.2.2 or 4.2.3.2 in ISO 21587-1:2007;

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

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

### 14.1 Principle

After adjusting the acid concentration of an aliquot portion of stock solution (S1) or (S′1), ammonium molybdate and L(+)-ascorbic acid are added. The absorbance of the molybdenum blue colour is measured.

### 14.2 Procedure

Carry out the determination using the following procedure.

Pipette 5 ml of stock solution (S1) or (S′1), obtained in 4.2.2.3 or 4.2.3.3 in ISO 21587-1:2007 into a 250 ml beaker.

5 ml of aliquot volume is a typical amount. An aliquot volume of stock solution (S1) or (S′1) should be adjusted according to the mass fraction of phosphorus(V) oxide in the sample as shown in Table 7.

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

| Mass fraction of phosphorus(V) oxide<br>% | Volume of stock solution (S1) or (S′1)<br>ml |
|---|---|
| Less than 0,4 | 25 |
| 0,4 to 1,0 | 10 |
| 1,0 to 2,0 | 5 |
| More than 2,0 | 2 |

Add 2 or 3 drops of *p*-nitrophenol solution as indicator, and add sodium hydroxide solution, dropwise, until the colour changes to yellow. Next, add sulfuric acid (1+1), again dropwise, until colourless, then add an excess of 2 or 3 drops. Then add 5 ml of ammonium molybdate solution (20 g/l) and 2 ml of L(+)-ascorbic acid solution and dilute to about 150 ml with water. Heat for approximately 15 min in a steam bath, then cool with running water. Transfer the solution to a 250 ml volumetric flask, dilute to the mark with water and mix. Measure the absorbance of the solution, against water, in 10 mm cells at a wavelength of 830 nm.

NOTE 1 If stock solution (S′1) is used, turbidity can appear, caused as a result of using poly(ethylene oxide) as a coagulating agent in 4.2.2.4 in ISO 21587-1:2007. In this case, treat as follows. Transfer 5 ml of stock solution (S1) or (S′1) to a 100 ml beaker, add 5 ml of nitric acid and 2 ml of sulfuric acid (1+1), and heat on a sand bath until white fumes appear. After cooling, add approximately 30 ml of water, heat, filter with a filter paper (closed-pore), and wash several times with warm water. Place the filtrate and washings in a beaker (100 ml). Reduce the volume, if necessary, by evaporation and transfer to a 100 ml volumetric flask and complete as above (14.2).

NOTE 2 If this is allowed to evaporate to dryness, the determined phosphorus(V) oxide content can be in error.

### 14.3 Blank test

Using the same volume of blank solution (B1) or (B′1) obtained in 4.2.2.4 or 4.2.3.4 in ISO 21587-1:2007 and carry out the procedure described in 14.2.

### 14.4 Plotting the calibration graph

Transfer 0 (as reference), 5, 10, 15, 20 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) into each of several 100 ml volumetric flasks. Carry out the procedure described in 14.2 on each solution. 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, $w_{\mathrm{P_2O_5}}$, as a percentage, using the following equation from the absorbance obtained in 14.2 and 14.3, and the calibration prepared in 14.4.

$$w_{\mathrm{P_2O_5}} = \frac{m_1 - m_2}{m} \times \frac{500}{V} \times 100 \tag{15}$$

where

$m_1$ is the mass, in g, of phosphorus(V) oxide in the aliquot portion of stock solution (S1) or (S′1);

$m_2$ is the mass, in g, of phosphorus(V) oxide in the aliquot portion of blank solution (B1) or(B′1);

$V$ is the aliquot portion volume, in ml, of stock solution (S1) or (S′1) described in 14.2;

$m$ is the mass, in g, of the test portion in 4.2.2.2 or 4.2.3.2 in ISO 21587-1:2007.

## 15 Test report

Prepare a test report in accordance with ISO 26845.

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