INTERNATIONAL STANDARD

ISO 21814

First edition
2019-02

Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods for chemical analysis of aluminium nitride powders

Céramiques techniques — Méthodes d'analyse chimique des poudres de nitrure d'aluminium

Reference number
ISO 21814:2019(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Methods for chemical analysis of aluminium nitride powders

1 Scope

This document specifies methods for the chemical analysis of fine aluminium nitride powders used as the raw material for fine ceramics.

This document stipulates the determination methods of the aluminium, total nitrogen, boron, calcium, copper, iron, magnesium, manganese, molybdenum, nickel, potassium, silicon, sodium, titanium, tungsten, vanadium, zinc, zirconium, carbon, chlorine, fluorine, and oxygen contents in aluminium nitride powders. The aluminium content is determined by using either an acid pressure decomposition-CyDTA-zinc back titration method or an acid digestion-inductively coupled plasma-optical emission spectrometry (ICP-OES) method. The total nitrogen content is determined by using an acid pressure decomposition-distillation separation-acidimetric titration method, a direct decomposition-distillation separation-acidimetric titration method, or an inert gas fusion-thermal conductivity method. The boron, calcium, copper, iron, magnesium, manganese, molybdenum, nickel, potassium, silicon, sodium, titanium, tungsten, vanadium and zinc contents are determined by using an acid digestion-ICP-OES method or an acid pressure decomposition-ICP-OES method. The sodium and potassium contents are determined via an acid pressure decomposition-flame emission method or an acid pressure decomposition-atomic absorption spectrometry method. The oxygen content is determined by using an inert gas fusion-IR absorption spectrometry method, while that of carbon is determined via a combustion-IR absorption spectrometry method or a combustion-conductometry method. The chlorine and fluorine contents are determined by using a pyrohydrolysation method followed by ion chromatography or spectrophotometry.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 2828, *Aluminium oxide primarily used for the production of aluminium — Determination of fluorine content — Alizarin complexone and lanthanum chloride spectrophotometric method*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

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

ISO 21068-3:2008, *Chemical analysis of silicon-carbide-containing raw materials and refractory products — Part 3: Determination of nitrogen, oxygen and metallic and oxidic constituents*

ISO 21438-2, *Workplace atmospheres — Determination of inorganic acids by ion chromatography — Part 2: Volatile acids, except hydrofluoric acid (hydrochloric acid, hydrobromic acid and nitric acid)*

ISO 21438-3, *Workplace atmospheres — Determination of inorganic acids by ion chromatography — Part 3: Hydrofluoric acid and particulate fluorides*

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Analytes and ranges

a) Aluminium (Al), range of 40 % to 70 % (mass fraction).

b) Total nitrogen (T.N), range of 20 % to 40 % (mass fraction).

c) Boron (B), range of 0,001 % to 0,03 % (mass fraction).

d) Calcium (Ca), range of 0,001 % to 0,03 % (mass fraction).

e) Copper (Cu), range of 0,001 % to 0,03 % (mass fraction).

f) Iron (Fe), range of 0,001 % to 0,03 % (mass fraction).

g) Magnesium (Mg), range of 0,001 % to 0,03 % (mass fraction).

h) Manganese (Mn), range of 0,001 % to 0,03 % (mass fraction).

i) Molybdenum (Mo), range of 0,001 % to 0,03 % (mass fraction).

j) Nickel (Ni), range of 0,001 % to 0,03 % (mass fraction).

k) Potassium (K), range of 0,001 % to 0,03 % (mass fraction).

l) Silicon (Si), range of 0,001 % to 0,03 % (mass fraction).

m) Sodium (Na), range of 0,001 % to 0,03 % (mass fraction).

n) Titanium (Ti), range of 0,001% to 0,03 % (mass fraction).

o) Tungsten (W), range of 0,001 % to 0,03 % (mass fraction).

p) Vanadium (V), range of 0,001 % to 0,03 % (mass fraction).

q) Zinc (Zn), range of 0,001 % to 0,03 % (mass fraction).

r) Carbon (C), range of 0,01 % to 6 % (mass fraction).

s) Chlorine (Cl), range of 0,001 % to 0,5 % (mass fraction).

t) Fluorine (F), range of 0,001 % to 0,2 % (mass fraction).

u) Oxygen (O), range of 0,05 % to 5 % (mass fraction).

5 Preparation of the test sample

5.1 General

The sample preparation method shall be in accordance with ISO 8656-1, unless otherwise mutually agreed upon by the analyser and customer.

5.2 Sampling

The sample shall be collected in accordance with ISO 8656-1.

5.3 Drying

Place 10 g sample into a flat-type weighing bottle (60 mm × 30 mm) and spread it uniformly over the bottom of the bottle. Place the bottle in an air bath at 110 °C ± 5 °C for 2 h, uncovered, and cool in a desiccator (desiccant: magnesium perchlorate), covered, for 1 h.

5.4 Weighing

Weigh the sample to the nearest 0,1 mg of the required quantity using a balance.

6 Reporting the analytical values

6.1 Number of analyses

Analyse the sample twice on different days.

6.2 Blank test

Upon analysis, perform a blank test to correct the measured values.

6.3 Evaluation of the analytical values

When the difference between the two analytical values does not exceed the tolerance value (Table 1), the average value shall be reported. When the difference between the two analytical values exceeds the tolerance value, perform two additional analyses. When the difference of these further two analyses does not exceed the tolerance value, the average value thereof shall be reported. If the difference also exceeds the tolerance value, the median of four analytical values shall be reported.

6.4 Expression of the analytical values

The analytical values shall be presented in % (mass fraction), in dryness.

a) Aluminium, total nitrogen, and oxygen: express the results in four figures to two decimal places.

b) Others: express the results to three decimal places.

Table 1 — Tolerances for the analytical values

Units: % (mass fraction)

Component	Al	Total N	Si, Ti, Fe, Ca, Mg, V, Mo, W, Cu, Ni, Zn, Mn, B, Na, K, F, Cl	O	C
Tolerance	0,20[a] 0,30[b]	0,20[c] 0,40[d]	0,001[e] 0,005[f]	0,05	0,005

[a] Acid pressure decomposition-CyDTA-zinc back titration method.

[b] Acid decomposition-ICP-OES method.

[c] Acid pressure decomposition (or direct decomposition)-distillation separation-acidimetric titration method.

[d] Inert gas fusion-thermal conductivity method.

[e] Applicable to content of less than 0,01 % (mass fraction).

[f] Applicable to content of not less than 0,01 % (mass fraction).

7 Determination of the aluminium content

7.1 Classification of the determination methods

Method A, acid decomposition-ICP-OES method.

Method B, acid pressure decomposition-CyDTA-zinc back titration method.

If analytical results with four significant figures are required, use method B; if two or three significant figures are required, method A can be used.

7.2 Acid decomposition-ICP-OES method

7.2.1 Principle

A portion of the sample is decomposed in water, sulfuric acid, and hydrogen peroxide. After making up to the required volume, the emission intensity of the aluminium present in the test solution is measured by ICP-OES at one or more of the wavelengths: 396,15 nm, 309,28 nm and 394,40 nm.

7.2.2 Reagents

Reagents of analytical grade shall be used. Reagent solutions shall be preserved in plastic bottles.

7.2.2.1 Water, grade 1 or superior as specified in ISO 3696.

7.2.2.2 Sulfuric acid (1+9, 1+180).

7.2.2.3 Hydrochloric acid (1+1, 1+3).

7.2.2.4 Aluminium stock solution (Al 10 mg/ml).

Wash the surface of the aluminium (more than 99,999 % purity by mass fraction) with a hydrochloric acid solution (1+3). Wash the oxidised layer with water, ethanol (99,5 %), and diethyl ether. Dry the aluminium in a desiccator (desiccant: magnesium perchlorate). Weigh 5 g aluminium in a PTFE beaker and cover with a PTFE watch glass. Add 50 ml hydrochloric acid solution (1+1) and heat to dissolve on a steam bath. After cooling, transfer the solution to a 500 ml volumetric flask, dilute with water to the mark and mix well.

NOTE The SI traceable commercial standard solution is also available.

7.2.2.5 Aluminium standard solution (Al 1 mg/ml).

Place 10 ml aluminium stock solution (7.2.2.4) in a 100 ml plastic volumetric flask. Dilute with sulfuric acid (1+180) to the mark and mix well.

7.2.3 Apparatus and instruments

Use ordinary laboratory apparatus and instruments for the chemical analyses in accordance with ISO 26845:2008, Clause 4.

7.2.3.1 ICP-OES.

7.2.4 Procedure

7.2.4.1 Sample decomposition

Weigh 0,30 g test sample and transfer it into a 250 ml beaker. Add 15 ml water, 20 ml sulfuric acid, and 10 ml hydrogen peroxide. Covering the beaker with a watch glass and heat at 280 °C until the test sample has been completely dissolved. After cooling, transfer the solution to a 500 ml volumetric flask, dilute with water to the mark and mix well.

NOTE If the test sample is not completely dissolved, add a little more hydrogen peroxide and continue heating to dissolve the sample. Alternatively, you may apply the acid pressure decomposition method (7.3).

7.2.4.2 Measurement

Transfer a 10 ml aliquot of the stock solution to a 100 ml volumetric flask, dilute with water to the mark and mix well. Spray a portion of the test solution into the flame of the ICP–OES and measure the emission intensity for aluminium at one or more of the wavelengths: 396,15 nm, 309,28 nm and 394,40 nm.

7.2.5 Blank test

Perform the operation described in 7.2.4 without taking a sample to obtain the blank test value.

7.2.6 Drawing of the calibration curve

Transfer 0 ml, 1 ml, 2 ml, 3 ml, 4 ml, and 5 ml aliquots of the aluminium standard solution (7.2.2.5) separately to six 100 ml volumetric flasks. To each flask add 10 ml sulfuric acid (1+10), dilute with water to the mark and mix well.

Spray a portion of each solution into the flame of the ICP–OES and measure the emission intensity for aluminium at one or more of wavelengths: 396,15 nm, 309,28 nm and 394,40 nm. Interferences may be encountered. Carefully choose the optimum wavelength that is free from concomitants.

7.2.7 Calculation

Determine the concentration of the aluminium in the test solution and blank from the calibration curve. Calculate the aluminium content, *Al*, expressed as a percent mass fraction, from Formula (1).

$$Al = \left[\left(A - A_0\right)/m\right] \times 500/10 \times 100 \quad (1)$$

where

Al is the aluminium content, in % (mass fraction);

A is the mass of aluminium in the test solution, in g;

A_0 is the mass of aluminium in the blank solution, in g;

m is the mass of the sample, in g.

7.3 Acid pressure decomposition-CyDTA-zinc back titration method

7.3.1 Principle

A sample is pressure-decomposed with sulfuric acid in a decomposition vessel, to which excess CyDTA solution is added. After adjusting the pH with hexamethylenetetramine, the solution is titrated with a zinc standard solution using xylenol orange as an indicator.

7.3.2 Reagents

Reagents of analytical grade shall be used. The reagent solutions shall be preserved in plastic bottles. Use the reagents described in 7.2.2 together with the following:

7.3.2.1 Hexamethylenetetramine solution (200 g/l).

7.3.2.2 CyDTA solution (0,02 mol/l).

Add 16 ml sodium hydroxide solution (100 g/l) and 300 ml water to 7,3 g cyclohexanediaminetetraacetic acid (hydrate) and heat until dissolved. After cooling, transfer the solution to a 1 000 ml volumetric flask, dilute with water to the mark and mix well.

7.3.2.3 Zinc standard solution (0,02 mol/l).

7.3.2.4 Xylenol orange solution (1 g/l).

7.3.2.5 Sulfuric acid (1+2).

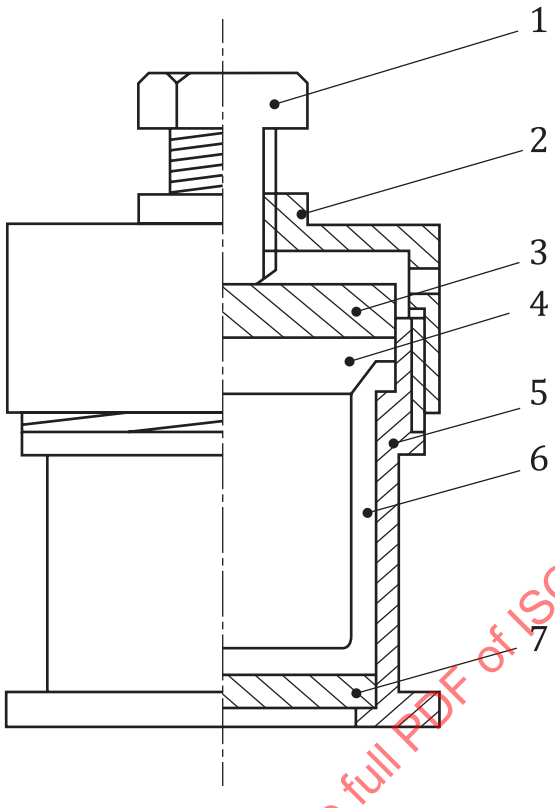
7.3.3 Apparatus and instruments

Use ordinary laboratory apparatus and instruments for the chemical analyses in accordance with ISO 26845:2008, Clause 4.

7.3.3.1 Platinum crucible (20 ml).

7.3.3.2 Pressure decomposition vessel, in general a commercial pressure decomposition vessel may be used.

An example is presented in Figure 1. Use the vessels exclusively for this analysis to avoid cross-contamination.



Key

1 centre screw
2 screw cap
3 top plate
4 PTFE cap
5 cylinder
6 PTFE bottle
7 bottom plate

Figure 1 — Example of a sealed decomposition vessel

7.3.3.3 Air bath, capable of heating at 200 °C ± 5 °C.

7.3.4 Procedure

7.3.4.1 Sample decomposition

Weigh 0,75 g sample in a platinum crucible and add 15 ml sulfuric acid (1+2). Place the crucible into a pressure decomposition vessel and close according to the manufacturer instructions. If an antistatic device is available during weighing, the sample can be directly weighed into the vessel. Place the vessel into an air bath and heat at 200 °C ± 5 °C for 16 h. Acid pressure decomposition under microwave irradiation can also be performed if available.

After cooling, disassemble the vessel. Use plastic tweezers to take out the platinum crucible, and transfer the solution to a 100 ml plastic beaker. Wash the platinum crucible, polytetrafluoroethylene (PTFE) bottle, and plastic tweezers with 5 ml hydrochloric acid (1+1) and warm water. Next, add water to the 100 ml plastic beaker to cool the solution to room temperature. Transfer the solution to a 100 ml volumetric flask, dilute with water to the mark and mix well.

7.3.4.2 CyDTA-zinc back titration

Precisely measure 20 ml sample solution prepared in 7.3.4.1 and place into a 250 ml glass volumetric flask. Dilute with water to the mark and mix well. Pour 50 ml from this solution into a 300 ml glass beaker and add 50 ml of 0,02 mol/l CyDTA solution. Next, add the hexamethylene tetramine solution (200 g/l) to attain pH in the range 5,5 to 5,8 (measured with a pH meter). Add three or four drops xylenol orange solution as an indicator and titrate with the 0,02 mol/l zinc standard solution until the endpoint is reached. The endpoint shall be considered the point at which the solution changes from yellow to red. Record the volume of the titrant at this point.

7.3.5 Blank test

Perform the operation described in 7.3.4 without taking a sample to obtain the blank test value.

7.3.6 Calculation

Calculate the aluminium content in the sample according to Formula (2).

$$Al = \left[\left(V_1 - V_0\right) \times 0{,}000\,539\,6 \times F\right] / \left(m \times 20/100 \times 50/250\right) \times 100 \quad (2)$$

where

Al is the aluminium content in the sample, in % (mass fraction);

V_1 is the integral value of the sample;

V_0 is integral value of the blank test;

F is the factor of the 0,02 mol/l zinc standard solution;

m is the mass of the sample, in g.

8 Determination of the total nitrogen content

8.1 Classification of the determination methods

Method A, acid pressure decomposition-distillation separation-acidimetric titration method.

Method B, direct decomposition-distillation separation-acidimetric titration method.

Method C, inert gas fusion-thermal conductivity method.

If analytical results with four figures are required, use method A or B. If two figures are required, method C can be used.

8.2 Acid pressure decomposition-distillation separation-acidimetric titration method

8.2.1 Principle

The sample is decomposed in a pressure decomposition vessel with sulfuric acid to convert the total nitrogen into ammonia. The solution is transferred to a distillation flask and sodium hydroxide is added. Steam distillation is then performed. The distilled ammonia is mixed with a known amount of amidosulfuric acid and the excess acid is back-titrated with a standardised sodium hydroxide solution.

8.2.2 Reagents

Reagents of analytical grade shall be used. Reagent solutions shall be preserved in plastic bottles.

8.2.2.1 Water, grade 1 or superior as specified in ISO 3696.

8.2.2.2 Sodium hydroxide solution (500 g/l), prepared by dissolving sodium hydroxide in water.

8.2.2.3 Ammonium sulfate, of more than 99,5 % purity (mass fraction).

Ammonium sulfate (more than 99,5 % purity, mass fraction) shall be dried for 3 h at 110 °C ± 5 °C, then cooled down to room temperature in a desiccator (desiccant: magnesium perchlorate).

8.2.2.4 Amidosulfuric acid, of more than 99,0 % purity (mass fraction).

8.2.2.5 0,1 mol/l amidosulfuric acid solution, shall be prepared by weighing 10,0 g amidosulfuric acid and dissolving it in water to make up a volume of 1 000 ml. Calculate the factor of this solution according to Formula (3).

$$F = m \times P / (9{,}7095 \times 100) \tag{3}$$

where

F is the factor of the 0,1 mol/l amidosulfuric acid solution;

m is the mass of amidofulfuric acid, in g;

P is the purity of amidosulfuric acid, in % (mass fraction).

8.2.2.6 0,1 mol/l sodium hydroxide solution, shall be prepared by dissolving sodium hydroxide in water in accordance with ISO 21068-3:2008, 5.2.2.8. Take exactly 50 ml of the 0,1 mol/l amidosulfuric acid solution in a 200 ml beaker and add 50 ml water. Titrate this solution with 0,1 mol/l sodium hydroxide solution. Measure the pH with a pH meter and record the volume of the titrant at the endpoint. The endpoint shall be considered as pH 5,5. Calculate the factor of this solution according to Formula (4).

$$F_1 = F \times 50{,}00 / V \tag{4}$$

where

F_1 is the factor of the 0,1 mol/l sodium hydroxide solution;

F is the factor of the 0,1 mol/l amidosulfuric acid solution;

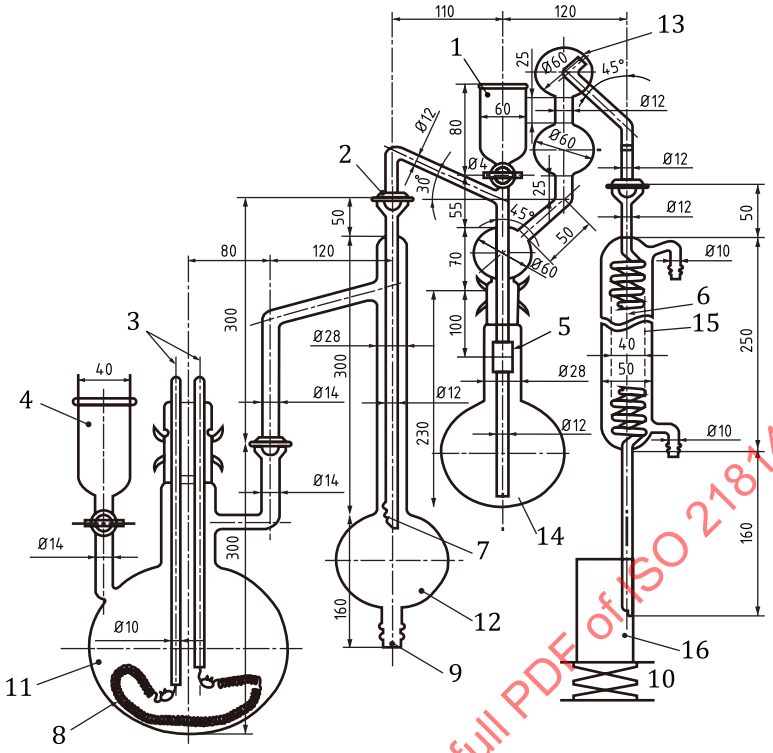
V is the titration volume of the 0,1 mol/l sodium hydroxide solution, in ml.

8.2.3 Apparatus

Use ordinary laboratory apparatus for the chemical analyses together with the following:

8.2.3.1 Steam distillation apparatus, comprises the components listed below. An example of the apparatus is illustrated in Figure 2. Each component shall be made of hard glass coupled by common ground joints and fixed by springs or clamps.

Dimensions in mm



Key

1 funnel
2 ball joint
3 Dumet wire
4 funnel with stopcock
5 rubber tube
6 13 to 15 coils
7 small holes
8 electric heater
9 connection of rubber tube with pinchcock
10 jack
11 flask (2,5 l) for generation of steam
12 trap (500 ml)
13 sphere and tube
14 distillation flask (750 ml)
15 Graham condenser
16 receiver

Figure 2 — Example of the steam distillation apparatus

8.2.3.2 Steam generation flask (2,5 l), equipped with a funnel and stopcock, throw-in heater (1 kW nichrome wire), and steam outlet tube.

8.2.3.3 Trap, the bottom of a bulb shall be connected to a rubber tube with a pinchcock for a drain. The tip of the steam leading-out tube shall have several small holes.

8.2.3.4 Bulb, equipped with, for example, a steam leading-in tube, funnel and stopcock, and splash-proof trap. The steam leading-in tube shall be cut in the middle, enabling the exchange of the tip by connecting to a rubber tube.

8.2.3.5 Distillation flask (750 ml).

8.2.3.6 Coiled condenser.

8.2.3.7 Receiver, a tall 300 ml beaker shall be used.

8.2.3.8 pH meter, readable to the smallest value of 0,1 and equipped with a glass electrode.

8.2.4 Procedure

8.2.4.1 Preparation for the steam distillation

Precisely measure 20 ml of sample solution obtained in 7.3.4.1 and place into a distillation flask. Assemble the steam distillation apparatus and precisely add 50 ml of 0,1 mol/l amidosulfuric acid solution to the receiver. Fix the coiled condenser so that its tip is immersed in the solution. Pour 50 ml sodium hydroxide solution (500 g/l) through the funnel of the distillation flask. By washing the funnel with water, make up the solution volume about 150 ml and then close the funnel stopcock.

8.2.4.2 Steam distillation

Perform the steam distillation by heating the steam generation flask to generate steam. When the distillate in the receiver reaches about 170 ml, lower the receiver to expose the tip of the coiled condenser above the liquid surface and continue the distillation until 200 ml distillate is collected roughly. Wash the exterior of the tip with a small amount of water. The washing water shall be poured into the receiver.

8.2.4.3 Titration

Titrate the distillate with 0,1 mol/l sodium hydroxide solution. Measure the pH with a pH meter and record the volume of the titrant at the endpoint. The endpoint shall be considered as pH 5,5.

8.2.5 Recovery measurement

Weigh 1,210 g ammonium sulfate in a 20 ml platinum crucible. Perform the operation according to 8.2.4 followed by 7.3.4.1. Calculate the recovery from Formula (5). The recovery shall be not less than 99 %.

$$R = \left[\left(50{,}00 \times F\right) - \left(V \times F_1\right)\right] \times 0{,}001\,400\,7 / \left(m \times 0{,}212\,0\right) \times 100 \quad (5)$$

where

R is recovery, in %;

F is the factor of the 0,1 mol/l amidosulfuric acid solution;

V is the titration volume of the 0,1 mol/l sodium hydroxide solution, in ml;

F_1 is the factor of the 0,1 mol/l sodium hydroxide solution;

m is the amount of ammonium sulfate taken into a distillation flask, in g.

8.2.6 Calculation

Calculate the total nitrogen content in the sample according to Formula (6).

$$T.N = \left\{\left[\left(50{,}00 \times F\right) - \left(V \times F_1\right)\right] \times \left(0{,}001\,400\,7 \times 100 / R\right)\right\} / m \times 100 \quad (6)$$

where

$T.N$ is the total nitrogen content in the sample, in % (mass fraction);

F is the factor of the 0,1 mol/l amidosulfuric acid solution;

V is the titration volume of the 0,1 mol/l sodium hydroxide solution, in ml;

F_1 is the factor of the 0,1 mol/l sodium hydroxide solution;

R is the recovery, in %;

m is the amount of the test sample taken into a distillation flask, in g.

8.3 Direct decomposition-distillation separation-acidimetric titration method

8.3.1 Principle

Sodium hydroxide solution is added to the sample and thermal decomposition and steam distillation are performed. The distilled ammonia is then mixed with amidosulfuric acid solution. Finally, the excess acid solution is titrated with a sodium hydroxide solution.

8.3.2 Reagents

Use the reagents described in 8.2.2. Reagents of analytical grade shall be used. Reagent solutions shall be preserved in plastic bottles.

8.3.3 Apparatus

Use the apparatus described in 8.2.3. Use ordinary laboratory apparatus for the chemical analyses.

8.3.4 Procedure

Place 0,15 g sample into a distillation flask and follow the procedure described in 8.2.4.

8.3.5 Recovery measurement

Place 0,240 g ammonium sulfate into a dry distillation flask and follow the procedure described in 8.2.4. Calculate the recovery according to Formula (5). The recovery shall be not less than 99 %.

8.3.6 Calculation

Calculate the total nitrogen content in the test sample according to Formula (6).

8.4 Inert gas fusion-thermal conductivity method

8.4.1 Principle

A sample is fused with a flux in a graphite crucible under inert gas flow to extract nitrogen and other gases from it. The elemental nitrogen content is determined using a thermal conductivity detector after the removal of concomitants such as carbon monoxide, carbon dioxide, other gases, and moisture.

8.4.2 Reagents

The reagents can vary substantially for different instruments; however, in all cases, the reagents specified by the instrument manufacturer shall be used. The reagents shall be the following:

8.4.2.1 Helium, of more than 99,99 % purity (volume fraction).

8.4.2.2 Flux, in shot or basket form made of tin or nickel.Use a combination of metals different from that of the capsule.

8.4.2.3 Boron nitride-certified reference materials (CRM), shall be heated at 1 000 °C for 2 h and subsequently cooled in a desiccator (desiccant: magnesium perchlorate) before use.

NOTE Silicon CRM can also be used.

8.4.3 Apparatus

8.4.3.1 Capsule, made of nickel or tin, designated for each apparatus.

8.4.3.2 Graphite crucible, suitable for use in an impulse furnace. Examples are presented in Figure 3.

Dimensions in mm

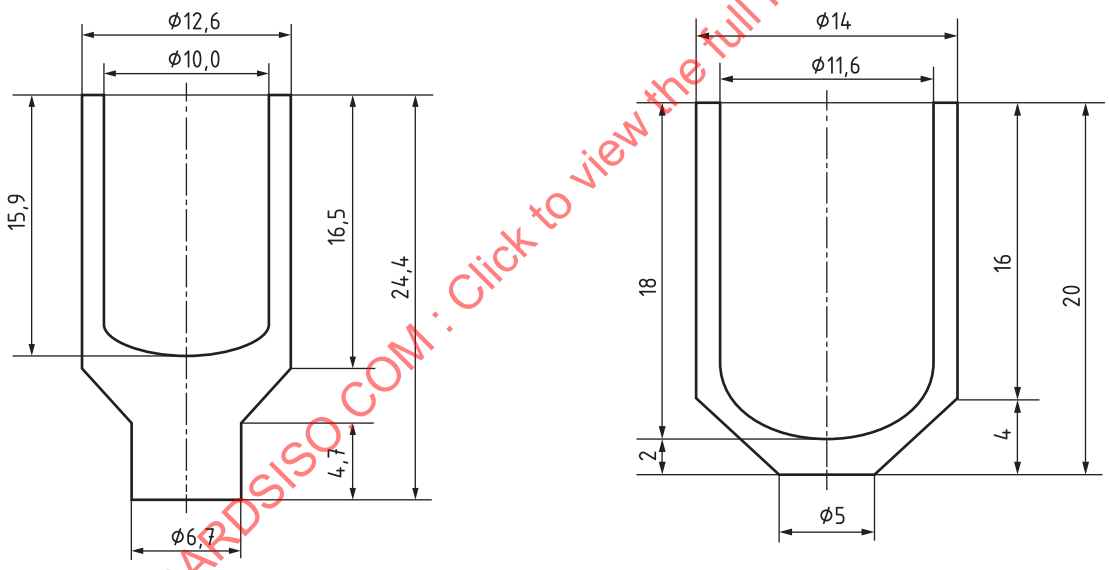
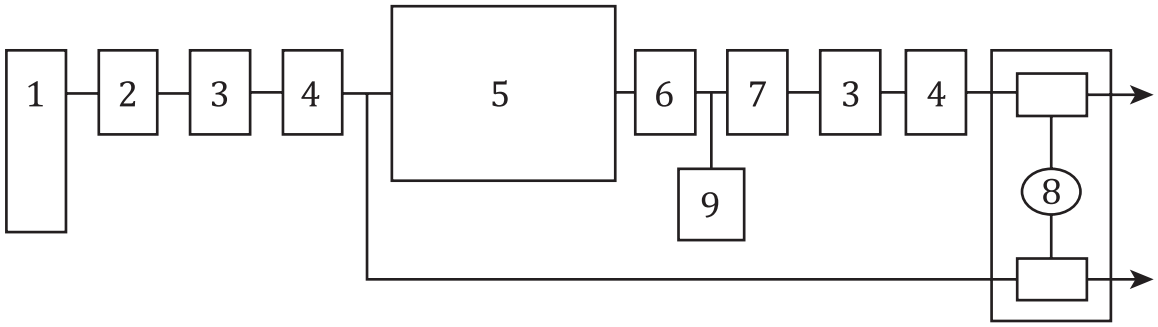


Figure 3 — Examples of a graphite crucible

8.4.4 Instrument

A commercial nitrogen analyser may be used for the determination of nitrogen. It usually comprises an inert gas refiner, gas extractor, extracted gas refiner, and nitrogen detector. Figure 4 is a block diagram of such an instrument.



Key

1 helium supplier
2 oxygen trap with electric heater
3 carbon dioxide trap
4 dehydration tube
5 impulse furnace
6 dust collector
7 oxidation tube with electric heater
8 thermal conductivity detector
9 IR spectrometer for oxygen determination (optional)

NOTE Each instrument has its own unique design characteristics and operational requirements.

Figure 4 — Block diagram of the inert gas fusion-thermal conductivity method

8.4.4.1 Inert gas refiner, comprises, for example, a deoxidation tube (reduced copper) with an electric furnace, a carbon dioxide absorption tube (sodium hydroxide shots), and a dehydration tube (magnesium perchlorate). A denitrification tube (sponge titanium) is attached in some instruments.

8.4.4.2 Gas extractor, comprising, for example, a sample feeder and impulse furnace. The sample feeder can place the sample-embedded capsule into the graphite crucible in the impulse furnace under inert gas flow. The impulse furnace shall be capable of attaining about 3 000 °C. The upper water-cooled copper electrode is fixed, whereas the lower electrode moves vertically. The graphite crucible is sandwiched between both electrodes.

8.4.4.3 Extracted gas refiner, consisting of, for example, a dust collecting tube (glass wool), carbon dioxide absorption tube (sodium hydroxide shots), oxidation tube with an electric furnace, and a dehydration tube (magnesium perchlorate).

8.4.4.4 Gas detector, consisting of a thermal conductivity detector and an integration meter.

NOTE Other commercial instruments for nitrogen analysis are available. Each has its own unique design characteristics and operational requirements. Operation details can be found in the instrument manufacturer's instruction manual.

8.4.5 Procedure

8.4.5.1 Starting up of the instrument

Switch on the instrument and set the controls to the specified values in accordance with the manufacturer instructions for operation. Wait until it becomes stable.

8.4.5.2 Preliminary heating

Set a new graphite crucible to the specified position of the impulse furnace. Flow the inert gas and then turn on the furnace. Heat the graphite crucible to the degassing temperature for the specified period and subsequently, to the gas extraction temperature. The temperature adjustment of the graphite crucible is achieved by adjusting the electrical current, voltage or power. The relationship between the temperature and current/voltage must be determined before the equipment is used.

Read the integration meter (hereafter referred to as "integral value"). Repeat degassing and gas extraction steps until a stable integral value is obtained.

8.4.5.3 Degassing of the graphite crucible

Measure the specified amount of bath metal into a new graphite crucible and place it at the specified position of the impulse furnace. Weigh 0,02 g to 0,04 g sample in a capsule and enclose it using a jig.

Place the capsule at the specified position of the sample blower. Feed the inert gas and energize the graphite crucible. Heat it at the degassing temperature for the specified period and degas the graphite crucible and bath metal. The degassing temperature shall be 100 °C higher than the gas extraction temperature.

8.4.5.4 Measuring

Place the sample-embedded capsule into the graphite crucible. Energise the crucible, and heat the sample at the gas extraction temperature for the specified period. Read the integral value. The optimum gas extracting temperature shall be determined beforehand by using a CRM of known nitrogen content (8.4.2.3).

NOTE Because the commercial instruments for analysing nitrogen might be fully automated, many of the steps described above can be carried out without the need of any operator intervention.

8.4.6 Blank test

Perform the operation described in 8.4.5 without taking a sample to obtain the blank test value. Repeat this operation three to five times and calculate the average value.

8.4.7 Calculation of the calibration coefficient

A nitrogen-CRM of known content shall be used. Some boron nitride powder (or silicon nitride powder) reference materials are also available. Perform the operation described in 8.4.5, using the sample for calibration. Average the values from three to five measurements and calculate the calibration coefficient according to Formula (7).

$$K = \left(m \times T.N / 100\right) / \left(A_1 - A_0\right) \tag{7}$$

where

K is the calibration coefficient, in g/integral value;

m is the mass of the sample for calibration, in g;

$T.N$ is the total nitrogen content in the sample for calibration, in % (mass fraction);

A_1 is the integral value of the sample for calibration;

A_0 is the integral value of the blank test.

8.4.8 Calculation

Calculate the total nitrogen content in the sample according to Formula (8).

$$T.N = \left(A_2 - A_0\right) \times K / m \times 100 \quad (8)$$

where

$T.N$ is the total nitrogen content in the sample, in % (mass fraction);

A_2 is the integral value of the sample;

A_0 is the integral value of the blank test;

K is the calibration coefficient, in g/integral value;

m is the mass of the sample, in g.

NOTE Some commercial instruments can calculate the blank test value. In this case, the calibration coefficient and the total nitrogen content are calculated automatically.

9 Determination of the sodium and potassium contents

9.1 Classification of the determination methods

Method A, acid pressure decomposition-flame emission method.

Method B, acid pressure decomposition-atomic absorption method.

NOTE The sodium and potassium contents can be simultaneously determined with those of the other trace elements by using the methods described in Clause 10.

9.2 Acid pressure decomposition-flame emission method

9.2.1 Principle

The sodium and potassium contents are measured by flame emission spectrometry, using the sample solution obtained in 7.3.4.1.

9.2.2 Reagents

Reagents of analytical grade shall be used. Reagent solutions shall be preserved in plastic bottles. Use the reagents described in 7.2.2 as well as the following:

9.2.2.1 Ammonia solution (1+1).

9.2.2.2 Cobalt solution (0,5 g/100 ml).

Weigh 0,500 g cobalt (more than 99,9 % purity by mass fraction) in a quartz beaker and cover with a quartz watch glass. Add 50 ml sulfuric acid (1+9) and heat to dissolve on a heater. After cooling, transfer the solution to a 100 ml plastic volumetric flask, dilute with water to the mark and mix well.

9.2.2.3 Aluminium solution (4,93 g/500 ml) for matrix compensation

Wash the surface of the aluminium (more than 99,999 % purity by mass fraction) with hydrochloric acid solution (1+3). Wash the oxidised layer with water, ethanol (99,5 %), and diethyl ether. Dry the aluminium in a desiccator (desiccant: magnesium perchlorate). Weigh 4,93 g aluminium in a PTFE

beaker and cover with a PTFE watch glass. Add 100 ml sulfuric acid solution (1+1), 50 ml water, 50 ml hydrochloric acid, and 6,0 ml cobalt solution. Heat to dissolve on a steam bath. After cooling, transfer the solution to a 500 ml volumetric flask, dilute with water to the mark and mix well.

9.2.2.4 Sodium standard solution (Na 1 mg/ml).

9.2.2.5 Potassium standard solution (K 1 mg/ml).

9.2.2.6 Mixed standard solution (Na, K: 50 mg/l), place 5 ml each of sodium (9.2.2.4) and potassium standard solutions (9.2.2.5) in a 100 ml plastic volumetric flask. Dilute with sulfuric acid (1+180) to the mark and mix it well. Attention shall be paid to ensure that no precipitation occurs during the mixing. This standard solution shall be freshly prepared before use.

9.2.2.7 Wavelength adjustment solution (Na, K: 5 mg/l), place 10 ml mixed standard solution (9.2.2.6) in a 100 ml plastic volumetric flask. Dilute with sulfuric acid (1+180) to the mark and mix well.

9.2.3 Instruments

9.2.3.1 Flame emission spectrometer.

9.2.4 Procedure

9.2.4.1 Adjustment of the flame photometer

Spray a wavelength adjustment solution (9.2.2.7) in the flame, and adjust it to its highest emission intensity [589,0 nm (Na) or 766,5 nm (K)]. Subsequently, spray water to adjust the indication to zero.

9.2.4.2 Measurement of the flame emission intensity

Spray the solution prepared from 7.3.4.1 into the flame and measure the emission intensity of sodium at 589,0 nm or potassium at 766,5 nm.

9.2.5 Blank test

Perform the operation described in 9.2.4.2 using the blank test solution obtained in 7.3.5. Record the results.

9.2.6 Drawing of the calibration curve

Pour 50 ml aluminium solution (9.2.2.3) and 2,6 ml ammonia solution (1+1) separately into five 100 ml plastic volumetric flasks. Add 0 ml, 1 ml, 2 ml, 3 ml, 4 ml, and 5 ml of the mixed standard solution (9.2.2.6) stepwise and precisely. Dilute with sulfuric acid (1+180) to the mark and mix each solution well. These shall be designated as calibration curve solutions. Repeat the operations described in 9.2.4.2, using the calibration curve solutions. Translate a correlation curve of the emission intensity and amount of sodium added and construct a calibration curve that runs through the origin point.

9.2.7 Calculation

Determine the amount of sodium from the emission intensity measured in 9.2.4.2 and the calibration curve obtained in 9.2.6. Calculate the percentage content of sodium or potassium in the sample according to Formula (9).

$$W_i = \left(m_i - m_0\right) / m \times 100 \quad (9)$$

where

W_i is the each element content, in %(mass fraction);

m_i is the mass of each element in the test solution, in g;

m_0 is the mass of each element in the blank solution, in g;

m is the mass of the test portion, in g.

9.3 Acid pressure decomposition-atomic absorption spectrometry method

9.3.1 Principle

Sodium and potassium are measured by atomic absorption spectrometry using the sample solution obtained in 7.3.4.1.

9.3.2 Reagents

Reagents of analytical grade shall be used. Reagent solutions shall be preserved in plastic bottles.

9.3.3 Instruments

9.3.3.1 Atomic absorption spectrometry.

9.3.4 Procedure

To measure the absorbance of sodium or potassium, use a hollow cathode lamp designed for sodium or potassium analysis to spray the sample solution obtained in 7.3.4.1.

9.3.5 Blank test

Perform the operation described in 9.3.4 using the blank test solution obtained in 7.3.5. Record the results.

9.3.6 Drawing of the calibration curve

Use the calibration curve solutions obtained in 9.2.6 to measure the atomic absorption in 9.3.4. Prepare and translate a correlation curve of the absorbance and amount of sodium or potassium added to create a calibration curve that runs through the origin point.

9.3.7 Calculation

Record the amount of sodium or potassium from the absorption intensity measured in 9.3.4 and the calibration curve obtained in 9.3.6. Calculate the percentage content of sodium or potassium in the sample according to Formula (9).

10 Determination of the trace element contents

10.1 Classification of the determination methods

Method A, acid decomposition-ICP-OES method.

Method B, acid pressure decomposition-ICP-OES method.

10.2 Acid decomposition-ICP-OES method

10.2.1 Principle

To prepare the test solution, the sample is decomposed in sulfuric acid and hydrogen peroxide. Boron, calcium, copper, iron, magnesium, manganese, molybdenum, nickel, potassium, silicon, sodium, titanium, tungsten, vanadium, and zinc are determined by ICP-OES at the selected wavelength.

10.2.2 Reagents

Use the reagents described in 7.2.2 as well as the following:

10.2.2.1 Aluminium solution (4,93 g/500 ml), according to 9.2.2.3.

10.2.2.2 Ammonia solution (1+1).

10.2.2.3 Elemental standard solutions.

— Boron standard solution (B 1 mg/ml).

— Calcium standard solution (Ca 1 mg/ml).

— Copper standard solution (Cu 1 mg/ml).

— Iron standard solution (Fe 1 mg/ml).

— Magnesium standard solution (Mg 1 mg/ml).

— Manganese standard solution (Mn 1 mg/ml).

— Molybdenum standard solution (Mo 1 mg/ml).

— Nickel standard solution (Ni 1 mg/ml).

— Potassium standard solution (K 1 mg/ml).

— Silicon standard solution (Si 1 mg/ml).

— Sodium standard solution (Na 1 mg/ml).

— Titanium standard solution (Ti 1 mg/ml).

— Tungsten standard solution (W 1 mg/ml).

— Vanadium standard solution (V 1 mg/ml).

— Zinc standard solution (Zn 1 mg/ml).

NOTE The SI traceable commercial standard solutions are available.

10.2.2.4 Mixed standard solution (each element 50 mg/l).

Place 5 ml each of the solutions prepared from 10.2.2.3 in a 100 ml plastic volumetric flask. Dilute with sulfuric acid (1+180) to the mark and mix well. Attention shall be paid to ensure that no precipitation occurs during mixing. The solution shall be freshly prepared before use.

10.2.3 Apparatus and instruments

Use the apparatus and instruments described in 7.2.3.

10.2.4 Procedure

10.2.4.1 Sample decomposition

Weigh 0,30 g test sample and transfer it into a 250 ml beaker. Add 15 ml water, 20 ml sulfuric acid, and 10 ml hydrogen peroxide. Covering the beaker with a watch glass and heat at 280 °C until the test sample has been completely dissolved. After cooling, transfer the solution to a 100 ml volumetric flask, dilute with water to the mark and mix well. This solution is designated as the test solution.

10.2.4.2 Measurement

Spray a portion of the test solution into the flame of the ICP–OES and measure the emission intensity at an appropriate wavelength (Table 2). Interferences may be encountered. Thus, carefully choose the optimum wavelength that is free from the concomitants.

Table 2 — Examples of the analytical wavelength for each element[a]

Element	Wavelength 1 nm	Wavelength 2 nm
B	249,77	—
Ca	317,93	393,37
Cu	327,40	324,75
Fe	238,20	259,94
Mg	285,21	279,55
Mn	257,61	259,37
Mo	202,03	—
Ni	231,60	221,65
K	766,49	—
Si	251,61	212,41
Na	589,69	588,95
Ti	334,94	336,12
W	207,91	—
V	290,88	292,40
Zn	213,856	206,200

[a] Considering the spectral interferences and the sensitivities, choose the higher-order spectral lines if available.

10.2.5 Blank test

Perform the operation described in 10.2.4 without taking a sample to obtain the blank test value.

10.2.6 Drawing of the calibration curve

Pour 50 ml aluminium solution (10.2.2.1) and 2,6 ml ammonia solution (1+1) separately into five 100 ml plastic volumetric flasks. Add 0 ml, 1 ml, 2 ml, 3 ml, 4 ml, and 5 ml of the mixed standard solution (10.2.2.4) stepwise and precisely. Dilute each solution with sulfuric acid (1+180) to the mark and mix well. Spray a portion of each solution into the flame of ICP–OES and measure the emission intensity at an appropriate wavelength.

10.2.7 Calculation

Determine the concentration of each element in the test solution and in the blank from the calibration curve. Calculate the element content from Formula (9).

NOTE The analytical results obtained from a round robin test are listed in Annex A.

10.3 Acid pressure decomposition-ICP-OES method

10.3.1 General

Boron, calcium, copper, iron, magnesium, manganese, molybdenum, nickel, potassium, silicon, sodium, titanium, tungsten, vanadium, zinc and zirconium are measured by the ICP-OES method, using the sample solution obtained in 7.3.4.1.

10.3.2 Reagents

Use the reagents described in 10.2.2. Reagents of analytical grade shall be used. Reagent solutions shall be preserved in plastic bottles.

10.3.3 Apparatus and instruments

Use the apparatus and instruments described in 7.3.3.

10.3.4 Procedure

Using the sample solution obtained in 7.3.4.1, measure the emission intensity of the trace elements following the procedure described in 10.2.4.2.

10.3.5 Blank test

Perform the procedure described in 10.3.4 using the blank test solution obtained in 7.3.5. Record the results.

10.3.6 Drawing of the calibration curve

Perform the operation in accordance with 10.2.6.

10.3.7 Calculation

Determine the concentration of each element in the test solution and in the blank from the calibration curve. Calculate the element content from Formula (9).

11 Determination of the oxygen content

11.1 Principle

A sample is fused with flux in a graphite crucible under inert gas flow. The oxygen generated from the sample reacts with the carbon of the graphite crucible to form carbon monoxide. After removing concomitants such as moisture and dust, the carbon monoxide content is determined after conversion into carbon dioxide or directly using IR spectrometry.

11.2 Reagents

Use the reagents described in 8.4.2 as well as the following:

11.2.1 Helium, of more than 99,99 % purity (volume fraction).

11.2.2 Yttrium oxide, of more than 99,99 % purity (mass fraction); heated at 1 000 °C for 2 h and cooled in a desiccator (desiccant: magnesium perchlorate) before use.

11.2.3 Magnesium perchlorate, reagent grade, with the particle size range 0,7 mm to 1,2 mm for the absorption of moisture. Use the purity specified by the instrument manufacturer.

11.3 Apparatus

Use the apparatus described in 8.4.3.

11.4 Instruments

Use the instruments described in 8.4.4, except for the components of the gas separator and the detector. The extracted gas purifier shall not include a carbon dioxide absorption tube (sodium hydroxide shots) or a dehydration tube (magnesium perchlorate). An IR spectrometer instead of a thermal conductivity detector shall be used. A commercial oxygen analyser is available.

11.5 Procedure

Measure 0,02 g to 0,04 g sample and follow the procedure described in 8.4.5.

11.6 Blank test

Perform the operation described in 11.5 without taking a sample to obtain the blank test value. Repeat this operation three to five times and calculate the average value.

11.7 Calculation of the calibration coefficient

Use 0,010 g yttrium oxide or 0,030 g nitride of known oxygen content for the calibration. Some silicon nitride powder (or boron nitride powder) reference material are also available. Average the values from three to five measurements and calculate the calibration coefficient according to Formula (10).

$$K = \left(G \times O / 100\right) / \left(A_1 - A_0\right) \tag{10}$$

where

K is the calibration coefficient, in g/integral value;

G is the mass of the sample for calibration, in g;

O is the oxygen content of the sample for calibration, in % (mass fraction);

A_1 is the integral value of the sample for calibration;

A_0 is the integral value of the blank test.

11.8 Calculation

Calculate the oxygen content in the sample according to Formula (11).

$$O = \left[\left(A_2 - A_0\right) \times K / m\right] \times 100 \tag{11}$$

where

O is the oxygen content, in % (mass fraction);

A_2 is the Integral value of the sample;

A_0 is the integral value of the blank test;

K is the calibration coefficient, in g/integral value;

m is the mass of the sample, in g;

NOTE Some commercial instruments can calculate the blank test value, the calibration coefficient and the oxygen content automatically.

12 Determination of the carbon content

12.1 Classification of the determination methods

Carbon shall be determined using any of the following methods:

Method A, combustion (resistance furnace)-IR absorption spectrometry.

Method B, combustion (radio frequency heating furnace)-thermal conductometry.

Method C, combustion (radio frequency heating furnace)-IR absorption spectrometry.

12.2 Combustion (resistance furnace)-IR absorption spectrometry

12.2.1 Principle

A sample is combusted in an oxygen flow with combustion accelerators in a resistance furnace. The generated carbon oxides are determined using an IR spectrometer, and the integrated values are converted to a value corresponding to the carbon contnet.

12.2.2 Reagents

12.2.2.1 Oxygen, of more than 99,99 % purity (volume fraction).

12.2.2.2 Combustion accelerators, tin, in sandy form.

Combustion accelerators (other than tin) or a combination of metals (such as tin and iron) may be used, provided that they satisfy the criteria for combustion accelerators.

12.2.2.3 Calcium carbonate, of more than 99,9 % purity (mass fraction), heated at 500 °C to 550 °C for 2 h and cooled in a desiccator (desiccant: magnesium perchlorate).

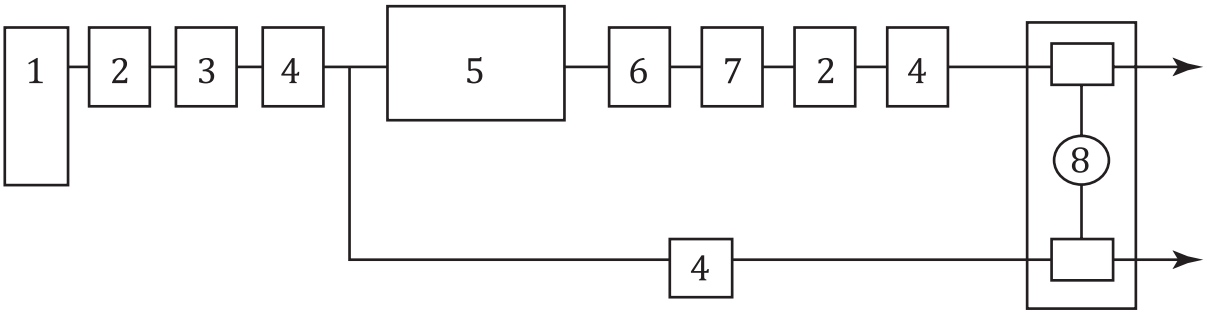
12.2.3 Apparatus

12.2.3.1 Combustion tube, use the tube specified by the apparatus manufacturer.

12.2.3.2 Combustion boat, use the boat specified by the apparatus manufacturer. Perform a preliminary combustion at 1 350 °C before use.

12.2.4 Instrument

A commercial carbon analyser may be used for the determination of the carbon content. It usually comprises an oxygen refiner, furnace, combustion gas refiner, and carbon detector. Figure 5 illustrates a block diagram of such an instrument.



Key

1 oxygen supplier
2 oxidation tube with electric furnace
3 carbon dioxide trap
4 dehydration tube
5 combustion tube
6 dust collector
7 sulfur dioxide trap
8 IR spectrometer

NOTE Each instrument has its own unique design characteristics and operational requirements.

Figure 5 — Block diagram of a carbon analyser for combustion (resistance furnace)-IR absorption spectrometry

12.2.4.1 Oxygen refiner, comprising, for example, an oxidation tube (copper oxide) with an electric furnace, a carbon dioxide absorption tube (sodium hydroxide shots), and dehydration tube (magnesium perchlorate).

12.2.4.2 Furnace for combusting the sample, comprising, for example, a tubular electric resistance furnace and combustion tube. The tubular electric resistance furnace shall be able to regulate the temperature of the central section (100 mm or longer) of the combustion tube to 1 350 °C.

12.2.4.3 Combustion gas refiner, comprising, for example, a dust collecting tube (glass wool), sulfur dioxide trap (manganese dioxide), oxidation tube (copper oxide or platinum-based catalyst) with an electric furnace, and dehydration tube (magnesium perchlorate).

12.2.4.4 Carbon detector, comprising an IR spectrometer for carbon dioxide detection and other components. The IR spectrometer detects the differential between the intensities (carbon dioxide) of the IR absorption of a sample cell and that of the reference cell as electric signals using a detector. It then converts this value to the carbon content via linearisation and integration circuits. The result is displayed on an integration meter. Another type of instrument measures the IR absorption of carbon dioxide and carbon monoxide separately and displays the sum of both measurements as the carbon content.

NOTE Some commercial IR spectrometers measure carbon dioxide without a reference cell.

12.2.5 Procedure

12.2.5.1 Stabilisation of the instrument

Switch on the instrument and set each of the components to the designated conditions. Wait for the components to stabilise.

12.2.5.2 Mixture of the sample and combustion accelerators

Weigh 0,3 g to 0,5 g sample in the combustion boat and spread it evenly. Next, evenly cover the sample with 2 g combustion accelerators. Alternatively, mix the sample with tin and spread evenly or spread the sample so that it is sandwiched between 1 g of combustion accelerator on each side.

12.2.5.3 Sample combustion

Remove the plug of the combustion tube, insert the boat loaded with the sample and combustion accelerators into the centre of the combustion tube and immediately attach the plug. Switch on the oxygen flow and operate the furnace for the designated duration.

12.2.5.4 Measurement

Propel the generated carbon dioxides, along with the oxygen, through the combustion gas refiner and into the IR spectrometer. After the designated time has elapsed, measure the integrated values.

12.2.6 Blank test

Perform the operation described in 12.2.5 without taking a sample to obtain the blank test value.

12.2.7 Calculation of the calibration coefficient

Use 0,250 g calcium carbonate or 0,500 g nitride of known carbon content (calibration sample) and follow the procedure described in 12.2.5. Calculate the calibration coefficient according to Formula (12) or Formula (13), respectively.

a) When calcium carbonate is used, refer to Formula (12).

$$K = \left(G \times 0{,}120\ 0\right) / \left(A_1 - A_0\right) \tag{12}$$

where

K is the calibration coefficient, in g/integral value;

G is the mass of calcium carbonate, in g;

A_1 is the integral value of the sample for calibration;

A_0 is the integral value of the blank test.

b) When the nitride sample is used, refer to Formula (13).

$$K = \left(G \times C / 100\right) / \left(A_1 - A_0\right) \tag{13}$$

where

K is the calibration coefficient, in g/integral value;

G is the mass of the sample for calibration, in g;

C is the carbon content in the sample for calibration, in % (mass fraction);

A_1 is the integral value of the sample for calibration;

A_0 is the integral value of the blank test.

12.2.8 Calculation

Calculate the carbon content in the sample according to Formula (14).

$$C = \left[\left(A_2 - A_0\right) \times K / m\right] \times 100 \tag{14}$$

where

C is the carbon content, in % (mass fraction);

A_2 is the integral value of the sample;

A_0 is the integral value of the blank test;

K is the calibration coefficient, in g/integral value;

m is the mass of the sample, in g.

12.3 Combustion (radio frequency heating furnace)-thermal conductometry

12.3.1 Principle

A sample is combusted under an oxygen flow with combustion accelerators in a radio frequency heating furnace. The generated carbon dioxide is determined using a thermal conductometer and the integrated value is converted to a value corresponding to the carbon content.

12.3.2 Reagents

Use the reagents described in 12.2.2 except for the combustion accelerator. For combustion accelerators, use granular copper, tungsten, chipped iron, or any other substances recommended by the instrument manufacturer.

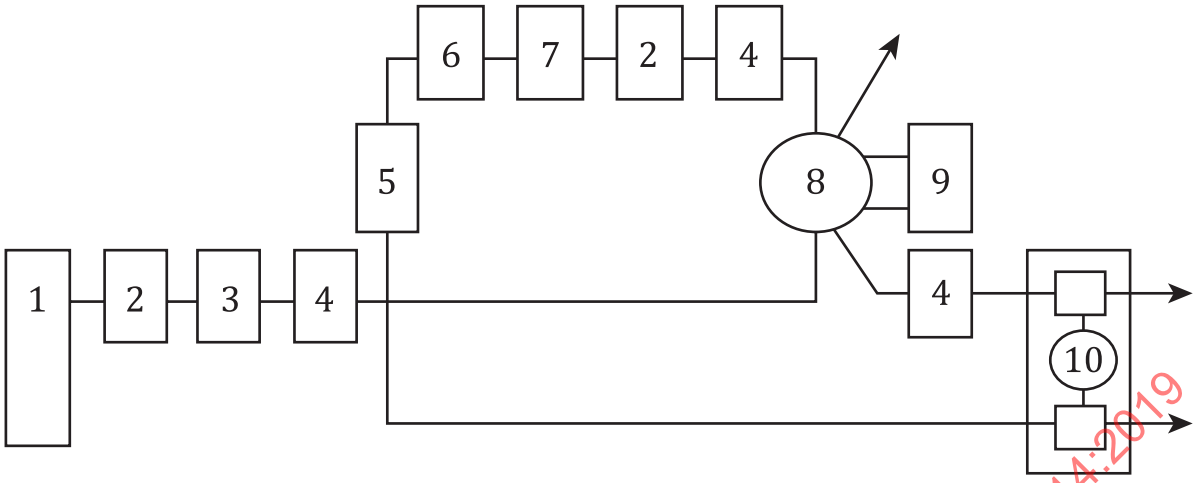
12.3.3 Apparatus

12.3.3.1 Combustion crucible, the type specified by the apparatus manufacturer.

12.3.3.2 Receptacle, the type specified by the apparatus manufacturer.

12.3.4 Instrument

A commercial carbon analyser may be used for the determination of carbon. It usually comprises an oxygen refiner, furnace, combustion gas refiner, and carbon detector. Figure 6 presents a block diagram of such an instrument.



Key

1 oxygen supplier
2 oxidation tube with electric furnace
3 carbon dioxide trap
4 dehydration tube
5 combustion tube
6 dust collector
7 sulfur dioxide trap
8 duct converter
9 carbon dioxide collector
10 thermal conductometer

NOTE Each instrument has its own unique design characteristics and operational requirements.

Figure 6 — Block diagram of carbon analyser for a combustion (radio frequency heating furnace)-thermal conductometry

12.3.4.1 Oxygen refiner, according to 12.2.4.1.

12.3.4.2 Furnace for combusting the sample, comprising, for example, a radio frequency heating furnace and oscillator.

12.3.4.3 Combustion gas refiner, according to 12.2.4.3.

12.3.4.4 Carbon detector, comprising, for example, a flow converter, a carbon dioxide collection tube (synthetic zeolite), and thermal conductometer.

12.3.5 Procedure

12.3.5.1 Stabilisation of the instrument

Switch on the instrument and set each of the components to the designated conditions. Wait for the components to stabilise.

12.3.5.2 Mixture of the sample and combustion accelerators

Weigh a sample in the combustion crucible and add 1 g each of copper and iron or copper and tungsten over it.

12.3.5.3 Sample combustion

Place the crucible inside the furnace and close the furnace. Supply oxygen at the designated pressure and flow rate. Operate the furnace for the designated duration.

12.3.5.4 Measurement

Propel the combustion gas to the collection tube for absorption. Change the oxygen flow rate and heat up the collection tube for the designated duration to release the carbon dioxide, along with theoxygen, to the thermal conductometer. Measure the integrated values.

NOTE Some commercial instruments perform the operations described in 12.3.5.3 and 12.3.5.4 automatically.

12.3.6 Blank test

Perform the operation described in 12.3.5 without taking a sample to obtain the blank test value.

12.3.7 Calculation of the calibration coefficient

Use 0,250 g calcium carbonate or 0,500 g nitride of known carbon content (calibration sample) and follow the procedure described in 12.3.5. Calculate the calibration coefficient according to Formula (12) or Formula (13), respectively.

12.3.8 Calculation

Calculate the carbon content in the sample according to Formula (14).

12.4 Combustion (radio frequency heating furnace)-IR absorption spectrometry

12.4.1 Principle

A sample is combusted under an oxygen flow with combustion accelerators in a radio frequency heating furnace. The amount of generated carbon oxides is determined using IR spectrometry and the integrated values are converted to a value corresponding to the carbon content.

12.4.2 Reagents

Use the reagents described in 12.3.2.

12.4.3 Apparatus

Use the apparatus described in 12.3.3.

12.4.4 Instrument

A commercial carbon analyser may be used for the determination of the carbon content. This usually comprises an oxygen refiner, furnace, combustion gas refiner, and carbon detector. Figure 7 presents a block diagram of such an instrument.