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**Aluminium and aluminium alloys — Chemical analysis — Inductively coupled plasma optical emission spectrometric analysis**

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## National foreword

This British Standard is the UK implementation of EN 14242:2023. It supersedes BS EN 14242:2004, which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee NFE/35, Light metals and their alloys.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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English Version

# Aluminium and aluminium alloys - Chemical analysis - Inductively coupled plasma optical emission spectrometric analysis

Aluminium et alliages d'aluminium - Analyse chimique  
- Analyse par spectrométrie d'émission optique avec  
source à plasma induit par haute fréquence

Aluminium und Aluminiumlegierungen - Chemische  
Analyse - Optische Emissionsspektrometrie mit  
induktiv gekoppeltem Plasma

This European Standard was approved by CEN on 23 January 2023.

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## European foreword

This document (EN 14242:2023) has been prepared by Technical Committee CEN/TC 132 “Aluminium and aluminium alloys”, the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2023, and conflicting national standards shall be withdrawn at the latest by September 2023.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 14242:2004.

The main changes compared to the previous edition are listed below:

- modification of the title and Scope;
- new subclause 5.15.6;
- several editorial modifications.

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Türkiye and the United Kingdom.

## 1 Scope

This document specifies an inductively coupled plasma optical emission spectrometric method (ICP-OES) for the analysis of aluminium and aluminium alloys.

This method is applicable to the determination of silicon, iron, copper, manganese, magnesium, chromium, nickel, zinc, titanium, gallium, vanadium, beryllium, bismuth, calcium, cerium, cobalt, lithium, sodium, lead, antimony, tin, strontium and zirconium in aluminium and aluminium alloys.

The content of the elements to be determined should be at least 10 times higher than the corresponding detection limits.

## 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.

EN 12258-2:2004, *Aluminium and aluminium alloys - Terms and definitions - Part 2: Chemical analysis*

EN 14361, *Aluminium and aluminium alloys - Chemical analysis - Sampling from metal melts*

EN ISO 648, *Laboratory glassware - Single-volume pipettes (ISO 648)*

EN ISO 1042, *Laboratory glassware - One-mark volumetric flasks (ISO 1042)*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12258-2:2004 apply.

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

- IEC Electropedia: available at <https://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

## 4 Principle

A test portion is dissolved with:

- a sodium hydroxide solution followed by acidification with a mixture of nitric acid and hydrochloric acid; or
- nitric acid and hydrofluoric acid; or
- a mixture of hydrochloric acid and nitric acid; or
- hydrochloric acid and hydrogen peroxide

according to the alloy type and the element to be determined.

After suitable dilution and, if necessary, addition of an internal reference element, the solution is nebulized into an inductively coupled plasma optical emission spectrometer and the intensity of the emitted light (including, where appropriate, that of the internal reference element) is measured. The

emission signals on the selected analytical lines (see Annex A) are then compared with those of the calibration solutions.

NOTE 1 The ranges of application and the accuracy of the method or any alternative steps are validated by the laboratory. Approximate ranges of application are given in Annex A.

NOTE 2 All instrumentation, including software used in the laboratories, are different and subject to change. Therefore, only general criteria for calibration and measurement are specified.

## 5 Reagents

During the analysis, unless otherwise specified, use only reagents of recognized analytical grade and only grade 2 water as specified in EN ISO 3696 or equivalent quality.

The same reagents should be used for preparation of calibration solutions and of sample solutions.

**5.1 Aluminium**, purity  $\geq 99,99\%$  by mass.

**5.2 Sodium carbonate ( $\text{Na}_2\text{CO}_3$ )**

**5.3 Potassium carbonate ( $\text{K}_2\text{CO}_3$ )**

**5.4 Sodium nitrite ( $\text{NaNO}_2$ )**

**5.5 Potassium disulphate ( $\text{K}_2\text{S}_2\text{O}_7$ )**

**5.6 Nitric acid**,  $\rho = 1,40$  g/ml approximately.

**5.7 Nitric acid solution**, 1 + 1

Carefully add 500 ml of nitric acid (5.6) to 400 ml water, allow to cool, dilute to 1 l with water and mix.

**5.8 Nitric acid solution**, 4 mol/l

Carefully add 27,7 ml of nitric acid (5.6) to 50 ml water, allow to cool, dilute to 100 ml with water and mix.

**5.9 Hydrochloric acid**,  $\rho = 1,19$  g/ml, approximately

**5.10 Hydrochloric acid solution**, 1 + 1

Carefully add 500 ml of hydrochloric acid (5.9) to 400 ml water, allow to cool, dilute to 1 l with water and mix.

**5.11 Hydrofluoric acid**,  $\rho = 1,14$  g/ml, approximately.

**5.12 Sulphuric acid**,  $\rho = 1,84$  g/ml approximately.

**5.13 Hydrogen peroxide**, 30 % (by mass) solution.

**5.14 Sodium hydroxide solution**, 400 g/l

Transfer 400,0 g of sodium hydroxide ( $\text{NaOH}$ ) into a plastic beaker with a lid and carefully add 500 ml of water. Transfer the solution into a 1 000 ml volumetric plastic flask.

Dilute to the mark with water and mix.

## 5.15 Standard solutions

The standard solutions shall be traceable to international units mass or amount of substances i.e. kilogram or mol. They should be prepared from pure metals or stoichiometric compounds.

Standard solutions containing sulphate ions shall not be used for the determination of elements which form insoluble compounds with sulphate ions.

Standard solutions and calibration solutions with element concentrations  $\leq 50 \text{ mg/l}$  can be unstable and shall be controlled before use.

NOTE 1 Calibration solutions can be prepared directly from standard solutions by weighing (see 8.6).

NOTE 2 For routine analysis, commercial standard solutions with stated traceability can also be used.

In the case of solution prepared before use and stored in appropriate receptacles, their concentration shall either be controlled before use or their stability over time be documented.

### 5.15.1 Antimony standard solution, 200 mg/l

Transfer 0,100 g of antimony (purity  $\geq 99,99\%$  by mass) into a 250 ml beaker with a lid. Add 50 ml of hydrochloric acid (5.9) and 2 ml of nitric acid (5.6), heat to complete the dissolution. Allow to cool. Carefully add 50 ml of water and 50 ml of hydrochloric acid (5.9), transfer the solution quantitatively into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 200  $\mu\text{g}$  of antimony.

### 5.15.2 Beryllium standard solution, 1 g/l

Transfer 1,000 g of beryllium (purity  $\geq 99,99\%$  by mass) into a 400 ml beaker with a lid. Add 20 ml of hydrochloric acid solution (5.10). Heat gently, if necessary, until the dissolution is complete, allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of beryllium.

### 5.15.3 Bismuth standard solution, 1 g/l

Transfer 1,000 g of bismuth (purity  $\geq 99,99\%$  by mass) into a 400 ml beaker with a lid. Add 18 ml of hydrochloric acid (5.9), 6 ml of nitric acid (5.6) and 10 ml of water. Heat gently, if necessary, until the dissolution is complete. Add 160 ml of hydrochloric acid solution (5.10), transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of bismuth.

### 5.15.4 Cadmium standard solution, 1 g/l

Transfer 1,000 g of cadmium (purity  $\geq 99,99\%$  by mass) into a 400 ml beaker with a lid. Add of 10 ml of water and 30 ml of nitric acid (5.6). Heat gently, if necessary, until the dissolution is complete, allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of cadmium.

### 5.15.5 Calcium standard solution, 1 g/l

Transfer 2,497 3 g of calcium carbonate (purity  $\geq 99,99\%$  by mass), previously dried at 200 °C to constant mass into a 400 ml beaker with a lid. Dissolve in 40 ml of hydrochloric acid solution (5.10), transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.



1 ml of this solution contains 1 mg of calcium.

#### **5.15.6 Chromium standard solution, 1 g/l**

##### **5.15.6.1 Preparation using chromium metal**

Transfer 0,5 g of chromium (purity  $\geq 99,99$  % by mass) into a 250 ml beaker with a lid. Add 40 ml of water and 20 ml of hydrochloric acid (5.9). Heat gently, if necessary, until the dissolution is complete, allow to cool and transfer the solution quantitatively into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of chromium.

##### **5.15.6.2 Preparation using potassium dichromate**

Transfer 2,828 9 g of potassium dichromate ( $K_2Cr_2O_7$ ) previously dried at 130 °C to constant mass, into a 400 ml beaker with a lid. Add 10 ml of water, 20 ml of hydrochloric acid solution (5.10) and dropwise 20 ml of hydrogen peroxide (5.13). Heat gently, without boiling, to evaporate the excess of hydrogen peroxide. Allow to cool and transfer the solution quantitatively into a 1 000 ml volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of chromium.

#### **5.15.7 Cobalt standard solution, 1 g/l**

Transfer 1,000 g of cobalt (purity  $\geq 99,99$  % by mass) into a 400 ml beaker with a lid. Add 10 ml of water, 18 ml of hydrochloric acid (5.9) and 6 ml of nitric acid (5.6). Heat gently, if necessary, until the dissolution is complete, allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of cobalt.

#### **5.15.8 Copper standard solution, 1 g/l**

Transfer 1,000 g of copper (purity  $\geq 99,99$  % by mass) into a 400 ml beaker with a lid. Add 40 ml of hydrochloric acid solution (5.10) and stepwise 5 ml of hydrogen peroxide (5.13) while stirring. Heat until the solution boils, allow to cool, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of copper.

#### **5.15.9 Gallium standard solution, 1 g/l**

Transfer 1,000 g of gallium (purity  $\geq 99,99$  % by mass) into a 400 ml beaker with a lid. Add 10 ml of water and 30 ml of nitric acid (5.6). Heat gently, if necessary, until the dissolution is complete, allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of gallium.

#### **5.15.10 Iron standard solution, 1 g/l**

Transfer 1,000 g of iron (purity  $\geq 99,99$  % by mass) into a 400 ml beaker with a lid. Add 40 ml of hydrochloric acid solution (5.10). Heat gently, if necessary, until the dissolution is complete, allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of iron.

#### 5.15.11 Lead standard solution, 1 g/l

Transfer 1,000 g of lead (purity  $\geq 99,99$  % by mass), into a 250 ml beaker with a lid. Add 10 ml of water and 10 ml of nitric acid solution (5.7). Heat gently, if necessary, until the dissolution is complete, then boil until nitrous fumes have been expelled. Allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of lead.

#### 5.15.12 Lithium standard solution, 1 g/l

Transfer 5,324 0 g of lithium carbonate (purity  $\geq 99,99$  % by mass), previously dried at 200 °C to constant mass into a 400 ml beaker with a lid. Dissolve in 40 ml of hydrochloric acid solution (5.10), transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of lithium.

#### 5.15.13 Magnesium standard solution, 1 g/l

Transfer 1,000 g of magnesium (purity  $\geq 99,99$  % by mass) into a 400 ml beaker with a lid. Add, by small fractions, 40 ml of hydrochloric acid solution (5.10). Heat gently, if necessary, until the dissolution is complete, allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of magnesium.

#### 5.15.14 Manganese standard solution, 1 g/l

The manganese (purity  $\geq 99,99$  % by mass) used to prepare the solution is released from superficial oxide possibly present by introducing a few grams of metal in a 250 ml beaker containing 150 to 160 ml of water and 15 to 20 ml of sulphuric acid (5.12). Shake and after a few seconds, allow the solution to settle and add water. Repeat the water cleaning several times. Remove the metallic manganese and rinse with acetone. Dry the metal in an oven at 100 °C for 2 minutes or with a hair dryer. Cool in a desiccator.

Transfer 1,000 g of manganese, precleaned as described above, into a 400 ml beaker with a lid. Add 40 ml of hydrochloric acid solution (5.10). Heat gently, if necessary, until the dissolution is complete, allow to cool, transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of manganese.

#### 5.15.15 Nickel standard solution, 1 g/l

Transfer 1,000 g of nickel (purity  $\geq 99,99$  % by mass) into a 400 ml beaker with a lid. Add 40 ml of hydrochloric acid solution (5.10). Heat gently, if necessary, until the dissolution is complete, allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of nickel.

#### 5.15.16 Sodium standard solution, 1 g/l

Transfer 2,305 1 g of sodium carbonate (purity  $\geq 99,99$  % by mass), previously dried at 200 °C to constant mass into a 400 ml beaker with a lid. Dissolve in 40 ml of hydrochloric acid solution (5.10), transfer the solution into a 1 000 ml volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of sodium.

#### 5.15.17 Silicon standard solution, 100 mg/l

In a large platinum crucible with a lid, fuse 0,213 92 g of silica ( $\text{SiO}_2$ , purity  $\geq 99,999\%$  by mass), previously calcined at 1 000 °C to constant mass, with 2 g of a mixture of equal parts of sodium carbonate (5.2) and potassium carbonate (5.3). Continue the fusion until a clear melt is obtained. Allow to cool, transfer the melt into a 600 ml PTFE beaker with a lid, dissolve the fused mass with 40 ml of water. Heat gently until the dissolution is complete. Slowly add 40 ml of nitric acid (5.6) while stirring strongly if possible by means of a magnetic stirrer. Transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask, dilute to the mark with water and mix.

1 ml of this solution contains 100  $\mu\text{g}$  of silicon.

NOTE This solution can be used for about 2 weeks.

#### 5.15.18 Strontium standard solution, 1 mg/l

Transfer 1,685 0 g of strontium carbonate (purity  $\geq 99,99\%$  by mass), previously dried at 150 °C to constant mass into a 400 ml beaker with a lid. Dissolve in 40 ml of hydrochloric acid solution (5.10), transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of strontium.

#### 5.15.19 Tin standard solution, 500 mg/l

Transfer 0,500 g of tin (purity  $\geq 99,99\%$  by mass) into a 400 ml beaker with a lid. Add 100 ml of hydrochloric acid (5.9). Heat gently until the dissolution is complete, allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 500  $\mu\text{g}$  of tin.

#### 5.15.20 Titanium standard solution, 1 g/l

Transfer 0,200 g of titanium (purity  $\geq 99,99\%$  by mass) into a 250 ml beaker with a lid. Add 50 ml of hydrochloric acid solution (5.10) and 5 drops of hydrofluoric acid (5.11). Heat gently, if necessary, until the dissolution is complete, allow to cool and transfer the solution quantitatively into a 200 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of titanium.

#### 5.15.21 Vanadium standard solution, 1 g/l

Transfer 0,5 g of vanadium (purity  $\geq 99,99\%$  by mass) into a 250 ml beaker with a lid. Add 30 ml of hydrochloric acid (5.9) and 10 ml of nitric acid (5.6). Heat gently, if necessary, until the dissolution is complete, allow to cool and transfer the solution quantitatively into a 500 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of vanadium.

#### 5.15.22 Zinc standard solution, 1 g/l

Transfer 1,000 g of zinc (purity  $\geq 99,99\%$  by mass) into a 400 ml beaker with a lid. Add 40 ml of hydrochloric acid solution (5.10). Heat gently, if necessary, until the dissolution is complete, allow to cool and transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of zinc.

#### 5.15.23 Zirconium standard solution, 1 g/l

In a large platinum crucible with a lid, fuse 1,350 8 g of zirconium dioxide  $ZrO_2$ , (purity  $\geq 99,99$  % by mass), previously calcined at 1 000 °C to constant mass, with 10 g of potassium disulphate (5.5). After cooling, dissolve the fused product with 25 ml of hydrochloric acid solution (5.10). Transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the mark with water and mix.

1 ml of this solution contains 1 mg of zirconium.

#### 5.16 Internal reference element solutions

##### 5.16.1 Lanthanum solution, 1 g/l

Transfer 1,173 g of lanthanum oxide  $La_2O_3$ , (purity  $\geq 99,99$  %, by mass), previously dried at 150 °C to constant mass, into a 400 ml beaker with a lid. Dissolve in 40 ml of hydrochloric acid solution (5.10), transfer the solution into a 1 000 ml volumetric flask, dilute to the mark with water and mix.

##### 5.16.2 Lanthanum solution, 200 mg/l

Transfer 20,0 ml of the lanthanum solution (5.16.1) into a 100 ml volumetric flask, add 1 ml of hydrochloric acid solution (5.10), dilute to the mark with water and mix.

##### 5.16.3 Scandium solution, 0,1 g/l

Transfer 100,0 mg of scandium (purity  $\geq 99,99$  %, by mass) into a 400 ml beaker with a lid. Add 20 ml of hydrochloric acid solution (5.10). Heat gently, if necessary, until the dissolution is complete. Allow to cool, transfer the solution into a 1 000 ml volumetric flask, dilute to the mark with water and mix.

NOTE Scandium solutions can be unstable.

##### 5.16.4 Scandium solution, 20 mg/l

Transfer 20,0 ml of the scandium solution (5.16.3) into a 100 ml volumetric flask, add 1 ml of hydrochloric acid solution (5.10), dilute to the mark with water and mix.

##### 5.16.5 Molybdenum solution, 1 g/l

Dissolve 1,84 g of ammonium molybdate  $(NH_4)_6Mo_7O_{24} \times 4H_2O$  into a 1 000 ml volumetric flask with water. Dilute to the mark with water and mix.

##### 5.16.6 Molybdenum solution, 200 mg/l

Transfer 20,0 ml of the molybdenum solution (5.16.5) into a 100 ml volumetric flask, add 1 ml of hydrochloric acid solution (5.10), dilute to the mark with water and mix.

## 6 Apparatus

All volumetric glassware shall be class A and calibrated in accordance with EN ISO 648 or EN ISO 1042, as appropriate.

Ordinary laboratory equipment can be the following.

### 6.1 Optical emission spectrometer, equipped with inductively coupled plasma.

This shall be equipped with a nebulization system. The instrument used will be satisfactory if, after optimizing in accordance with the manufacturer's instructions, it meets the performance criteria given in Annex B.

The spectrometer can be either a simultaneous or a sequential one (for analytical wavelengths, see Annex A). If a sequential spectrometer can be equipped with an extra arrangement for simultaneous measurement of the internal reference element line, it can be used with the internal reference method. If the sequential spectrometer is not equipped with this arrangement, an internal reference cannot be used and an alternative measurement technique without internal reference element shall be used.

For solutions containing hydrofluoric acid the instrument shall be equipped with a fluoride-resistant nebulizer.

The nebulizer should be selected carefully. Many of the available fluoride-resistant nebulizers are not as stable as the glass nebulizers.

**6.2 PTFE-beakers**, 400 ml, with lids.

**6.3 Platinum crucibles**, 100 ml to 120 ml, with lids.

**6.4 Plastic bottles**, approximately 100 ml, with small neck and caps.

Plastic vessel should be selected carefully to avoid losses and contaminations.

## 7 Sampling

### 7.1 General

Sampling from aluminium melts shall be carried out in accordance with EN 14361.

The results of the analysis should represent the average chemical composition of a test sample, a laboratory sample or an inspection lot.

### 7.2 Test sample

The test sample is normally in the form of millings or drillings.

If the chemical composition of the test sample is thought to be not homogeneous, e.g. in case of unwrought products, its preparation shall follow a documented procedure to ensure the required representativity.

If necessary, e.g. for the determination of low contents of alkaline or alkaline earth elements, the test sample should be cleaned in accordance with a suitable procedure (solution, temperature and time). The cleaning procedure shall be adapted to the alloy, analytes and the content to be determined.

The test sample should be sufficiently homogeneous regarding the quantity of the test portion.

## 8 Procedure

### 8.1 Test portion

Weigh, to the nearest 0,001 g, about 0,5 g of the test sample.

### 8.2 Dissolution procedure I with sodium hydroxide solution

**8.2.1** This procedure is applicable for the determination of silicon, iron, copper, manganese, magnesium, chromium, nickel, zinc, titanium, vanadium, beryllium, cadmium, cobalt, lead, antimony, tin, strontium and zirconium in aluminium and aluminium alloys.

**8.2.2** Transfer the test portion (8.1) into a 400 ml PTFE-beaker with a lid (6.2) or into a platinum crucible with a lid (6.3).

**8.2.3** Cover the test portion with water. Add 6 ml of sodium hydroxide solution (5.14). After the dissolution reaction ceases, add 1 ml hydrogen peroxide (5.13). Heat gently until the dissolution is complete.

**8.2.4** If the silicon content of the test sample is more than 0,5 % by mass carefully evaporate to a syrupy consistency. Allow to cool a little, carefully add 30 ml of water and heat gently until the dissolution is complete. If a platinum crucible (6.3) has been used, transfer the solution quantitatively into a 400 ml PTFE-beaker, containing 25 ml of nitric acid solution (5.7) and 10 ml of hydrochloric acid solution (5.10).

**8.2.5** Make up to volume to about 100 ml with water and add 25 ml of nitric acid solution (5.7) and 25 ml of hydrochloric acid solution (5.10) while stirring. Heat the solution immediately while frequently and carefully shaking to complete the dissolution. In case of precipitation of manganese (IV) oxyhydrate (in the case of alloys having high manganese contents), dissolve the precipitate with a spatula tip of sodium nitrite (5.4). Allow to cool and transfer the clear solution into a 500 ml volumetric flask.

**8.2.6** If a platinum crucible (6.3) has been used, add into the empty crucible some drops of nitric acid solution (5.8) and a spatula tip of sodium nitrite (5.4), cover the crucible and heat. Rinse the lid and the walls of the crucible with water. Allow to cool. Transfer the solution quantitatively into the 500 ml flask.

**8.2.7** When appropriate, add about 10 g, weighed to the nearest 0,01 g or 10,00 ml of one of the internal reference element solutions (5.16.1, 5.16.3 or 5.16.5) (see Clause 9).

**8.2.8** Dilute to the mark with water and mix.

### **8.3 Dissolution procedure II with nitric acid and hydrofluoric acid**

**8.3.1** This procedure is applicable for the determination of iron, copper, manganese, magnesium, chromium, nickel, zinc, titanium, gallium, vanadium, beryllium, bismuth, calcium, cadmium, cobalt, lithium, sodium, lead, antimony, tin, strontium and zirconium in aluminium and aluminium alloys.

**8.3.2** Transfer the test portion (8.1) into a previously weighed 100 ml plastic bottle (6.4).

**8.3.3** Add 10 ml of nitric acid solution (5.7) and 2 ml of hydrofluoric acid (5.11).

**8.3.4** Cover the bottle with a cap. Do not screw the cap.

**8.3.5** Heat in a water bath or in a thermoblock (max. 70 °C) to complete the dissolution.

**8.3.6** When appropriate add about 2 g, weighed to the nearest 0,002 g of the internal reference element solution (5.16.5) or 10,00 ml of the internal reference element solution (5.16.6) (see Clause 9).

**Do not add lanthanum or scandium solutions in order to avoid fluoride precipitation.**

**8.3.7** Make up to 100,0 g with water and mix.

In case of magnesium contents higher than 1 % by mass, insoluble magnesium fluoride may precipitate. In such cases, another dissolution procedure should be selected.

### **8.4 Dissolution procedure III with a mixture of hydrochloric acid and nitric acid**

**8.4.1** This procedure is applicable for the determination of iron, copper, manganese, magnesium, chromium, nickel, zinc, titanium, gallium, vanadium, beryllium, bismuth, calcium, cadmium, cobalt, lithium, sodium, lead, antimony, tin, strontium and zirconium in aluminium and aluminium alloys having silicon contents < 0,3 % by mass.

**8.4.2** Transfer the test portion (8.1) into a 250 ml glass beaker with lid.

**8.4.3** Add 10 ml of an acid mixture [3 parts of hydrochloric acid (5.9) and 1 part of nitric acid (5.6)]. Heat gently to complete the dissolution.

**8.4.4** Transfer the solution quantitatively into a 100 ml volumetric flask.

**8.4.5** When appropriate, add about 2 g, weighed to the nearest 0,002 g, of one of the internal reference element solutions (5.16.1, 5.16.3 or 5.16.5) or 10,00 ml from one of the internal reference element solutions (5.16.2, 5.16.4 or 5.16.6) (see Clause 9).

**8.4.6** Dilute to the mark with water and mix.

## **8.5 Dissolution procedure IV with hydrochloric acid**

**8.5.1** This procedure is applicable for the determination of iron, copper, manganese, magnesium, chromium, nickel, zinc, titanium, gallium, vanadium, beryllium, calcium, cadmium, cobalt, lithium, sodium, lead, tin, strontium, and zirconium in aluminium and aluminium alloys.

NOTE If silicon content is higher than 0,3 % by mass, this procedure cannot be used for the determination of cadmium.

**8.5.2** Transfer the test portion (8.1) into a 250 ml glass beaker with lid.

**8.5.3** Add 10 ml of hydrochloric acid (5.9). Heat gently, if necessary. When the reaction ceases, add 1 ml of hydrogen peroxide (5.13) and heat gently to complete the dissolution.

**8.5.4** If undissolved residues remain, indicating the presence of more than 0,3 % by mass of silicon, filter the solution through an ashless filter paper and collect the filtrate in a 100 ml one-mark volumetric flask. Wash the filter, transfer the filter paper and its content into a platinum crucible (6.3), dry and ignite first at a relatively low temperature (until all carbonaceous matter is removed) and then at about 850 °C for at least 15 min. Allow to cool. Add 3 ml hydrofluoric acid (5.11) and dropwise, about 1 ml of nitric acid (5.6). Evaporate near to dryness and allow to cool.

Add into the crucible 3 ml of hydrochloric acid solution (5.10), and heat gently to dissolve the residue.

**8.5.5** Allow the crucible to cool and transfer the solution quantitatively to the filtrate in the 100 ml one-mark volumetric flask.

NOTE This step can increase the blanks, especially for calcium and sodium.

**8.5.6** When appropriate add about 2 g, weighed to the nearest 0,002 g, of one of the internal reference element solutions (5.16.1, 5.16.3 or 5.16.5) or 10,00 ml from one of the internal reference element solutions (5.16.2, 5.16.4 or 5.16.6) (see Clause 9).

**8.5.7** Dilute to the mark with water and mix.

## **8.6 Calibration solutions and drift correction solution**

### **8.6.1 General**

In all cases the aluminium and the acid concentration of the calibration solutions shall correspond to the concentrations of the test solutions.

The number of calibration solutions depends on the required accuracy. A minimum of five calibration solutions is required. The precision as well as the trueness of the results can be checked by analysing some Certified Reference Materials (CRMs).

If, for measurement purposes, a blank is required, prepare a solution containing only the same amounts of acids as in the test solution.

The solution with the highest analyte concentrations shall be used as drift correction solution.

## 8.6.2 Preparation of the calibration solutions

### 8.6.2.1 Dissolution procedures

Dissolution procedure I: Weigh, to the nearest 0,001 g about 0,5 g of aluminium (5.1) into a series of 400 ml PTFE beakers with lids (6.2) and follow the procedure described from 8.2.3 to 8.2.7. Continue as described in 8.6.2.2.

Dissolution procedure II: Weigh, to the nearest 0,001 g about 0,5 g of aluminium (5.1) into a series of previously weighed 100 ml plastic bottles (6.4) and follow the procedure described from 8.3.3 to 8.3.6. Continue as described in 8.6.2.2.

Dissolution procedure III: Weigh, to the nearest 0,001 g about 0,5 g of aluminium (5.1) into a series of 250 ml glass beakers and follow the procedure described from 8.4.3 to 8.4.5. Continue as described in 8.6.2.2.

Dissolution procedure IV: Weigh, to the nearest 0,001 g about 0,5 g of aluminium (5.1) into a series of 250 ml glass beakers and follow the procedure described from 8.5.3 to 8.5.6. Continue as described in 8.6.2.2.

For all dissolution procedures the mass of 0,5 g of aluminium (5.1) has to be reduced by the sum of the mass of the elements which are added under 8.6.2.2, if this sum is more than 2 % of the total mass.

**8.6.2.2** Transfer into each of the one-mark volumetric flasks or plastic bottles the weights or volumes of the standard solutions (5.15). Dilute to the mark with water and mix.

## 8.7 Measurements

### 8.7.1 Adjustment of the apparatus

Start the inductively coupled plasma optical emission spectrometer and let it stabilize in accordance with the manufacturer's instructions before carrying out any measurements.

Optimize the instrument according to the manufacturer's instructions.

Check the instrument performance in accordance with the criteria given in Annex B.

Prepare the software to measure the intensity, and for the calculation of the mean value and relative standard deviation corresponding to each wavelength.

Each time an internal reference element is used prepare the software to calculate the ratio between each analyte intensity and internal reference element intensity.

### 8.7.2 Measurement of the calibration solutions

Measure the absolute intensities or the ratioed intensities at the wavelengths preselected beginning with the zero member of the calibration and ending up with the calibration solution of highest concentration. Measure each of the calibration solutions two times and calculate the corresponding average values.

Nebulize water between each measurement.



### 8.7.3 Measurement of the test solutions

Measure the absolute intensity or ratioed intensity of the test solution two times and calculate the corresponding average values.

Nebulize water between each measurement.

### 8.8 Calibration curves

Establish the calibration curves using the measured intensities or the ratioed intensities and the corresponding analyte amounts.

Use the appropriate software of the spectrometer for regression calculations and calculate each correlation coefficient.

The coefficients of correlation shall meet the criteria given in Annex B.

## 9 Correction of short-term fluctuations and drift

### 9.1 General

The accuracy and comparability of measured values can be influenced by short-term fluctuations, drift and matrix effects in solutions. Sometimes, it is difficult to separate the causes of these influences from one another. In any case, all three effects shall be taken into account appropriately. To reduce certain error inducing effects caused by the matrix, care shall be taken to ensure that the salt and, above all, acidic concentrations of the test, blank, calibration solutions are comparable and, as a consequence, a comparable nebulizing rate is achieved.

### 9.2 Short-term fluctuations

Due to the different evaporation of solutions and different element concentrations caused by the sample preparation and the preparation of the calibration solutions, the viscosity and surface tension of solutions can be different and, as a result, can influence the transport efficiency of the feeding to the atomiser. This, in turn, can affect the performance of the peristaltic pump. Short-term fluctuations may be corrected by adding an internal reference element (e.g. lanthanum, molybdenum or scandium) to the test solution, blank solution and calibration solutions; in this case analyte and internal reference element shall be measured simultaneously.

### 9.3 Drift

Drift can be caused, for example, by changes in the excitation temperature or changes in the performance of the generator.

To correct a signal drift of the test solutions and calibration solutions, at least the low concentration and the high concentration solutions of the calibration series, shall be measured alternately throughout the entire analytical series so that the drift effect can be corrected.

## 10 Investigation of interferences

The magnitude of the spectral and chemical interferences depends on the chemical composition of the sample and the resolution and the stability of the optical part of the spectrometer. The following tests shall be performed for each element which can interfere the analyte signal and shall be repeated periodically depending on the stability of the optical part of the spectrometer.

Prepare four matrix solutions according to 8.6.2. The mass of aluminium (5.1) shall be 0,5 g in all four solutions.

Add to three matrix solutions within the linear range of the calibration curve, weights or volumes of the standard solutions (5.15) of the element to be checked.

The concentration of the analyte in the matrix solutions used for those tests should be below the detection limit of the method. Investigate blanks of interfering elements in the matrix solution and in the standard solutions using several analytical wavelengths or by using an independent method (e.g. electrothermal atomic absorption spectrometry).

Measure according to 8.7.3.

Record the signal intensities of the solutions at the selected wavelength and calculate the interference per equivalent mass content of the interfering element (correction factors).

## 11 Expression of the results

### 11.1 Correction

Calculate, for each solution, the ratio of the measured intensity of the analyte and the intensity of the reference element recorded simultaneously.

Calculate the element concentrations of the solutions, taking into account the correction factors, the drift correction (ratio of the relative intensities of the calibration solutions and the drift correction solution).

### 11.2 Result

Calculate the element content of the sample taking into account the weight of the test portion and the dilution (if any), expressed as percentage by mass.

## 12 Test report

The test report shall contain the following information:

- a) identification of the test sample;
- b) test method used by reference to the present document;
- c) results as well as the units in which they are expressed;
- d) any unusual characteristics noted during the determination;
- e) any operation not included in this document or in the document to which reference is made or regarded as optional;
- f) date of the test and/or date of preparation or signature of the test report;
- g) signature of the responsible person.

**Annex A**  
(informative)

**Analytical wavelengths**

Table A.1 lists the recommended wavelengths for the analysis of aluminium and aluminium alloys by ICP-OES. This Table also shows the range of application and the interferences corresponding to each wavelength.

**Table A.1 — Wavelengths, range of application and interferences**

Element	Wavelength	Range <sup>a</sup> %	Interfering element <sup>b</sup>
Si	251,611 288,158	0,005 to 25 0,01 to 25	Ca
Fe	259,940 238,207	0,002 to 5 0,002 to 2	Mn
Cu	222,778 324,754	0,1 to 5 0,001 to 5	
Mn	257,610 260,730	0,001 to 2 0,001 to 3	Al, Ni Fe, Cr
Mg	279,079 279,553 280,270	0,002 to 5 0,000 5 to 1 0,000 5 to 1	V
Cr	267,716 276,650	0,001 to 1 0,001 to 1	Mg, Mn, P Cu, Fe, Mg, Mn, V
Ni	231,600 221,650	0,001 to 5 0,001 to 5	Co, Mg Al, Cu, Fe, Mg, Mn, Si, Zn
Zn	213,856 206,200	0,000 5 to 1 0,01 to 10	Al, Ni, Cu Al, Cu, Cr, Fe, Mg, Pb, V, Zr
Ti	334,940 337,280 368,520	0,001 to 2 0,001 to 5 0,001 to 5	Zr
Ga	294,363	0,001 to 0,05	
V	292,400 310,230	0,000 5 to 0,1 0,001 to 0,1	
Be	234,861 313,042	0,000 1 to 0,01 0,000 1 to 0,01	Fe
Bi	222,830 223,061	0,02 to 2	Cu, Ti
Ca	317,933 393,366	0,001 to 0,02 0,000 5 to 0,02	Zr, V
Cd	214,439 226,502 228,802	0,001 to 0,05 0,001 to 0,05 0,001 to 0,05	Pt, Sb Co, Sc, As
Co	228,620	0,001 to 5	Ti

Element	Wavelength nm	Range <sup>a</sup> %	Interfering element <sup>b</sup>
La <sup>c</sup>	333,754 379,478 398,852		
Li	670,783	0,000 1 to 0,01	
Mo <sup>c</sup>	202,030		
Na	588,995	0,001 to 0,02	
Pb	220,353 283,306	0,04 to 1 0,04 to 2	Al Fe
Sb	217,581	0,05 to 1	Al, Mg
Sc <sup>c</sup>	361,383		
Sn	189,989	0,02 to 1	Al, Mg
Sr	407,771	0,000 1 to 0,1	
Zr	339,198 343,823	0,001 to 2 0,001 to 2	Cr, Fe, Mo

<sup>a</sup> Approximate ranges corresponding to the test portions and dilutions described in the procedures.

<sup>b</sup> The interferences of elements should have to be checked in any case. The interfering elements and the extent of interferences depend on the spectrometer.

<sup>c</sup> Internal reference element.

## Annex B (informative)

### Plasma optical emission spectrometer — Suggested performance criteria to be checked

#### B.1 Short- and long-term stability

The evaluation of the short-term stability consists of the calculation of the repeatability standard deviation of a series of measurements carried out with the spectrometer.

For each analyte, a series of ten consecutive intensity measurements of its highest multi-element calibration solution is carried out using the typical integration time for the system. The average  $I_{\text{avrg}}$  and the standard deviation  $S_I$  of the ten measurements are calculated. In addition, the calculation of the relative standard deviation  $RSD_I$  is carried out in accordance to Formula (B.1):

$$RSD_I = (S_I / I_{\text{avrg}}) \times 100 \text{ (in \%)} \quad (\text{B.1})$$

In inductively coupled plasma optical emission spectrometry, for solutions with concentrations of at least twice the background-equivalent concentration (BEC),  $RSD_I$ -values between 0,3 % and 1,0 % are generally accepted. Multi-elemental calibration solutions may be used for measurement at various wavelengths present in simultaneous optics.

Long term stability assessment is a measurement of the instrument drift. This is only required if the spectrometer is set up to work for long intervals of time. It consists of carrying out the same short-term stability tests at specific intervals of time, 15 min to 1 h, and plotting the deviation of the average found for each short-term test against time. Deviations of more than 2 % per hour should not be accepted. In case the instrument is not able to perform better, the control calibration solution should be measured more often during the analysis and the mean results of the test sample solutions should be recalculated by interpolation between two consecutive measurements.

#### B.2 Evaluating the background equivalent concentration

The background equivalent concentration (BEC) is used as an evaluation of the instrument sensitivity. Since the analyte signal has usually a relatively high background, its correction by the background intensity is recommended. The background equivalent concentration is calculated in accordance to Formula (B.2):

$$\text{BEC} = (I_{\text{BG}} / I_{\text{net}}) \times C_{\text{Analyte}} \quad (\text{B.2})$$

where

$I_{\text{BG}}$  is the intensity of the background;

$I_{\text{net}}$  is the intensity of the analyte (overall intensity minus intensity of the background);

$C_{\text{Analyte}}$  is the concentration of analyte that yields  $I_{\text{net}}$ .

The background equivalent concentration values for the elements to be analysed can be found in wavelength tables (usually part of the instrument software). The lower the value of the background concentration, the better the instrument sensitivity.

### B.3 Evaluating the limit of detection

Operate as follows:

- for each element, measure 10 times under repeatability conditions, the intensity or the ratioed intensity of the "blank solution" and calculate the corresponding average ( $I_0$ ) and standard deviation ( $\sigma_0$ );
- then, for each element, measure 10 times under repeatability conditions, the intensity or the ratioed intensity of the lowest concentration ( $C_x$ ) standard solution (lowest limit of each proposed range of content) and calculate each corresponding average ( $I_x$ );
- taking into account the statistical risks  $\alpha$  and  $\beta$  each of them at the 95 % level, the limit of detection ( $C_{lim}$ ) is calculated according to Formula (B.3):

$$C_{lim} = \frac{4,65 \times \sigma_0 \times C_x}{I_x - I_0} \quad (B.3)$$

### B.4 Linearity of the calibration curves

The linearity of the calibration curves is checked by calculating the corresponding correlation coefficients: they shall be higher than 0,999.

## Bibliography

- [1] EN 10351, *Chemical analysis of ferrous materials - Inductively coupled plasma optical emission spectrometric analysis of unalloyed and low alloyed steels - Determination of Si, Mn, P, Cu, Ni, Cr, Mo, V, Co, Al (total) and Sn [Routine method]*
- [2] EN 15605, *Copper and copper alloys - Inductively coupled plasma optical emission spectrometry*

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