SLOVENSKI STANDARD
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Aluminij in aluminijeve zlitine - Kemična analiza - Analiza s spektrometrijo optične emisije z induktivno sklopljeno plazmo

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

Aluminium und Aluminiumlegierungen - Chemische Analyse - Optische Emissionspektralanalyse mit induktiv gekoppelter Plasmaanregung

Aluminium et alliages d'aluminium - Analyse chimique - Analyse par spectrométrie d'émission optique en plasma induit

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77.040.30	Kemijska analiza kovin	Chemical analysis of metals
77.120.10	Aluminij in aluminijeve zlitine	Aluminium and aluminium alloys

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English Version

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

Aluminium et alliages d'aluminium - Analyse chimique - Analyse par spectrométrie d'émission optique en plasma induit

Aluminium und Aluminiumlegierungen - Chemische Analyse - Optische Emissionspektralanalyse mit induktiv gekoppelter Plasmaanregung

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

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European foreword

This document (prEN 14242:2022) has been prepared by Technical Committee CEN/TC 132 “Aluminium and aluminium alloys”, the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 14242:2002.

In comparison with the previous edition, the following technical modifications have been made:

— Modification of the scope;

— New subclause 5.15.6;

— Several editorial modifications.

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, cadmium, 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 detection limits of the method.

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 12258-2:2001, *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 12258-2:2001 apply.

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, nebulisation of the solution into an inductively coupled plasma emission spectrometer and measurement of the intensity of the emitted light (including, where appropriate, that of the internal reference element).

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 is 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.

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

5.1 Aluminium, purity ≥ 99,999 % by mass.

5.2 Sodium carbonate (Na_2CO_3)

5.3 Potassium carbonate (K_2CO_3)

5.4 Sodium nitrite ($NaNO_2$)

5.5 Potassium disulphate ($K_2S_2O_7$)

5.6 Nitric acid, ρ = 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, ρ = 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, ρ = 1,14 g/ml, approximately.

5.12 Sulphuric acid, ρ = 1,84 g/ml approximately.

5.13 Hydrogen peroxide, 30 % (mass fraction) solution.

5.14 Sodium hydroxide solution, 400 g/l

Transfer 400,0 g of sodium hydroxide (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 ≤ 50 mg/l can be unstable and shall be prepared just before use.

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

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

5.15.1 Antimony standard solution, 200 mg/l

Transfer 0,100 g of antimony (purity ≥ 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 volume with water and mix.

1 ml of this solution contains 200 µg of antimony.

5.15.2 Beryllium standard solution, 1 g/l

Transfer 1,000 g of beryllium (purity ≥ 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 volume 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 ≥ 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 volume 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 ≥ 99,99 % by mass) to 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 volume with water and mix.

1 ml of this solution contains 1 mg of cadmium.

5.15.5 Calcium standard solution, 1 g/l

Transfer 2,4973 g of calcium carbonate (purity ≥ 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 volume 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 ≥ 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 volume 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 40 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 volume 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 ≥ 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 volume 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 ≥ 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 volume 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 ≥ 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 volume 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 ≥ 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 volume 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 ≥ 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 volume with water and mix.

1 ml of this solution contains 1 mg of lead.

5.15.12 Lithium standard solution, 1 g/l

Transfer 5,3240 g of lithium carbonate (purity ≥ 99,99 % by mass), previously dried at 200 °C to constant mass into a 400 ml beaker with a lid. Dissolve in 40 ml hydrochloric acid solution (5.10), transfer the solution quantitatively into a 1 000 ml one-mark volumetric flask. Dilute to the volume 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 ≥ 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 volume 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 ≥ 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 volume 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 ≥ 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 volume with water and mix.

1 ml of this solution contains 1 mg of nickel.

5.15.16 Sodium standard solution, 1 g/l

Transfer 2,3051 g of sodium carbonate (purity ≥ 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 to a 1 000 ml volumetric flask. Dilute to the volume 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, decompose 0,2139 2 g of pure silica (SiO_2, purity ≥ 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 to a 600 ml PTFE beaker with a lid, dissolve the fused mass with 400 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 volume with water and mix.

1 ml of this solution contains 100 µg of silicon.

NOTE This solution can be used for about two weeks.

5.15.18 Strontium standard solution, 1 g/l

Transfer 1,685 0 g of strontium carbonate (purity ≥ 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 volume 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 ≥ 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 volume with water and mix.

1 ml of this solution contains 500 µg of tin.